MINES
Saint-Étienne
1816
2016

La métallurgie, quel avenir !

colloque organisé dans le cadre du bicentenaire

du 27 juin au 1er juillet 2016
à l'École des Mines de Saint-Étienne

INSPIRING
INNOVATION
SINCE 1816

INSTITUT Mines-Télécom

SF2M Société Française de Métallurgie et de Matériaux

RNM Réseau National de la Métallurgie

cnrs

SOMMAIRE

ÉDITORIAL

L'École des Mines de Saint-Étienne fête ses 200 ans !

La Société Française de Métallurgie et de Matériaux (SF2M), qui n'a que 71 ans…, et le Réseau National de la Métallurgie (RNM), créé en 2014, sont très honorés d'organiser avec elle le colloque « La Métallurgie, quel avenir ! », dans le contexte de cet anniversaire remarquable.

Nous perpétuons ainsi une mission déjà inscrite dans les statuts initiaux de la SF2M, publiés en janvier 1945. Voici les premières lignes du chapitre « But et Activités », sans coupure :

> *La Société Française de Métallurgie a pour objet de contribuer à l'avancement et à la propagation de la science et de la technique métallurgique et des sciences et industries annexes*
>
> *Elle assure la liaison entre les savants et les ingénieurs métallurgistes français et étrangers*
>
> *La Société Française de Métallurgie organise des conférences et en général toutes manifestations destinées à l'expansion de la métallurgie (…)*

Nous sommes bien au cœur du sujet, dans cette période ou le mot « *métallurgie* » n'est pas souvent porteur d'enthousiasme dans l'esprit de nos concitoyens.

Or la métallurgie est bien une discipline scientifique à part entière, moderne, en évolution permanente dans ses techniques expérimentales et ses conquêtes aux frontières du savoir. Elle est aussi une ingénierie, indispensable dans les champs très nombreux de l'industrie (transport terrestre, aéronautique, énergie, électronique, recyclage, …). Elle est source de progrès techniques et aussi réservoir d'emplois. En effet, les matériaux métalliques sont omniprésents dans notre vie courante et dans toutes les applications de haute technicité : l'optimisation des alliages et la création de matériaux métalliques nouveaux est souvent la clé du succès de projets d'innovation ambitieux, et un des facteurs déterminants de la compétition technologique mondiale, dans la plupart des domaines d'activité.

Les laboratoires de recherche français occupent aujourd'hui une place incontestée à l'échelle mondiale. C'est un atout, à la fois pour le rayonnement de la science française, et pour l'activité en France des industriels, qui peuvent développer des coopérations fortes entre les chercheurs et les ingénieurs, ceux qui conçoivent nos équipements de demain.

L'excellence dans le domaine des matériaux métalliques est porteuse d'enjeux industriels déterminants, et il ne fait aucun doute que la compétition scientifique et technologique à l'échelle mondiale ne s'arrêtera pas dans l'avenir. Nous formons le vœu que ce colloque contribue à rapprocher encore plus nos laboratoires et nos industriels, et à enrichir les connaissances de chacun.

La qualité des laboratoires et des structures R&D des entreprises liés à la métallurgie en France n'est plus à démontrer ! S'alimentant les uns et les autres, ils contribuent à la renommée scientifique et technique de la France dans ce domaine.

C'est donc dans cet esprit de synergie qu'est né le Réseau National de la Métallurgie (RNM) en janvier 2015.

Au sein de la SF2M, le RNM structure les communautés académiques et industrielles dans le domaine de la métallurgie, afin de mettre en place une série d'actions destinées à promouvoir la formation et la recherche en métallurgie, renforçant ainsi l'attractivité du secteur auprès des jeunes diplômés, des ingénieurs et des chercheurs.

Dans la lignée de sa stratégie visant à rapprocher « académiques » et « industriels », le RNM est honoré d' être l'un des organisateurs de ce colloque qui sera la première manifestation publique de grande envergure dans laquelle cette jeune structure sera impliquée.

Marjorie CAVARROC, présidente RNM

Hubert SCHAFF, président SF2M

Les équipes de Mines de Saint-Etienne sont fières d'accueillir le colloque « La Métallurgie, quel avenir ! » en cette année de Bicentenaire de notre Ecole. Je souhaite la bienvenue à tous les participants et ne doute pas que les échanges et rencontres qui auront lieu pendant cette semaine seront fructueux et enrichissants autour d'un champ scientifique historiquement majeur pour l'Ecole. Aujourd'hui, le Centre des Sciences des Matériaux et des Structures (SMS) de Mines Saint-Etienne focalise ses recherches sur les relations entre procédés, microstructures et propriétés des matériaux. Notre volonté de maîtriser ces relations dans les alliages métalliques, les matériaux céramiques et les composites implique une modélisation multi-physique et multi-échelle qui est à la base du concept « Matérials by design ». Le centre SMS est moteur dans la structuration de la recherche dans le cadre de la fédération de recherche Lyon-Saint-Etienne, de l'initiative ManuTech, du réseau RAFAM sur la fabrication additive métallique. Cette dernière est à la base d'un projet structurant pour l'Ecole : **Manufacture du futur.**

Véritable accélérateur de transition industrielle, Manufacture du futur met une chaîne de production pilote à disposition des PME – TPE – PMI. Cette ligne de production mettra en œuvre des procédés de fabrication additive métallique, de fabrication soustractive et de robotique-transitique comprenant un volet numérique intégral ; ceci avec une grande souplesse d’accès pour les entreprises, chercheurs, étudiants. Ce projet est représentatif des valeurs portées par Mines Saint-Etienne : un collectif se mobilise au service du développement économique du territoire !

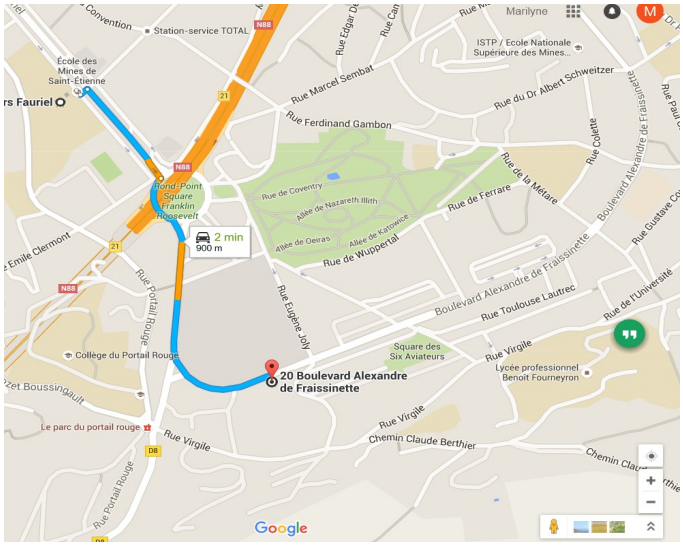
Pascal RAY, directeur de Mines de St-Etienne

INFORMATIONS GÉNÉRALES

Le colloque « La métallurgie, quel avenir ! » se déroule au sein du bâtiment historique de l'École des Mines de Saint-Étienne, au 158 cours Fauriel.

Les activités du soir se déroulent en différents lieux :

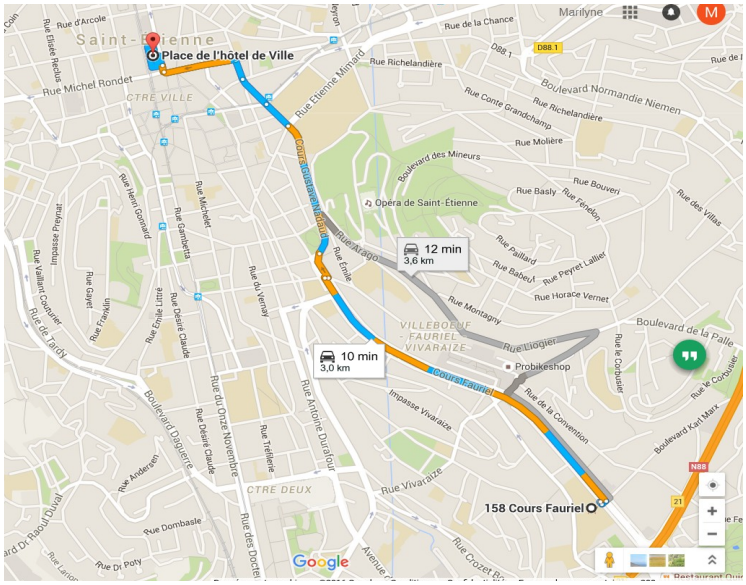
- **Lundi 27 juin 2016** : **Soirée de bienvenue :** Maison des élèves, 20 Boulevard Alexandre Fraissinette à 19h.



Pour s'y rendre à partir de l'École des Mines :

- À pied : 10-15 minutes (montée)
- En voiture : 2 minutes
- En bus : Ligne 6 sur le Cours Fauriel, direction La Marandinière, arrêt Lycée Jean Monnet

- **Mardi 28 juin 2016** : **Réception à la mairie de Saint Etienne**, place de l'Hôtel de Ville à 19h.



Pour s'y rendre à partir de l'École des Mines :

- En voiture : 15 minutes
- En bus : Ligne 6 sur le Cours Fauriel, direction Square violette, arrêt Square violette

- **Mercredi 29 juin 2016** : **visites en ville** au choix lors de l'inscription : musée des Verts, musée d'Art et d'Industrie ou Cité du Design.
  Départ en bus de l'Ecole des Mines

- **Jeudi 30 juin 2016** : **visites des caves et diner de clôture à Condrieu**
  Départ en bus à 17h de l'Ecole des Mines

COMITÉS

Comité du programme

- Gilles AUCLAIR, ASCO INDUSTRIES, Hagondange
- Jean-Luc BECHADE CEA, Saclay, RNM
- Marc BERNACKI MINES ParisTech, Sophia-Antipolis
- Nathalie BOZZOLO MINES ParisTech, Sophia-Antipolis
- Yves BRECHET Haut-Commissaire à l'Énergie Atomique, Paris
- Bruno CHENAL Constellium, Voreppe
- Jonathan CORMIER ISAE-ENSMA/Pprime, Poitiers
- Sabine DENIS IJL, Université de Lorraine, Nancy
- Anne DENQUIN ONERA, Châtillon
- Alexis DESCHAMPS SIMAP, INP Grenoble
- Christophe DONNET LaHC, Université Jean Monnet, St-Etienne
- Anna FRACZKIEWICZ MINES St-Etienne
- Jean-Yves GUEDOU SAFRAN-SNECMA, Paris
- Thierry IUNG ArcelorMittal Global R&D, Maizières-les-Metz
- Guilhem MARTIN SIMAP, INP Grenoble
- Frank MONTHEILLET CNRS / MINES St-Etienne
- François MUDRY IRT-M2P, Metz
- Gilles PERRIN AREVA, Paris
- Michel PIETTE VALLOUREC, Aulnoye-Aymeries
- André PINEAU Mines ParisTech Matériaux, Paris
- Isabelle POITRAULT ARCELOR-MITTAL, Le Creusot
- Frédéric PRIMA Chimie-ParisTech, Paris
- Hubert SCHAFF A&D Paris (groupe ERAMET) & SF2M
- Franck TANCRET Université de Nantes, IMN

Comité d’organisation

- Ségolène COURANT MINES St-Etienne
- Marjorie CAVARROC SAFRAN, RNM
- Anna FRACZKIEWICZ MINES St-Etienne
- Jean-Luc JACQUOT SF2M
- Jean LE COZE MINES St-Etienne
- Jean-Jacques MAILLARD RNM
- Marilyne MONDON MINES St-Etienne
- Hubert SCHAFF A&D Paris (groupe ERAMET) & SF2M

Comité d'organisation local

Ce colloque ne pouvait pas se mettre en place sans une implication forte du personnel de l'Ecole des Mines.

Les responsables des « pôles » : (…)

- Romain QUEY et Marilyne MONDON, documentation
- Delphine JUHEM et Yoann GARNIER, logistique
- Olivier BREUIL, informatique / services techniques
- Patrick GANSTER, coordination de la *get-together party* (lundi 27 juin)
- Ségolène COURANT, organisation des réceptions et visites (mardi 28 juin et mercredi 29 juin)

(…) ont été assistés par de nombreux collègues : permanents, doctorants ou stagiaires du Centre SMS :

- Sophie CARRIER, Jérémy CROQUESEL, Arnaud DUCOULOMBIER, Géraldine FOURNIER-MOULIN, Clément GAYTON, Fares HADDAD, Helmut KLOCKER, Matthieu LENCI, Ada LOZINKO, Michal MROZ, Michella AL NAJJAR, Anaïs NICOLAS, Julia OLSZEWSKA, Mathieu REGNIERE, Sergio SAO-JOAO, Guillaume SMAGGHE, Jacques STOLARZ, Gaëlle VETARD et Adrian VILLALBA-Y-WEINBERG.

Un grand merci à eux tous !
Anna FRACZKIEWICZ, coordination « locale »

SESSIONS

Cinq sessions thématiques

- **ST1 : Procédés de fabrication innovants**

  Coordinateurs : **Christophe Donnet** et **Guilhem Martin**

  Atteindre de nouvelles fonctionnalités, réduire les coûts d'élaboration tout en préservant les ressources exigent un effort d'innovation important en matière de procédés de fabrication. Une attention particulière sera portée à la fonctionnalisation des surfaces (couches minces, procédés de texturation laser ou mécaniques), ainsi qu'aux procédés de fabrication additive.

  Date et lieu : lundi 15h50 - 18h00 (amphi F1), mardi 15h50 - 17h50 (amphi F2), mercredi 16h50 - 17h50 (amphi F2), jeudi 08h30 - 10h10 (amphi F1)

- **ST2 : Comportement sous sollicitations extrêmes**

  Coordinateurs : **Jean-Luc Bechade** et **Jonathan Cormier**

  Dans les domaines de l'énergie (thermique, nucléaire…) et du transport (aéronautique, aérospatial…), certains composants peuvent être amenés à fonctionner de manière continue ou ponctuelle dans des conditions de sollicitations extrêmes de température, de pression (dont chocs) éventuellement couplées avec des environnements agressifs (oxydation/corrosion, irradiation). Des présentations expérimentales et/ou numériques figurent dans cette session.

  Date et lieu : mardi 08h30 - 10h00 (amphi F1), mardi 15h50 - 17h50 (amphi F1), jeudi 10h30 - 12h30 (amphi F1), vendredi 08h30 - 10h10 (amphi F1)

- **ST3 : Grands moyens d'investigation**

  Coordinateurs : **Sabine Denis** et **Alexis Deschamps**

  Caractérisation quantitative de la structure, de la microstructure, des contraintes internes, des défauts ; caractérisation structurale, chimique ; échelles atomique, microscopique, mésoscopique ; grands instruments (synchrotron, neutrons), microscopies électroniques avancées, sonde atomique tomographique ; couplage des méthodes ; caractérisations in-situ, in operando, 3D.

  Date et lieu : lundi 15h50 - 17h40 (amphi F2), mardi 11h20 - 12h40 (amphi F1), mercredi 10h30 - 12h20 (amphi F2)

- **ST4 : Nouveaux matériaux, nouvelles propriétés**

  Coordinateurs : **Anna Fraczkiewicz** et **Franck Tancret**

  Conception, développement et caractérisation de nouveaux matériaux : nouvelles nuances (aciers, alliages d'aluminium, de titane, de nickel…), nouvelles familles (matériaux architecturés, métaux nanostructurés, alliages à forte entropie, verres métalliques, intermétalliques…), matériaux à propriétés originales (mémoire de forme, superélasticité, propriétés fonctionnelles…).

  Date et lieu : mardi 11h20 - 12h40 (amphi F2), mercredi 08h50 - 12h20 et 16h50 - 17h50 (amphi F1), jeudi 10h30 - 12h30 (amphi F2), vendredi 08h30 - 10h10 (amphi F2)

- **ST5 : Métallurgie numérique**

  Coordinateurs : **Marc Bernacki** et **Frank Montheillet**

  La compréhension et la modélisation de l'évolution des microstructures lors de sollicitations thermomécaniques est plus que jamais un sujet de première importance en métallurgie. Le développement massif de stratégies numériques éléments finis ou stochastiques pour lesquelles la microstructure est discrétisée à l'échelle des grains (dites à "champ complet"), de modèles ou la microstructure est discrétisée en catégories de grains (dits à "champ moyen") et de modèles analytiques ou seules les grandeurs moyennes sont discutées en est une parfaite illustration. L'évolution abyssale des moyens de calcul, des méthodes numériques, des modèles métallurgiques et des techniques expérimentales permet en effet aujourd'hui d'avoir accès à l'information locale telles que les phases et les grains, à différentes échelles, et de modéliser de manière plus ou moins précise leurs évolutions. Un axe important de développement consiste à faire dialoguer ces différentes approches de manière découplée aujourd'hui (améliorations/discussions des approches à champ moyen par approches à champ complet et réciproquement) et probablement couplée demain (réelles approches multiéchelles). Le but de cette session sera d'illustrer ces différentes tendances et les perspectives futures avec un large focus sur les méthodes, les modèles et les applications industrielles sous-jacentes.

  Date et lieu : lundi 15h50 - 17h40, mardi 08h30 - 10h00 (amphi F2), mercredi 08h30 - 10h10 (amphi F2), jeudi 08h30 - 10h10 (amphi F2)

Cinq sessions applicatives (tables rondes)

- **SA1 : Recyclage de matériaux**

  Animateur : **Marjorie Cavarroc**

- **SA2 : Energétique : développements, besoins et verrous technologiques**

  Animateur : **Jean-Luc Bechade**

- **SA3 : Transport terrestre : développements, besoins et verrous technologiques**

  Animateur *:* **Jean-Jacques Maillard**

- **SA4 : Formations en métallurgie et matériaux : évolution, besoins, perspectives de marché de l'emploi**

  Animateur : **Jean-Jacques Maillard**

- **SA5 : Aéronautique : développements, besoins et verrous technologiques**

  Animateur : **Jean-Yves Guédou**

Une session poster

Un prix sera remis au meilleur poster présenté par un doctorant. Le prix est sponsorisé par **SAFRAN, Aubert & Duval** et **ARCELOR-MITTAL.**

PROGRAMME GÉNÉRAL

	LUNDI 27 JUIN	MARDI 28 JUIN	MERCREDI 29 JUIN	JEUDI 30 JUIN	VENDREDI 1 JUILLET
08:30-08:40		Amphi F1 / Amphi F2	Amphi F1 / Amphi F2	Amphi F1 / Amphi F2	Amphi F1 / Amphi F2
08:40-08:50					
08:50-09:00					
09:00-09:10	Accueil, inscription, café	ST2 - Comportement sous sollicitations extrêmes : tenue à l'usure, frottement, tribologie / ST5 - métallurgie numérique : recrisatllisation (2/2)	ST4 - Nouveaux matériaux, nouvelles propriétés : nouveaux aciers / ST5 - Métallurgie numérique : solidification – deformation	ST1 - Procédés de fabrication innovants : fabrication voie solide / ST5 - Métallurgie numérique : precipitation / transformation de phases	ST2 - Comportements sous sollicitations extrêmes : tenue en fluage, fatigue / ST4 - Nouveaux matériaux, nouvelles propriétés : interphases
09:10-09:20					
09:20-09:30					
09:30-09:40					
09:40-09:50					
09:50-10:00					
10:00-10:10					
10:10-10:20		Pause café	Pause café	Pause café	Pause café
10:20-10:30					
10:30-10:40	Session d'inauguration : interventions SF2M, RNM, EMSE	Conférence plénière : Yves Bréchet	Amphi F1 / Amphi F2	Amphi F1 / Amphi F2	SA5 (table ronde) : Aéronautique Animateur : Jean-Yves GUEDOU
10:40-10:50					
10:50-11:00					
11:00-11:10					
11:10-11:20					
11:20-11:30		Amphi F1 / Amphi F2	ST4 - Nouveaux matériaux, nouvelles propriétés : nouveaux concepts, nouveaux materiaux / ST3 - Grand moyens d'investigations : utilisation du rayonnement synchrotron pour des observations in situ	ST2 - Comportements sous sollicitations extrêmes : tenue et evolution sous irradiation / ST4 - Nouveaux matériaux, nouvelles propriétés : aciers inox et superalliages a haute performance	
11:30-11:40					
11:40-11:50					
11:50-12:00	Conférence plénière : Jean Le Coze	ST3 - Grands moyens d'investigation : grands instruments / ST4 - Nouveaux matériaux, nouvelles propriétés : nouveaux matériaux			Prix Poster et clotûre du Colloque
12:00-12:10					
12:10-12:20					
12:20-12:30	Déjeuner	Déjeuner	Déjeuner + session posters	Déjeuner	Déjeuner
12:30-12:40					
12:40-12:50					
12:50-13:00					
13:00-13:10					
13:10-13:20					
13:20-13:30					
13:30-13:40					
13:40-13:50					
13:50-14:00					
14:00-14:10	SA1 (table ronde) : Recyclage Animatrice : Marjorie CAVARROC	SA2 (table ronde) : Energie Animateur : Jean-Luc BECHADE		Conférence plénière : Denis Gratias	
14:10-14:20					
14:20-14:30					
14:30-14:40					
14:40-14:50					
14:50-15:00					
15:00-15:10				Pause café	
15:10-15:20					
15:20-15:30	Annonces organisation	Annonces organisation	SA3 (table ronde) : Transport terrestre Animateur : Jean-Jacques MAILLARD	SA4 (table ronde) : Formations Animateur : Jean-Jacques MAILLARD	Visite du Centre SMS
15:30-15:40	Pause café	Pause café			
15:40-15:50					
15:50-16:00	Amphi F1 / Amphi F2	Amphi F1 / Amphi F2			
16:00-16:10					
16:10-16:20					
16:20-16:30	ST1 - Procédés de fabrication innovants : fabrication additive / ST3 - grands moyens d'investigation et ST5 - métallurgie numérique : méthodes d'investigation et recristallisation	ST2 - Comportements sous sollicitations extrêmes : tenue sous métal liquide / hydrogene / sulfides / ST1 - Procédés de fabrication innovants : frittage	Annonces organisation	Annonces organisation	
16:30-16:40			Pause café		
16:40-16:50					
16:50-17:00			Amphi F1 / Amphi F2		
17:00-17:10			ST4 - Nouveaux matériaux, nouvelles propriétés : alliages pour l'aeronautique / ST1 - Procédés de fabrication innovants : fabrication voies liquide ou gazeuse	17h00 - 23h00 : caves et dîner de clôture, Condrieu.	
17:10-17:20					
17:20-17:30					
17:30-17:40					
17:40-17:50					
17:50-18:00					
Activité du soir	19h00 - 21h30 : get-together party, Maison des Elèves	19h00 - xxx : réception - cocktail à la Mairie de St-Etienne	18h00 - 20h00 : visites en ville (au choix)		

LUNDI

09h00 - 10h30 : **Accueil, inscription et café**

10h30 - 11h30 : **Session d'inauguration,** interventions SF2M, RNM et EMSE

11h30 - 12h20 : **Conférence plénière - Amphi F1 :**

« Métallurgie @ MINES Saint-Etienne »

Jean Le Coze

MINES Saint-Etienne

12h20 - 13h50 : **Déjeuner**

13h50 - 15h30 : **Session applicative 1 (table ronde) - Amphi F1 :**

« Recyclage »

Animatrice : **Marjorie Cavarroc**

15h30 - 15h50 : **Pause café**

15h50 - 18h00 : **Sessions thématiques**

ST1 - Procédés de fabrication innovants : Fabrication addtive Chairman : Christophe Donnet - Amphi F1	
15h50 ST1-01	*Conférence invitée :* Nouveaux développements en fabrication additive par fusion laser **Patrice Peyre**
16h20 ST1-02	Modélisation numérique du procédé de fabrication additive SLM appliqué aux céramiques alumine/zircone - Etude de l'évolution de la formation du dépôt de matière **Quiang Chen**
16h40 ST1-03	Achieving Architectured Microstructures by coupling Electron Beam Melting and Spark Plasma Sintering **Guilhem Martin**
17h00 ST1-04	Structures « lattices » fabriquées par EBM **Rémy Dendievel**
17h20 ST1-05	ICME-design of high performance materials, and the ICME approach to the design for additive manufacturing **James Saal**
17h40 ST1-06	Du laboratoire à l'industrialisation pour les produits et procédés : l'étape pilote **Neill McDonald**

ST3 - Grands moyens d'investigations et ST5 - Métallurgie numérique : Méthodes d'investigation et recristallisation (1/2) Chairman : Frank Montheillet - Amphi F2	
15h50 ST5-01	*Conférence invitée :* Prédiction des évolutions microstructurales dans un contexte de mise en forme : vers la modélisation par l'industrie **Amico Settefrati**
16h20 ST3-01	ion CHanneling ORientation Determination (iCHORD) : cartographies d'orientations cristallines sans EBSD **Cyril Langlois**
16h40 ST5-02	Établissement et évolution des interfaces lors du soudage diffusion - Modélisation en champ complet du mécanisme de croissance de grains **Marc Bernacki**
17h00 ST5-03	Extension topologique dérivée de modèles mésoscopiques pour le couplage séquentiel des recristallisations dynamique discontinue et métadynamique **Guillaume Smagghe**
17h20 ST5-04	Full field modelling of lamella splitting and lath spheroidization in α/β titanium alloys **Danai Polychronopoulou**

19h00 - 21h30 : **Get-together party,** Maison des Elèves

MARDI

08h30 - 10h00 : Sessions thématiques

ST2 - Comportement sous sollicitations extrêmes : Tenue a l'usure, frottement, tribologie Chaiman : André Pineau - Amphi F1	
8h30 ST2-01	*Conférence invitée :* Chocs laser pour l'étude des matériaux en conditions extrêmes **Thibault De Rességuier**
9h00 ST2-02	Effect of Si addition on fretting-wear property of Ti-6Al-4V alloy **Jean Geringer**
9h20 ST2-03	Influence de la microstructure sur la durabilité de revêtements durs élaborés à partir de poudres métalliques **Maria-Rosa Ardigo-Besnard**

ST5 - Métallurgie numérique : Recristallisation (2/2) Chairman : Romain Quey - Amphi F2	
8h30 ST5-05	*Conférence invitée :* Modélisation de la recristallisation par champs moyens : avancées récentes, limites et perspectives **Julien Favre**
9h00 ST5-06	Recristallisation dynamique discontinue : Analyse expérimentale, assistée par la modélisation analytique, de la migration des joints de grains et de la germination des nouveaux grains **David Piot**
9h20 ST5-07	Simulation en champ complet de la recristallisation dynamique par une approche level-set **Romain Boulais-Sinou**
9h40 ST5-08	Effect of intragranular strain heterogeneity on recrystallization kinetics assessed by numerical simulation at the mesoscopic scale **Dmitrii Ilin**

10h00 - 10h30 : Pause café

10h30 - 11h20 : **Conférence plénière - Amphi F1:**

« La métallurgie, un héritage, une ressource, un avenir »

Yves Bréchet

Haut-Commissaire à l'Énergie Atomique, Paris, France

11h20 - 12h40 : **Sessions thématiques**

ST3 - Grands moyens d'investigation : grands instruments Chairman : Alexis Deschamps - Amphi F1	
11h20 ST3-02	Tomographie atomique en métallurgie physique: keep moving **Frederic Danoix**
11h40 ST3-03	Comportement viscoplastique de films d'oxydes thermiques caractérisé par diffraction sur Rayonnement Synchrotron **Felaniaina Rakotovao**
12h00 ST3-04	Correlative investigation of boron segregation at prior austenite grain boundaries using atom probe tomography **Claire Debreux**
12h20 ST3-05	Comparison between diffraction contrast tomography and high-energy diffraction microscopy **Andras Borbély**

ST4 - Nouveaux matériaux, nouvelles propriétés : nouveaux materiaux Chairman : Frédéric Christien - Amphi F2	
11h20 ST4-01	Maclage de déformation de l'alliage de titane à mémoire de forme Ti-25Ta-20Nb **Emmanuel Bertrand**
11h40 ST4-02	Architecturation de la microstructure et fonctionnalisation de la surface d'un alliage Mg-2pds%Ca pour application en implants biodégradables **Patricia Donnadieu**
12h00 ST4-03	Microstructure and mechanical behavior of equiatomic CoCuFeMnNi high-entropy alloy **Michal Mroz**
12h20 ST4-04	Conception d'un nouvel acier pour tubes **Florent Decultieux**

12h40 - 13h50 : Déjeuner

13h50 - 15h30 : **Session applicative 2 (table ronde) - Amphi F1 :**

« Energie : Développements, besoins et verrous technologiques »

Animateur : **Jean Luc Bechade**

15h30 - 15h50 : Pause café

15h50 - 17h50 : Sessions thématiques

	ST2 - Comportements sous sollicitations extrêmes : tenue sous métal liquide / hydrogène / sulfides **Chairman : Jean-Luc Bechade - Amphi F1**
15h50 ST2-05	Durabilité des alliages métalliques : vieillissement et effets de l'environnement **Krzysztof Wolski**
16h10 ST2-06	Couplage de techniques de microscopies électroniques, mesures de champs et calculs pour l'étude de l'influence des paramètres microstructuraux sur l'amorçage de fissures en corrosion sous contrainte **Eva Héripré**
16h30 ST2-07	Metallurgical challenge for drilling operations in extreme environments **Andry Ramarolahy**
16h50 ST2-08	Évaluation de la sensibilité à la corrosion sous contrainte des matériaux utilisés dans les alvéoles destinées à recevoir les déchets radioactifs de haute activité **Claude Duret-Thual**
17h10 ST2-09	Comportement en fatigue oligocyclique d'un acier HLE sous chargement cathodique d'hydrogène **Cédric Bosch**
17h30 ST2-10	Relation between microstructure and liquid sodium embrittlement of T91 steel **Ingrid Proriol Serre**

	ST1 - Procédés de fabrication innovants : frittage **Chairman : Damien Fabrègue - Amphi F2**
15h50 ST1-07	Optimisation de l'alliage de magnésium AZ91 élaboré par SPS à partir d'une poudre atomisée métastable **Mathieu Mondet**
16h10 ST1-08	Spark plasma sintering: from Finite Element Modeling of the process up to the elaboration of complex shapes **Christophe Laurent**
16h30 ST1-09	Alliages Ni-W élaborés par Frittage Flash : microstructures et propriétés mécaniques **David Tingaud**
16h50 ST1-10	High strength - high conductivity nanostructured copper and carbon nanotube - copper wires prepared by spark plasma sintering and room-temperature wire-drawing **Christophe Laurent**
17h10 ST1-11	Développements récents et perspectives du frittage sous charge **Frédéric Bernard**
17h30 ST1-12	Nanostructuration et durcissement par précipitation d'un alliage 7020 élaboré par métallurgie des poudres **Hippolyte Queudet**

19h00 : Réception et cocktail - Mairie de Saint-Étienne

MERCREDI

08h30 - 10h00 : Sessions thématiques

ST4 - Nouveaux matériaux, nouvelles propriétés : nouveaux aciers Chairman : Jean-Denis Mithieux - Amphi F1		ST5 - Métallurgie numérique : Solidification, déformation Chairman : Julien Favre - Amphi F2	
		08h30 ST5-09	Approche multi-maillage basée sur les éléments finis pour modéliser un problème magnéto-thermoélectrique lors de la solidification d'alliages métalliques **Olga Budenkova**
08h50 ST4-05	Recent developments in Third Generation Advanced High Strength Steels for Automotive **Antoine Moulin**	08h50 ST5-10	Multiphysic and multiscale models in modeling and simulation of solidification process **Hervé Combeau**
09h10 ST4-06	Nouveaux pas vers l'industrialisation de composants en aciers forgées à l'état semi-solide **Philippe Mangin**	09h10 ST5-11	Microstructure evolution during severe plastic deformation of metals; experiments and modeling **Laszlo S. Toth**
09h30 ST4-07	Détermination des équilibres de phases dans les alliages Fe-C-Mn-Al pour aciers duplex de 3ème génération **Aurore Mestrallet**	09h30 ST5-12	Développement des microtextures de déformation dans l'aluminium : expérience, modélisation à champ complet et modélisation à champ moyen **Romain Quey**
09h50 ST4-08	Low density steels **Ian Zuazo**	09h50 ST5-13	Modélisation numérique de la mise en com--pression par impulsions électromagnétiques **Emmanuel Sondé**

10h10 - 10h30 : Pause café

10h30 - 12h20 : **Sessions thématiques**

ST4 - Nouveaux matériaux, nouvelles propriétés : nouveaux concepts, nouveaux materiaux Chairman : Xavier Sauvage - Amphi F1		ST3 - Grand moyens d'investigations : utilisation du rayonnement synchrotron pour des observations in situ Chairman : Andras Borbély - Amphi F2	
10h30 ST4-09	*Conférence invitée :* Computational design of high strength stainless steels containing up to 10 elements 'from scratch' **Sybrand Van Der Zwaag**	10h30 ST3-06	*Conférence invitée :* 4D X-ray tomography: a brief history, applications and recent developments **Luc Salvo**
11h00 ST4-10	Conception d'alliages par optimisation combinatoire multi-objectif : métallurgie physique, thermodynamique, fouille de données et algorithmes génétiques **Franck Tancret**	11h00 ST3-07	Précipitation dans des alliages aéronautiques par diffusion centrale : des cinétiques aux cartographies de microstructure **Frédéric De Geuser**
11h20 ST4-11	Métallurgie à l'azote versus Métallurgie au carbone ! **Hugo P. Van Landeghem**	11h20 ST3-08	*In situ* investigations of partitioning mechanisms in Q&P steels by synchrotron diffraction experiments **Sebastien Allain**
11h40 ST4-12	Alliages multi-composants à haute entropie de mélange : Quelle définition pour quelles propriétés ? **Mathilde Laurent-Brocq**	11h40 ST3-09	Lattice rotations and internal stress analysis in individual grains during a cyclic stress-induced martensitic transformation in a CuAlBe polycristalline shape memory alloy **Benoit Malard**
12h00 ST4-13	Development of high strength austenitic HEA steels from CoCrFeMnNi family **Anna Fraczkiewicz**	12h00 ST3-10	Apport de la tomographie à la compréhension des mécanismes de fatigue dans les alliages d'aluminium **Erembert Nizery**

12h20 - 15h00 : Déjeuner et session posters

15h00 - 16h30 : **Session applicative 3 (table ronde) - Amphi F1 :**

« Transport terrestre : Développements, besoins et verrous technologiques »

Animateur : **Jean-Jacques Maillard**

16h30 - 16h50 : Pause café

16h50 - 17h50 : Sessions thématiques

ST4 - Nouveaux matériaux, nouvelles propriétés : alliages pour l'aéronautique Chairman : Franck Tancret - Amphi F1	
16h50 ST4-14	Nouveaux alliages de titane à très fort écrouissage : conception, microstructures et propriétés mécaniques **Cédrik Brozek**
17h10 ST4-15	Stratégies de développement de microstructures multi-échelles dans les alliages de Titane « proche beta » : Étude des relations procédés / microstructures / propriétés mécaniques **Frédéric Prima**
17h30 ST4-16	Evaluation of an aluminium - lithium alloy for aerospace application **Pierre-François Behaghel**

ST1 - Procédés de fabrication innovants : fabrication voies liquide ou gazeuse Chairman : Guillaume Kermouche - Amphi F2	
16h50 ST1-13	Vers la fonderie du futur : Polyvalence et Qualité **Laurent Mattéi**
17h10 ST1-14	Fabrication haute cadence de µ-objets par Metal Injection Molding **Denis Vincent**
17h30 ST1-15	Principles of Epanizing, a potential substitute to hot-dip galvanizing **Étienne Petit**

19h00 : Visites en ville (au choix)

JEUDI

08h30 - 10h10 : Sessions thématiques

ST1 - Procédés de fabrication innovants : fabrication voie solide Chairmen : Rémy Dendievel / Guilhem Martin - Amphi F1		ST5 - Métallurgie numérique : précipitation / transformation de phases Chairman : David Piot - Amphi F2	
08h30 ST1-16	Tailoring near-surface microstructures by sliding-based surface mechanical treatments **Guillaume Kermouche**	08h30 ST5-14	Modélisation du comportement mécanique après un transitoire thermique : application au cas de l'alliage à durcissement structural Inconel 718 pour le soudage TIG **Michel Perez**
08h50 ST1-17	Soudabilité de l'acier S460ML par les procédés hybrides laser-MAG et laser-bicathodes **Emmanuel Bertrand**	08h50 ST5-15	Metallurgical models for non-isothermal treatments of a 6061 aluminum alloy **Didier Bardel**
09h10 ST1-18	Un nouveau procédé pour l'élaboration d'un composite à matrice de titane renforcée par des filaments continus de carbure de silicium **Yann Le Petitcorps**	09h10 ST5-16	Influence de l'élasticité sur la cinétique de précipitation dans les superalliages monocristallins **Matthieu Degeiter**
09h30 ST1-19	Évolution microstructurale en soudage par friction malaxage - Application à la prédiction des propriétés mécaniques dans les tôles AA2024 **Gildas Guillemot**	09h30 ST5-17	Vieillissement statique et dynamique dans les alliages métalliques **Matthieu Mazière**
09h50 ST1-20	Premiers résultats de recristallisation d'un acier IF-Ti par chauffage laser **Frédéric Adamski**	09h50 ST5-18	Modélisation des transformations de phase dans les alliages de titane **Hocine Lebbad**

10h10 - 10h30 : Pause café

10h30 - 12h30 : **Sessions thématiques**

ST2 - Comportements sous sollicitations extrêmes : tenue et evolution sous irradiation Chairman : Krzysztof Wolski - Amphi F1		ST4 - Nouveaux matériaux, nouvelles propriétés : aciers inox et superalliages a haute performance Chairmen : Frank Tancret et Jacques Bellus - Amphi F2	
10h30 ST2-11	Corrosion d'alliages de zirconium en milieu primaire des REP - Comment étudier les effets de l'irradiation ? **Marc Tupin**	10h30 ST4-17	Mécanismes de précipitation induite par vieillissement dans des aciers austénitiques coulés par centrifugation et influence sur les propriétés en fluage **Xavier Sauvage**
10h50 ST2-12	Gainages avancés pour les futurs Réacteurs à Neutrons Rapides : défis et perspectives **Yann De Carlan**	10h50 ST4-18	Évolutions microstructurales au cours de traitements thermomécaniques sur un acier inoxydable austénitique stabilisé au niobium **Alexandre Hermant**
11h10 ST2-13	Nano-caractérisation d'alliages modèles FeCr irradiés **Estelle Meslin**	11h10 ST4-19	Propriétés et limites de nouveaux aciers inoxydables duplex - influence des éléments chimiques sur la fragilité de la ferrite et la précipitation de phases intermétalliques **Marc Mantel**
11h30 ST2-14	Étude des évolutions microstructurales sous irradiation de l'alliage d'aluminium 6061-t6 **Camille Flament**	11h30 ST4-20	Compréhension et optimisation du comportement en traction de nuances inoxydables "lean duplex" **Nicolas Meyer**

11h50 ST2-15	Single and Dual-beam in-situ TEM ion irradiation of pure iron and Fe-Cr alloys using the JANNuS facility **Brigitte Decamps**	11h50 ST4-21	Conception multi objectif de superalliages base nickel par thermodynamique analytique, fouille de données et algorithmes génétiques **Edern Menou**
12h10 ST2-16	Characterizations with the MARS beamline (synchrotron SOLEIL) of materials irradiated in nuclear reactors **Jean-Luc Béchade**	12h10 ST4-22	Recristallisation en hétéro-épitaxie dans les alliages à base Nickel γ/γ' à faible écart de paramètre de maille : mécanisme et cinétiques **Marie-Agathe Charpagne**

12h30 - 14h00 : Déjeuner

14h00 - 14h50 : **Conférence plénière - Amphi F1:**

« A la découverte des métadislocations… »

Denis Gratias

IRCP Chimie-ParisTech

14h50 - 15h10 : Pause-café

15h10 - 16h40 : Session applicative 4 (table ronde) - Amphi F1 :

« Formations en métallurgie et matériaux : évolutions, besoins, perspectives de marché de l'emploi »

Animateur : **Jean-Jacques Maillard**

17h00 - 23h00 : Visite de caves et diner de clôture, Condrieu (transfert en bus)

VENDREDI

08h30 - 10h10 : Sessions thématiques

ST2 – Comportements sous sollicitations extrêmes : tenue en fluage, fatigue Chairman : Jean-Yves Guédou - Amphi F1	
08h30 ST2-18	Effet de la diffusion de l'oxygène sur le comportement en fluage d'un alliage de titane. Expérimentations et simulations numériques par éléments finis **Dominique Poquillon**
08h50 ST2-19	Experimental and themodynamic analysis of differences in phase transformation of β-(Ni,Pt)Al coating during isothermal and cyclic oxidation **Vladimir Esin**
09h10 ST2-20	Étude des relations microstructure/propriétés mécaniques d'un nouveau superalliage base nickel : AD730™ **Louis Thebaud**
09h30 ST2-21	Impact of temperature, stress magnitude and crystal orientation on the primary creep response and lifetime of nickel-based single-crystal superalloys **Adriana Mattiello**
09h50 ST2-22	Influence of separate Si and C additions on microstructure and high temperature behaviour of the TNM γ-TiAl alloy **Anne Denquin**

ST4 –Nouveaux matériaux, nouvelles propriétés : interphases Chairwoman : Anna Fraczkiewicz - Amphi F2	
08h30 ST4-23	Effect of interface trapping kinetics on diffusion in polycrystalline materials: hydrogen transport in nickel **Dmitrii Ilin**
08h50 ST4-24	Nouvelles approches pour la quantification des ségrégations interfaciales dans les métaux **Frédéric Christien**
09h10 ST4-25	Some features of strain-induced grain boundary migration (SIBM) in pure Aluminium from SEM and AFM observations **Bermane Béucia**
09h30 ST4-26	Étude de la Mobilité des Joint de Grains proches de 40° autour de <111> et interaction avec des dislocations dans le Cu par dynamique Moléculaire **Zakaria El Omari**
09h50 ST4-27	Plasticité liée aux couplages migration / cisaillement dans les joints de grain **Marc Legros**

10h10 - 10h30 : Pause café

10h30 - 12h00 : Table ronde : Aéronautique - Amphi F1

« Session applicative 5 : développements, besoins et verrous technologiques »

Animateur : **Jean-Yves Guedou**

12h00 - 13h00 : Remise des prix poster et clôture du Colloque

13h30 - 14h00 : Déjeuner

14h30 - 16h00 : Visite du centre SMS

RÉSUMÉS ÉTENDUS

Les résumés sont classés par type / session, puis dans l'ordre d'apparition dans le programme :

- PLE-... : conférences plénières
- STx-... : sessions thématiques
- POS-... : posters

Le numéro d'un résumé (apparaissant en haut de page) permet de retrouver le jour et l'heure de la présentation associée dans le programme des journées.

MÉTALLURGIE @ MINES SAINT-ÉTIENNE

Jean Le Coze

École des Mines, Saint-Étienne, France

La région stéphanoise travaillait le fer depuis le 15^{e} siècle, mais n'avait aucune activité sidérurgique jusqu'en 1815 où des Anglais de Birmingham implantèrent une aciérie au creuset, au voisinage de St-Etienne. L'École des Mineurs fut fondée en 1816 par L.A. Beaunier qui, en 1819, pilota la création d'une deuxième aciérie au creuset au nord de St-Etienne. Ce n'était pas encore le début d'une activité métallurgique à l'École, même s'il en existait un enseignement. Quelques personnages du 19^{e} siècle, liés à l'École, ont, pour différentes raisons, laissé une empreinte dans l'environnement métallurgique stéphanois : de Gallois, Boussingault, Gruner, Le Verrier, Pourcel, Lan. Le plus illustre dans la première moitié du 20^{e} siècle est, sans conteste, Chevenard (Professeur à l'École de 1919 à 1935). Selon l'habitude courante, les laboratoires étaient dédiés à l'enseignement et c'est seulement dans les années 1960, que Claude Goux créa, à St-Etienne, un laboratoire de recherche, également destiné à l'enseignement des élèves. Les premiers axes de recherche, hérités du laboratoire de Vitry où Goux avait préparé sa thèse sous la direction de Georges Chaudron, furent l'étude des joints de grains sur des bicristaux orientés et l'influence d'impuretés sur les propriétés d'emploi des aciers, en utilisant des métaux purs préparés au Laboratoire. Ultérieurement, tout en maintenant ces outils et axes de recherche totalement originaux, d'autres domaines ont été et sont abordés grâce à des moyens et acteurs nouveaux : mise en forme, recristallisation, rupture, fatigue-corrosion, jusqu'aux problèmes de tribo-corrosion des prothèses articulaires et plus récemment, les alliages à "forte entropie".

DISCOVERING TWINS AND METADISLOCATIONS IN Z-MODULE BASED INTERMETALLIC STRUCTURES

Denis Gratias*

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Numerous complex intermetallic phases are built with atoms that are located at positions that can be expressed as linear integrer sums of N>3 generating vectors that are called Z-modules that are 3-dim projections of N-dim regular lattices. We call these structures Z-module based intermetallic structures (such is the case for example of the approximants of icosahedral and decagonal quasicrystals). This leads to a N-dim description that generally exhibits more symmetries than the standard 3-dim analysis. Consequently, new operations of symmetry breaking in the N-dim lattice can be defined that generate original defects in the 3-dim physical space. For example, 5-fold twins have been observed in orthorhombic NiZr phase that can be analyzed as merohedral twins in the original G. Friedel definition, in a 5-dim space. The same holds for dislocations in N–dim space that are defined in the very same way as usual dislocations with Burger vectors belonging to the N-dim lattice (or, equivalently, to its projected Z-module in the physical space). In the physical space, these dislocations are called metadislocations and can be viewed as generalisation to Z-modules of the usual dislocations built on lattice.

We shall present here this new approach of defining defects in N-dim space, (i. e. on Z-modules) for twins and dislocations and illustrate the technic in discussing the Dürer tiling and the defects observed in orthorhombic NiZr structure. We shall extend our study in showing how it is possible to generate what we call scalar dislocations using overabondant modules. These dislocations have Burger vectors orthogonal to the physical space and thus generate no elastic

NEW DEVELOPMENTS IN ADDITIVE LAYER MANUFACTURING OF METALS WITH LASERS

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Keywords: *Additive, Manufacturing, Laser*

Additive layer Manufacturing (ALM) with lasers has now reached an industrial maturity.Two complementary laser processes mostly exist for ALM: the Laser Metal Deposition (LMD) where the laser interacts with a powder stream to generate 3D clads, and the Selective Laser Melting (SLM) technique where laser beam melts a layer-by-layer pre-deposited powder bed. The main differences come from the aspect of manufactured parts: LMD shapes exhibit a lower degree of complexity and higher roughness, mostly due to the additive layer heights (in the 30-100 µm for SLM versus 100-1000 µm for LMD), and the laser beam diameters (around 100 µm for SLM and more than 1 mm for LMD).

As a whole, ALM laser processes clearly appear to be attractive processes for the so-called "Industry of the Future" even if several aspects of the processes still limit their wider dissemination.

Considering the two LMD and SLM processes, a summary on recent investigations carried out in PIMM Lab. is shown involving different specific aspects: (1) the physical analysis and understanding of laser – powder – melt-pool interaction using dedicated diagnostics. This includes the analysis of melt-pool size, hydrodynamic behaviours and instabilities (Fig.1), and their evolution with process parameters, (2) the numerical thermal modelling of the process, mostly focusing on LMD applied to Titanium alloys, (3) some recent investigations on surface finish issues, (4) the ALM manufacturing of titanium matrix composites, using a blend of Ti6Al4V + B_4C powders, and including process, microstructural and mechanical aspects.

Finally, a discussion is proposed concerning future R&D work, and interesting topics to be addressed for the future of laser-based ALM processes.

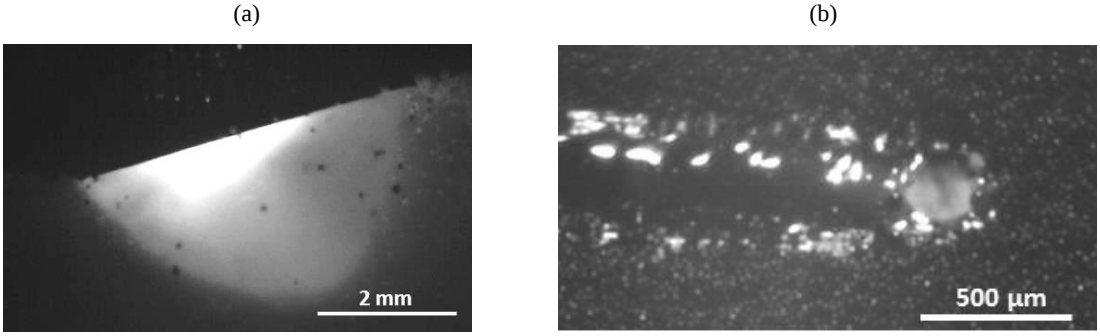

Figure 1: Fast camera analysis of laser-induced melt-pools obtained during the (a) LMD and (b) SLM processes

References

[1] M. Gharbi, P.Peyre, C.Gorny, R.Fabbro, M.Carin, S.Morville, P. le Masson and D.Carron, Influence of various process conditions on surface finishes induced by the direct metal deposition laser technique on a Ti-6Al-4V alloy, *J. Mat. Proc. Tech*. 213 (2013), pp. 791-800

[2] S. Morville, M. Carin, P. Peyre, M. Gharbi, D. Carron, R. Fabbro, 2D longitudinal modeling of heat transfer and fluid flow during multilayered DLMD process, *Journal of Laser Applications*, 24(3) (2012)

[3] S. Pouzet, Fabrication Additive de composites à matrice titane par fusion laser de poudre projetée, PhD Dissertation, Arts et Métiers ParisTech (2015)

NUMERICAL MODELING OF SLM ADDITIVE MANUFACTURING PROCESS - APPLICATION TO ALUMINA-ZIRCONIA CERAMICS

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Keywords: *Selective Laser Melting, FE modeling, ceramics, weld pool shape, solidification*

In the last thirty years, Additive Manufacturing (AM) technology has well developed, with different processes and applications to polymers, metals and also ceramics. The geometry flexibility of AM makes it possible to fabricate parts with complex geometry. These pieces are usually hard or expensive to produce by conventional manufacturing processes like casting or machining. Among AM processes, Selective Laser Melting (SLM) has been considered to be able to fabricate pieces with good mechanical properties as materials are totally melted and fully dense parts can be obtained. Its application to metal alloys is largely studied, while the applications to ceramics are delayed, despite their lightness, outstanding mechanical strength and excellent thermal and wear resistance. More especially the eutectic composition of alumina-zirconia with fine interpenetrated microstructures is of particular interest.

A numerical model with Level Set (LS) method, applied to alumina-zirconia ceramic is proposed [1]. Considering the low absorption of material and its variation in different regions (powder, melt pool, dense ceramic) related to the local concentration of dopants, a volume heat source model based on Beer-Lambert law is proposed. Material properties are averaged between present phases depending on local temperature. The shrinkage from powder to dense matter is modeled by a compressible Newtonian law. The significant effect of surface tension is taken into account by a stable semi-implicit implementation. The evolution of bead shape is tracked by LS and a dynamic mesh adaptation is used to well represent the geometry. Several simulations are conducted to study the influence of process parameters like scanning velocity, laser power and layer thickness, on temperature field and bead shape. The effects of material properties such as absorption and surface tension are also investigated. Different scanning strategies are compared for multiple passes (Fig. 1). At last the microstructure evolution modeled by Cellular Automaton - Finite Element (CAFE) method is illustrated to present the predicted grain structure in SLM process.

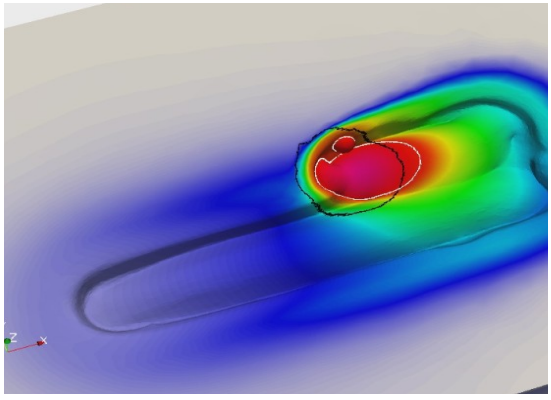

Figure 1: Temperature field during SLM with alternate scanning of a 30 µm powder layer. The black contour shows the location of laser beam and the white one is the liquidus. The bead shape is obtained under the effect of shrinkage from powder to dense matter and surface tension. Formation and fall of droplets can be observed at the border of the melt pool due to surface tension and gravity, respectively.

References

[1] Q. Chen, G. Guillemot, Ch.-A. Gandin and M. Bellet, Proceedings of the 12th International Conference on Numerical Methods in Industrial Forming Processes (NUMIFORM), Troyes, FR (2016).

ACHIEVING ARCHITECTURED MICROSTRUCTURES BY COUPLING ELECTRON BEAM MELTING AND SPARK PLASMA SINTERING

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Keywords: *metal/metal composites, titanium, sintering, additive manufacturing.*

A new route for elaborating architectured microstructures with a controlled 3D arrangement is proposed. A combination of an additive manufacturing technique (Electron Beam Melting) and sintering (Spark Plasma Sintering) is used to generate a microstructural composite made of Ti-alloys. A lattice structure made of one Ti-alloy is first fabricated by Electron Beam Melting (Fig 1a-c). The lattice structure is then filled with another Ti-alloy powder before spark plasma sintering (Fig 1d-f). A bulk specimen is thus obtained with two different microstructures: one inherited from the EBM process in the lattice and the other one resulting from sintering in the matrix (Fig 1g). This innovative technique will enable to achieve new types of metal composite with a controlled 3D architectured designed for a specific application.

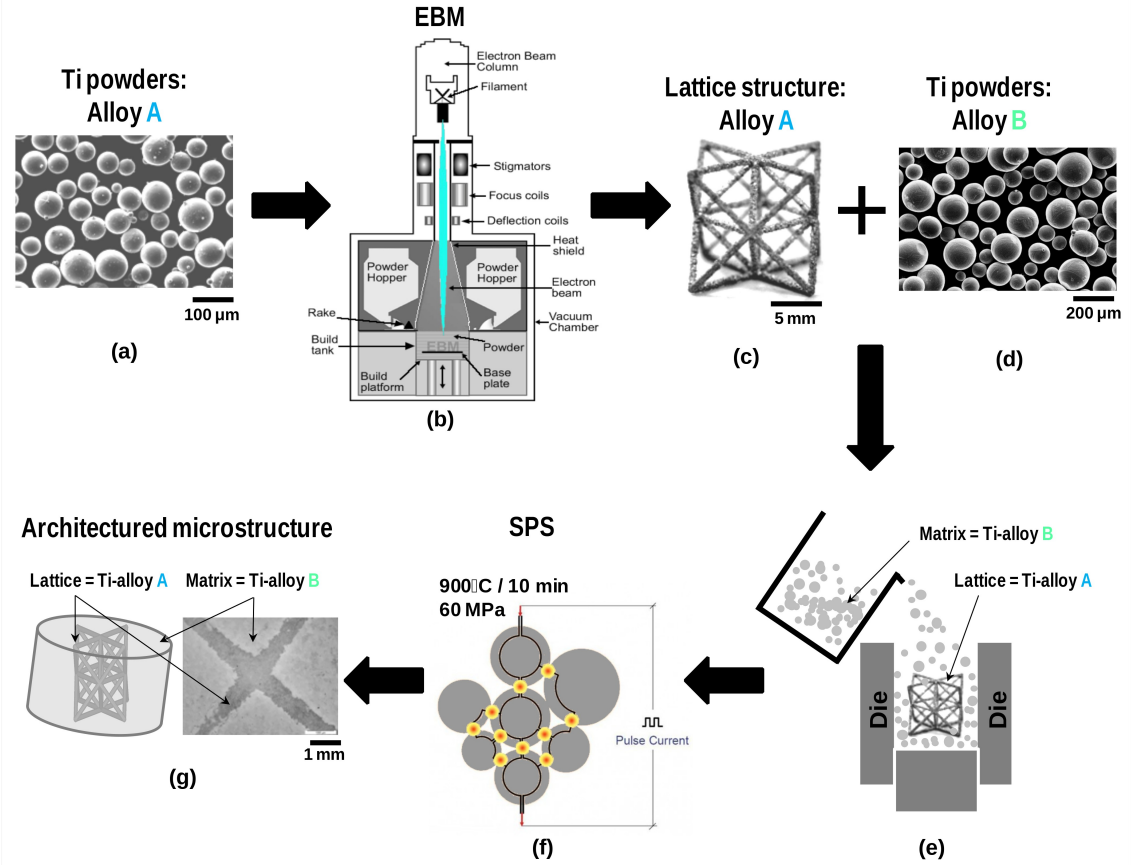

Figure 1: Overview of the innovative route to produce Ti-Ti architectured microstructures. (a) Raw material for producing lattice structures (powder A). (b) Schematic of the EBM process. (c) Lattice structure produced by EBM. (d) Raw material for the matrix (powder B). (e) Introduction of the lattice structure into the die and filling with powder B. (f) Schematic of the SPS process. (g) Ti-Ti architectured microstructures.

LATTICE STRUCTURES MADE BY EBM

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Keywords: *EBM , lattice structures, efficient matter.*

This work focuses on the structural and mechanical characterization of lattice structures produced by Electron Beam Melting. The structure of interest is a 3D periodic lattice whose unit cell is a regular octet-truss (Fig.1 left). The structural characterization mainly relies on X-ray tomography on single struts whereas mechanical properties are assessed by uniaxial compression on structures of different relative densities. For small strut size, the difference between the designed structure and the produced one is large enough to impact the desired mechanical properties. In particular, the matter corresponding to struts roughness or irregularities is not useful from a mechanical point of view. For purpose of simulation, this concept of “mechanically efficient matter” is taken into account by replacing the struts by a cylinder with an equivalent diameter (Fig.1 right). This mechanically equivalent diameter is identified through a numerical computation performed directly on the 3D image of each strut (FFT type simulation). This diameter depends in particular on strut size, strut orientation and melting strategies. After validation with the compression results, it has been used into "realistic" simulations and optimization procedures, thus taking into account the inherited EBM process constraints. In a second step, lattice structures have undergone a procedure of chemical etching. This leads to a clear reduction of the surface irregularities, and in consequence, to a substantial increase of the percentage of mechanically efficient matter. In a last part, different applications of such lattice structures are presented.

Figure 1: left: structure composed of 5x5 octet-truss unit cells -- right: X-ray view of a 1mm vertical single strut and its related mechanically equivalent diameter in brown

ICME-DESIGN OF HIGH PERFORMANCE MATERIALS, AND THE ICME APPROACH TO THE DESIGN FOR ADDITIVE MANUFACTURING

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Keywords: *ICME, materials design, additive manufacturing, high performance materials*

Integrated Computational Materials Engineering (ICME) technologies are becoming increasingly popular in the design and development of new alloys. This presentation will emphasize QuesTek's use of ICME models and tools (e.g., CALPHAD databases, strength models, etc) in the development of novel higher performance materials, as well as expose the ICME approach to the design for Additive Manufacturing (AM).

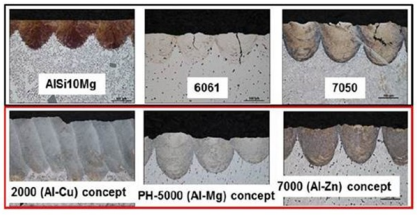
ICME technologies (based on predictive science of precipitation-strengthening and quantum physics cohesive energy calculations) have allowed the design and deployment of Ultra High-Strength (UHS) steels (yield strength over 220 ksi) with improved toughness and a consequent increase in resistance to stress corrosion cracking in demanding aerospace applications (e.g., Ferrium® M54® steel). Extrapolating the UHS steel design concepts to steels of interest to Oil and Gas services and by elucidating the key roles of strengthening mechanisms, grain-boundary segregation and cohesive energy levels, the design of novel HS steels (Yield Strength: 120-160 ksi) with high toughness (KIC: over 100 ksi√in) was achieved.

Recent application of ICME tools in the AM front demonstrates improved efficiency and accuracy in process optimization and qualification, helping to accelerate the maturation of the additive technology in various alloy systems. Case studies of recent development at QuesTek highlights the role of ICME in the process design, validation and uncertainly quantification of the AM of Ni-, Al-, and Ti- based alloys.

Table 1. Comparison of QuesTek's ICME-designed *Ferrium* M54 steel, showing improved strength, stress corrosion cracking and temperature resistance at equivalent toughness to incumbent Hy Tuf for T-45 hook shank application.

Alloy Typical Properties (Longitudinal)	AMS Number	YS (ksi)	UTS (ksi)	K_{Ic} Fracture Toughness (ksi-√in)	Elong (%)	RA (%)	K_{SCC} (ksi-√in)	Temper Temp (°F)
Ferrium®M54®	6516	251	293	115	15	61	100	960
Hy Tuf	6425	200	237	115	12	45	27	500

Figure 1. A completed T-45 hook shank landing gear made from *Ferrium* M54 steel. Rig testing showed >2x life versus incumbent Hy-Tuf Steel, and the Navy estimates $3 Million costs savings due to M54 implementation.



Alloy	Calculated TYS (ksi)
AlSi10Mg	41
6061-T6	52
7050-T74	73
2000-concept	**70**
PH5000-concept	**71**
7000-concept	**78**

Figure 2. QuesTek's use of ICME Technology allowed for the design of new aluminum alloys tailored for additive manufacturing that combine good processability and high strength.

FROM LAB- TO INDUSTRIAL-SCALE: PILOT TRIALS FOR PRODUCT AND PROCESS DEVELOPMENT

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Keywords: *titanium, recycling, powder, aerospace.*

Upscaling from laboratory trials to industrialization is a critical – and oftentimes poorly managed – step in the development of new metallurgical processes and products. Pilot trials are an important way to bridge this "valley of death" by providing data that can be used to better understand and model the industrial-scale process, thereby reducing investment risk and gaining time. That being said, pilot-scale tools are costly and complicated to operate and some uncertainty can remain as to the relevance of results.

MetaFensch is a French publicly funded research centre. Founded in 2014 and based in Uckange (eastern France, near the border with Luxembourg, Germany and Belgium), MetaFensch provides industrial and academic partners with a platform equipped with pilot-scale melting furnaces as well as the personnel and structure to operate them. The principal objectives are to reduce risk and to accelerate the development of innovative products and processes.

Two projects in partnership with Safran and Eramet will be presented in order to illustrate how these challenges of pilot-scale trials are being addressed. The first project's objective is to start from Ti scrap/turnings and produce aerospace-grade ingots. The second will study the atomization of these ingots in order to produce Ti powder for additive manufacturing applications.

From equipment specifications to result analysis and project management, each partner provides complementary skills with a shared view to reducing/eliminating problems related to upscaling these processes.

OPTIMISATION OF THE AZ91 MAGNESIUM ALLOY PRODUCED BY SPS FROM A METASTABLE AS-ATOMISED POWDER

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Keywords: *Magnesium alloys, Spark Plasma Sintering, Microstructure and Mechanical Characterisation.*

The reduction of the energy cost together with the environmental considerations has contributed to raise the scientists' interests in structure lightweighting for transport applications. In this context, magnesium appears to be a promising candidate in order to substitute the main engineering metals used – aluminium and steel – thanks to its low density, respectively 35% and 75% inferior to these metals. Due to its drawbacks, poor ductility and high oxidation sensitivity, magnesium is rarely used in its pure form but generally alloyed with specific elements. Among the Mg alloys, AZ91 is one of the most favourite ones thanks to its moderate cost, enhanced oxidation resistance and improved mechanical strength through a precipitation hardening by, mainly, $Al_{12}Mg_{17}$ precipitates [1]. The mechanical contribution of these precipitates depends on their characteristics (shape, size and amount), commonly controlled through homogenisation + precipitation after the initial casting process.

As casting produces coarse precipitates and grains, powder metallurgy appears to be an effective way to reduce segregation and promote a finer microstructure [2]. Among the available powder consolidation processes, Spark Plasma Sintering (SPS) stands out through its ability to establish short heat cycles [3], preserving fine microstructures via a limitation of the grain and precipitate growth. While SPS has been demonstrated as an efficient consolidation process [3], it is generally followed by post-treatments when precipitation strengthening is required and few investigations were carried out to show the potentiality of SPS to control the phase transformation processes [4, 5].

The present work aims at developing the AZ91 alloy in one step process production by SPS and optimising its mechanical properties by adjusting the SPS processing parameters (time, temperature) to get the full potential of the $Al_{12}Mg_{17}$ precipitation strengthening together with fine grain sizes. To this purpose, an as-atomised AZ91 powder having an initial metastable rapidly solidified microstructure, was used as raw material in order to get a fine precipitation which can be more easily handled during the subsequent SPS thermal cycle. As a result, in addition to a considerable time reduction of 88% compared to a conventional cast AZ91-T6 production [6], higher hardness (+16%) and compressive strength (YCS +59%, UCS +32%) have been obtained for the optimised SPS sintered AZ91 alloy in comparison with the conventional aged cast AZ91-T6, while maintaining a slightly superior ductility (+12%).

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SPARK PLASMA SINTERING: FROM FINITE ELEMENT MODELING OF THE PROCESS UP TO THE ELABORATION OF COMPLEX SHAPES

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Keywords: *Spark Plasma Sintering, modeling, complex shapes*

Pulsed Electric Current Sintering (PECS) techniques have known a huge development over the last two decades. In particular, Spark Plasma Sintering is an extremely powerful technique to sinter all classes of materials as well as their composites

Recently, the modeling of SPS by finite element method has known drastic developments. Coupling three main physics, Electric, Thermal and Mechanic (ETM), it allows now to predict the temperature, grain size and porosity during the process. The electrical and thermal parts of the ETM model are used to calibrate the contact (thermal and electrical) resistances and to calculate the temperature at any point of the SPS tool and column.

Creep parameters on dense and porous materials are determined and sintering models (Olevsky and Abouaf) used to predict the densification of the powders to be sintered. Last, a grain growth law coupled with the densification model may also be considered. Finally this type of modeling allowed us to define the optimized SPS parameters and tool geometry in order to minimize the porosity and microstructure gradients in a complex shape part.

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NI-W ALLOYS PROCESSES BY SPARK PLASMA SINTERING: MICROSTRUCTURES AND MECHANICAL PROPERTIES

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Keywords: *Ni-W alloys, microstructures, tensile tests.*

Innovative microstructures with two crystalline phases: nickel (Ni), cubic faced centred (cfc, ductile component) and tungsten (W), cubic centred (cc, hard component), has been processed by spark plasma sintering (SPS) with a mass fraction of W ranging from 0 to 65% *(Ni-xxW where xx is the mass fraction of W)* [1]. Dense samples (>99.5%) were obtained and their microstructures were characterized by XRD and EBSD. Due to the specificities of SPS, the sintering is sufficiently fast to avoid the formation of intermetallic phases at Ni/W interfaces but doesn't prevent the diffusion of W in the Ni phase, leading to a Ni(W) solid. For low W fraction, samples are thus only made of a Ni(W) solid solution while for higher content the Ni(W) solid solution is surrounding clusters of pure W grains. The amount of W within the Ni(W) solid solution gradually increases with the W mass fraction, up to 18% for the Ni-65W sample. Besides, the Ni grain size decreases with the W content from 19 to 3 µm while the W grain size remains unchanged at about 0.8 µm in all Ni-W alloys.

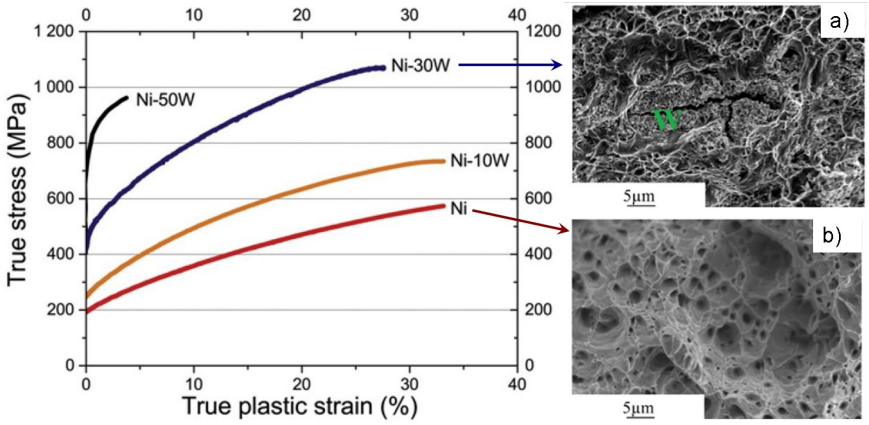

Figure 2: Tensile tests of the Ni-W samples (left) and SEM images of fracture surfaces of Ni (b) and Ni-30W (a) samples.

Tensile tests were carried out at room temperature at a strain rate of 5.6×10^{-4} s^{-1} (fig. 1) and compared to an unalloyed Ni processed in similar conditions. Except for Ni–65W alloy which shows a brittle behaviour, it is observed that increasing the W amount results in strength increase but at the expense of the ductility. Fracture surface analysis shows that the cfc counterpart contains profound dimples (fig. 1a, b) as it carries most of the plastic deformation. Cracks form within the W clusters (fig. 1a) and are systematically blunted by the surrounding ductile Ni(W) solid solution. The observed mechanical properties the processed alloys are combination of various effects and mechanisms such as grain rotation, dislocation accumulation, solid solution and Hall–Petch hardening.

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HIGH STRENGTH - HIGH CONDUCTIVITY NANOSTRUCTURED COPPER AND CARBON NANOTUBE - COPPER WIRES PREPARED BY SPARK PLASMA SINTERING AND ROOM-TEMPERATURE WIRE-DRAWING

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Keywords: *carbon nanotubes, copper, wires*

Conducting copper wires are ubiquitous in today's world and there is a demand for stronger yet lighter ones, in fields such as aeronautics, space and power transportation as well as in niche applications such as materials for high-field magnets. Strengthening copper is achieved by alloying, by introducing another phase or through grain refinement, but these methods also usually increase scattering of the conducting electrons, i.e. they decrease the electrical conductivity [1]. Tensile tests performed on macro- or microscopic dog-bone carbon nanotube - copper (CNT/Cu) composites showed that the presence of CNTs strengthens copper, but strongly decrease the electrical conductivity, which could in part reflect excessive carbon contents (typically 10-20 vol.%) in the samples.

Here, CNT/Cu wires with a very low carbon content (0.5 vol.%) are prepared by a combination of spark plasma sintering (SPS) and room-temperature wire-drawing. Compared to pure Cu wires prepared by the same route [2], the presence of double-walled CNTs (DWCNTs) increases the ultimate tensile strength of the wires by about 10%, whereas their electrical resistivity at 77 K is increased by only about 12%.

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RECENT DEVELOPMENTS AND PROSPECTS OF HOT PRESSING (SPS AND HIP)

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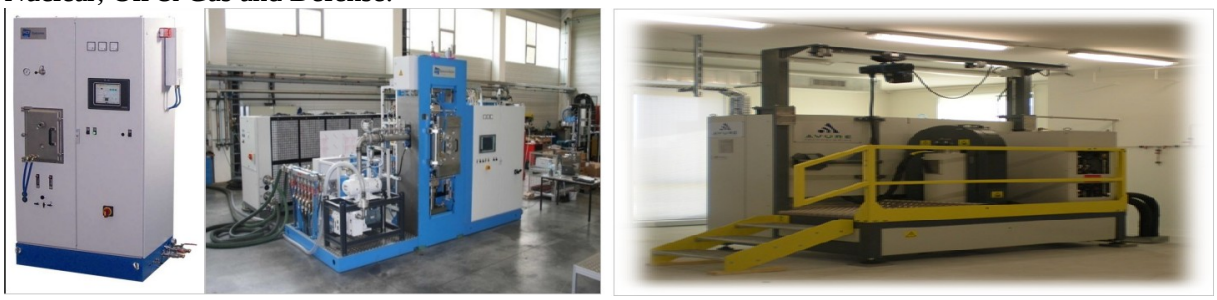
Keywords: *Powder Metallurgy, Spark plasma Sintering, Hot Isostatic Pressing.*

The Powder metallurgy via the sintering technologies under uniaxial load and isostatic pressure is an interesting alternative for the manufacture of metallic and ceramic parts with a well controlled microstructure. Many examples will illustrate this work which is located in the framework of the CICERON project which is a national plan for the deployment of HIP activities. The first stage concerns CICéRON 200 which is located in the new building named Institut Marey - Maison de la Métallurgie at Dijon. This R&D center is composed of three operational machines: two SPS (Spark Plasma Sintering) and one HIP (Hot Isostatic Pressing) (Figure 1) which will be able to work at high temperature (2000°C) and at high pressure (2000 bars). Indeed, the Université de Bourgogne associated to the French cluster of Nuclear Industry (PNB), wish to establish a center of excellence in powder metallurgy. This R&D center will have the ambition to offer the possibility to manufacture metallic and ceramic parts/assemblies with complex shape and / or large dimensions in a semi-industrial version. Moreover, it will be multi-sectoral and will serve all industries as Aerospace, Nuclear, Oil & Gas and Defense.



(a) (b) (c)

Figure 1: Equipments present in CICéRON 200 (Dijon): (a) SPS equipment for research (10 to 40mm) (b) SPS equipment for for both R&D and industrial manufacturing (30 to 160 mm) (c) HIP for both R&D and industrial manufacturing (30 to 200 mm)

One of the originalities of our activities is the mastery of the material microstructures (size of grain but also stoichiometry of heart and surface, nature of interfaces, …), and this, whatever the method of preparation (i) of powders via atomization processes, hydrothermal syntheses or mechanical alloying and (ii) of our sintered materials by SPS (spark plasma sintering) or by HIP. For this, a particular effort is made in the implementation of characterization tools adapted to the scales of our materials. This approach allows us to progress in the understanding of the reaction mechanisms involved in our processes in order to control the critical steps.

PRECIPITATION HARDENING IN A ULTRAFINE-GRAINED ALUMINUM ALLOY AA7020 DEVELOPED BY POWDER METALLURGY

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Keywords: *Strengthening mechanism, Al alloy, Ultrafine grained materials, Spark Plasma Sintering, mechanical properties, high energy ball milling.*

The heat treatable AA7020 aluminium alloy well-known for its high mechanical properties / behaviour was chosen for this study. Commercial powder of this alloy was high energy ball milled and then consolidated through the spark plasma sintering (SPS) technique, whose parameters were optimized to preserve the ultrafine microstructure while producing hardening precipitation. The proposed one-step consolidation process was proven successful and there was no need for further time-costing heat treatment usually performed to improve the mechanical behaviour of such alloys.

The developed material was mechanically tested in quasi-static and dynamic conditions, and compared to an un-milled material and to a commercial heat-treated AA7020-T651 alloy. A quasistatic yield stress of 610 MPa was obtained for the as-sintered milled aluminium (Figure 1). Ma et al. who studied heat treatable alloys of the 7000 serie developed a material by cryomilling followed by HIP, hot extrusion and further heat treatment. They obtained ultrafine grained materials with high yield stress of 734 MPa for an AA7075 [1] and 717 MPa for an AA709x[2]. The material in the present study has similar increase of mechanical properties but is produced with a much faster process.

The strengthening precipitation of the η-phase ($MgZn_2$) was highlighted by microstructural characterisations such as SEM, TEM and XRD. The Hall-Petch, Orowan and solid solution strengthening were quantified to dissociate the different contribution in order to prove the interest of such a process.

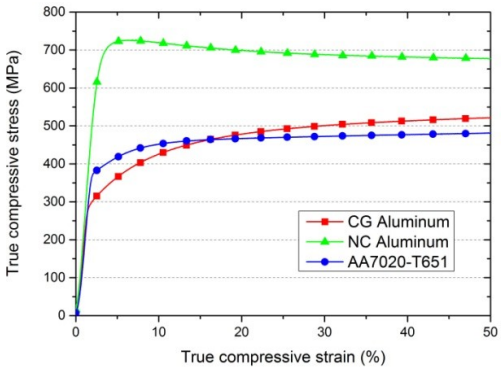

Figure 1: True compressive strain/stress of coarse grained (CG), nanocrystalline (NC) and commercial AA7020.

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THE FUTURE OF FOUNDRIES: QUALITY AND VERSATILITY

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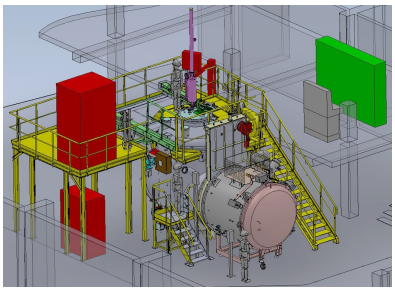
Keywords: *VIM, Vacuum induction melting, mould casting, controlled atmosphere, clean steel*

Foundry melting and casting allows the production of complex shapes and large pieces (oilpans, pump casings, propellers, turbine blades, valves, airplane reactors….) that can be difficult to produce via forging. This activity is, thus, strategic for many industries including automotive, rail, aerospace, construction, and energy. However, due to the current economic context and competition from lower cost base markets, French foundries must evolve to remain competitive, in particular via innovation, quality and the pooling of certain resources.

MetaFensch is a French publicly funded research center. Founded in 2014 and based in Uckange (eastern France, near the border with Luxembourg, Germany and Belgium), MetaFensch provides industrial and academic partners with a platform equipped with pilot-scale melting furnaces as well as the personnel and structure to operate them. The principal objectives are to reduce risk and to accelerate the development of innovative products and processes.

Starting in September 2016, MetaFensch will have a vacuum induction melting (VIM) furnace capable of melting and casting 10L, 30L or 50L of metal (aluminum alloys, steels, copper alloys…) into various moulds (**Figure 1a,b**). The control of various parameters, such as raw materials, melting atmosphere, casting conditions, mould temperature, etc., should improve quality (inclusions, porosity, etc.) and increase the possibility of incorporating recycled materials. Furthermore, this platform should result in prototypes that are representative of industrial-scale phenomena. Real-time measurements and monitoring will give access to all relevant process parameters. (**Figure 1c**).

In collaboration with the CTIF, a VIM project will be presented in which the influence of the furnace's characteristics on foundry quality will be studied. Tests will be carried out on stainless steel and Inconel cast into both sand and ceramics molds. Metallographic analysis of the resulting products will allow comparison between the tests.



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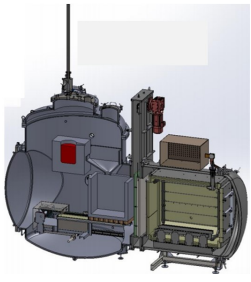

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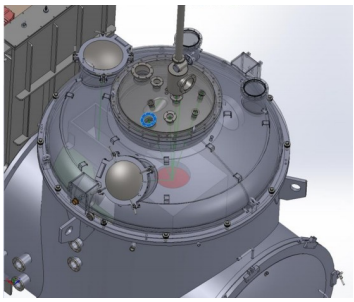

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Figure 1: a/ The VIM platform, b/ Furnace's section and c/ In situ functionalities of the VIM

µ-MIM FOR REDUCING THE COST BY INNOVATION

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Keywords: *MIM, µcomponent, cost reduction, sintering.*

The demand for micro components with more complex shape and higher dimensions precision is growing. The technology based on Metal Injection Molding (MIM) seems to be relevant, but requires still further development work to be efficient. The present work proposes to highlight the capacities of µ-MIM technology. At first the design of the µ-component was defined then the impact of the particles size on the mold filling during injection was studied, and finally an economic study has been established to determine the cost of each process step.
To highlight the interest of µ-MIM process, the project was to produce complex shape µ-parts (figure 1-a) close to existing medical tools, (figure 1-b) including a higher fork length (500 µm Lf) and a thinn[illegible]) of the functional part.

a) µ-MIM demonstrator design **b) Y Hook, nucleus rotator[1]**

Figure 1: µ-component design.

The influence of the particle size was investigated. Two metallic powders were turned into feedstocks for injection with a particle size respectively lower (d_{90}) than 7 (feedstock A) and 16 µm (feedstock B). Feedstocks were produced from metallic powders, polyethylene, paraffin and stearic acid by mixing in higher than the melting point of organic components with a Brabender internal mixer (Plastograph EC plus 350CC). The proportion of each component was adjusted to have suitable rheological properties for injection molding process and a high volume ratio of powder (62 - 70% vol).
The results of the injection tests have shown that the particle size has a strong impact on the mold filling. To succeed a good filling with a high volume powder ratio, the finest powder is preferable.
After debinding and sintering, no default was detected in the samples with a high density (upper 98%). The dimensional measurements have shown a good tolerance about $^{+}/_{-}$ 0.01 mm for the lowest sections Df1, Df2 and Df.

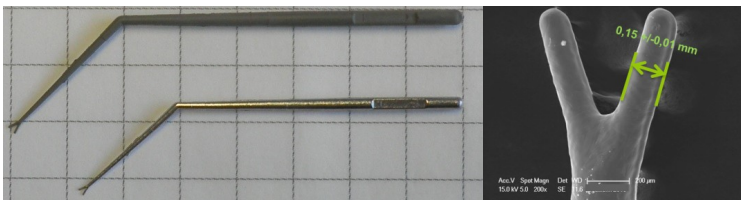

Figure 2: Y Hook elaborated by µPIM.

Finally, the cost distribution of µobject manufacturing by MIM was determined. For a production of 100 000 pieces per year, the cost is estimated between 1.45€ and 1.75€ where the facilities depreciation and tooling represent about 50 % of global cost. MIM process allows a very low cost of µ-parts manufacturing with a high dimensional precision, which can contribute therefore to a global cost reduction of tools available at commercial scale [2],

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PRINCIPLES OF EPANIZING, A POTENTIAL SUBSTITUTE TO HOT-DIP GALVANIZING

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Keywords: *galvanizing, anticorrosion, coatings.*

Hot-dip galvanizing is efficient for protecting steel against corrosion. Hot-dip galvanizing consists in dipping steel pieces or sheets in a molten zinc alloy kept at about 450°C, which reacts with steel. Most of the industrial throughput concerns : - either complex shape pieces batch coated after fabrication ; - either steel sheets continuously coated before stamping and assembling. The zinc-rich coating protects efficiently parts and assemblies against corrosion.

Epanizing is a new vapour-phase process applicable on complex-shape steel parts. The growth of intermetallic Zn-Fe compounds proceeds on hot steel pieces when they are exposed to zinc vapour in a vacuum chamber. The patent WO2010/089110 A1 describes the process [1].

Epanizing strongly reduces assets and running costs. As a matter of fact, no zinc bath is needed: hundreds of tons of zinc must not be maintained any more at 450°C all year long. The new process works on a larger range of temperature than galvanising (250°C <=> 600°C). Processing below 425°C - saves heating expenses, - reduces the thermal stresses in complex shape pieces, and limits the residual stress within the coating after cooling.

Epanizing produces anticorrosion coating similar to galvannealing. However, Epanizing is applicable to complex shape parts, whereas only continuously galvanized steel sheets can be galvannealed. The excellent adherence of painting on Zn-Fe compounds enables to produce long-live duplex coatings.

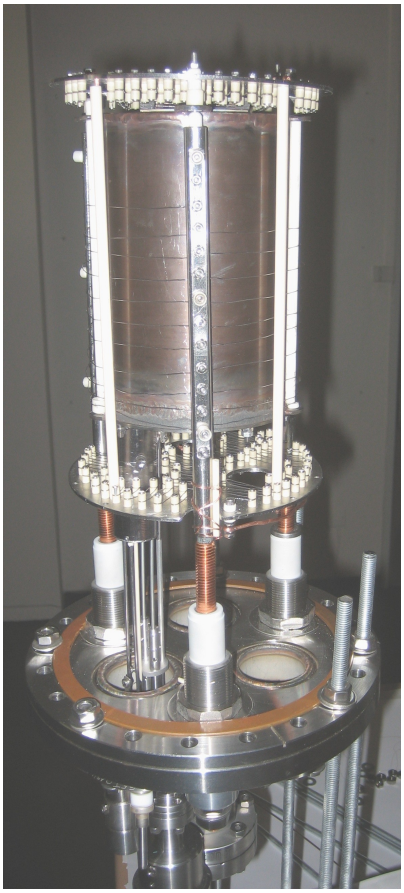

Figure 1: Experimental set up used to validate the process at the laboratory scale.

The simplification offered by this industrial breakthrough is accessible in a narrow range of pressure-temperature conditions, which provides a control of the adsorption-desorption of zinc, as well as its diffusion-controlled solid state reaction with steel. To this respect, Epanizing relates both to low pressure CVD and gas-phase chromatography. The underlying physical and thermo-chemical mechanisms that control zinc transport, reactivity, and the growth of the coating will be addressed during the talk.

Characteristics of process and products from galvanizing, epanizing, electroplating and sherardizing will be compared.

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TAILORING NEAR-SURFACE MICROSTRUCTURES BY SLIDING-BASED SURFACE MECHANICAL TREATMENTS

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Keywords: *Grain refinement, Recrystallisation, Wear resistance.*

The surface treatment notion is usually connected to the modification of surface properties by various actions of physical, chemical, thermal or metallurgical origin (quenching, nitriding, ...). However mechanical loadings alone can also result in the creation of compressive residual stress fields or sub-surface grain refinement. It is often named as “Surface Mechanical Treatments”. The most known SMT are those based on impact loadings, such as the shot peening process for compressive residual stress concerns or such as the SMAT for its ability to create a controlled nano-structured top layer. Sliding-based surface treatment can also be used to generate such interesting surface modifications. It was first related to the brittle “white layer” and viewed as an issue to overcome. It is in the past few years that the idea of optimizing sliding loadings to create a well-controlled grain-refined layer was considered.

In this paper, surface microstructural evolution induced by a new process based on repeated sliding contact is investigated. A set-up, initially designed for simulating contact pressures and cutting speed occurring during machining, is used to create a gradient of nano-micro-structure on a pure copper (Fig 1). It is composed of a top surface recrystallized layer and a sub-surface made of ultrafine grains over a depth larger than 100 µm. Induced-mechanical properties as well as resulting wear resistance are discussed and compared to results of impact-based processes. A conclusion is brought on the benefits of this new kind of surface mechanical treatments for optimizing surface durability by a suitable microstructure design.

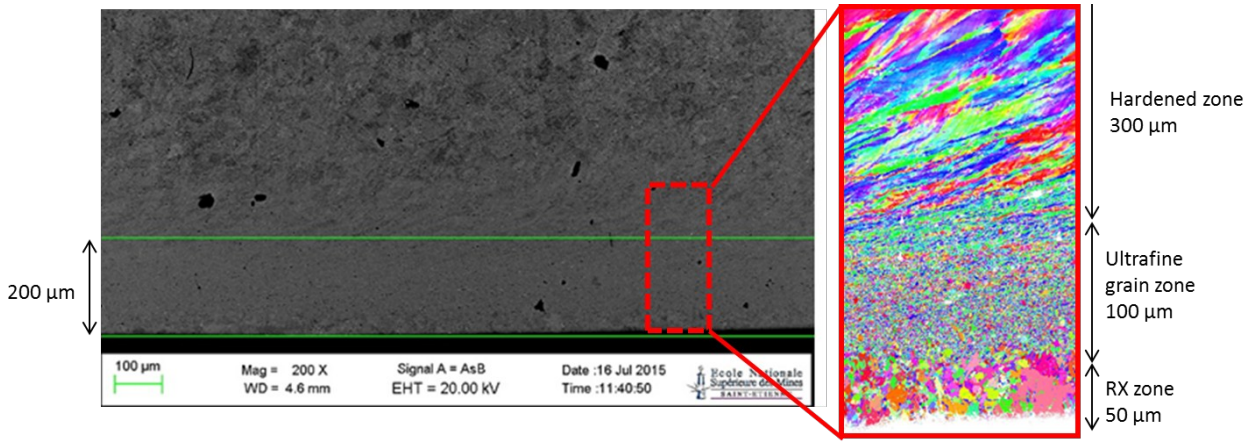

Figure 1: cross-section of the copper bar after surface mechanical treatment. EBSD mapping reveals a dynamically recrystallized zone in the near surface followed by a ultrafine grain region. A transition zone composed of a microstructure oriented in the sliding direction is observed below.

WELDABILITY OF S460ML STEEL USING LASER-MAG HYBRID AND LASER-BICATHODS MAG HYBRID PROCESS

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Keywords: *welding, steel, hybrid process, laser-MAG, bicathod.*

The reduction and performance improvement of the naval structures increase the industrial interest for High Limit of Elasticity steels called "HLE". Among these steels, thermomechanical steels like S460ML acquire their mechanical properties by refining the grain size (Figure 1) in a controlled hot rolling. These steels have a poor composition in carbon and alloying elements and thus have good weldability. ML shades also retain good toughness at low temperature (greater than 16 J/cm^2 at -50°C).

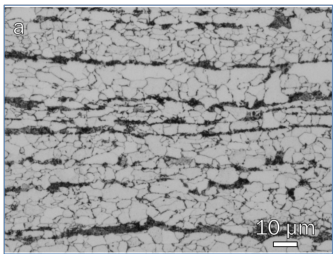

Figure 1: microstructure of base metal S460ML.

The hybrid Laser MAG welding (Figure 2a) has become commercially competitive for ten years and knows a growing success in the shipbuilding industry over conventional processes. However, a major drawback of the use of high density energy processes is the risk of martensite creation in heat affected zone which increases hardness and reduces its resilience. The consequences of adding an additional MAG torch (Figure 2b) on the microstructure and mechanical properties were evaluated.

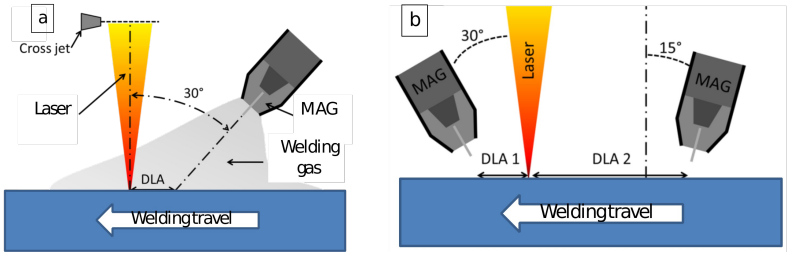

Figure 2: a) "classical" hybrid Laser Mag welding process, b) Bicathodes hybrid Laser MAG welding process.

Figure 3 shows the formation of martensite during conventional welding Hybrid Laser MAG. The hardness above 350 HV do not meet the standards and shipbuilding codes. The addition of a second MAG welding torch allows increasing the cooling time and thus avoiding the formation of martensite in the heat affected zone (Figure 4). Diffrent current were tested as smooth current, pulsed current or CMT® current.

Finally, the bicathodes method allows increasing the productivity by increasing the welding speed by 2, keeping a good quality of welds.

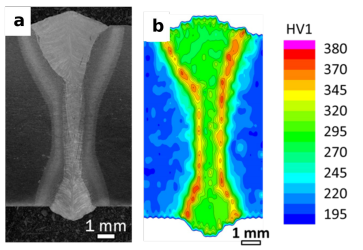

Figure 3: a) macrography of "classical" Laser MAG weld, b) microhardness cartography of weld.

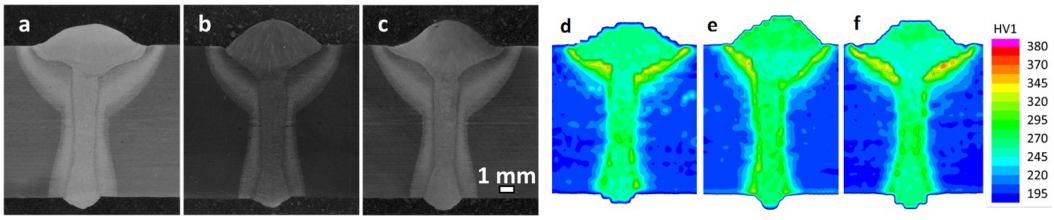

Figure 4: macrography of Bicathodes Laser MAG welds and corresponding microhardness maps with different courant for the second MAG torch: a) and d) smooth current; b) and e) pulsed current; c) and f) CMT® current.

NEW PROCESSES FOR THE FABRICATION OF TITANIUM MATRIX COMPOSITES

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Keywords: *Titanium Matrix Composites, Liquid coating, Strand of wires.*

Titanium Matrix Composites (TMCs) have been studied for nearly forty years [1]. Usually the titanium alloy ($\alpha+\beta$) is reinforced with continuous silicon carbide filaments (140 microns in diameter). In order to protect the SiC fiber against the chemical reaction with the matrix during the hot pressing, a thick (4 microns) pyrocarbon coating is deposited onto the fibre. The most common fibre is known as SCS6 ™.

Several processes have already been investigated in order to fabricate the TMCs such as the Foil/Fiber/Foil followed by a hot compaction (at 1000°C, 1000bar) or the Electron beam PVD coating of the matrix on individual fibres. Concerning the first one, the spacing between the fibres is not well controlled, for the second process, the spacing is perfect but the sputtering speed is low (few microns per hours) which is not possible for industrial applications.

Recently two new processes have been studied. For the first one, the SiC fiber is drawn at a high speed (few meter/second) through the melted titanium allowing the coating of the filament with the required thickness in order to reach a volume fraction of fiber around 30%. For the second one, 6 to 9 titanium wires are strand around a SiC fibre, the diameter of the titanium wire as well as the number of wires control the volume fraction. For the first process it is important to control the wettability and the reaction between the fibre and the matrix, for the second one, it is important to avoid the mechanical damage of the pyrocarbon on the surface of the fibre during the strand. For these two processes, the wrapped fibers are hot pressed in order to densify the composite. The advantages and drawbacks of these two processes will be discussed.

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MICROSTRUCTURAL EVOLUTION DURING FRICTION STIR WELDING, APPLICATION TO THE PREDICTION OF THE MECHANICAL PROPERTIES ON AA2024 ALUMINIUM ALLOYS

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Keywords: *Friction Stir Welding, Modelling, Precipitation, Differential Scanning Calorimetry.*

The standard riveting process used for joining stiffeners (AA 7075) to panels (AA 2024) in aeronautic industry has a detrimental effect on the weight of planes. Friction stir welding (FSW) process is seen as an interesting option and a promising alternative process to lighten aircraft structure. However the mechanical properties in these grades are linked to structural hardening due to the solid state development of fine and densely distributed precipitates. FSW processes are known to affect these precipitates and the final properties of the welds. Thus the estimation of precipitate evolutions is of prime importance in order to deliver reliable welds to the aircraft industry. A time-efficient solid-state precipitation modelling based on a reliable thermodynamic description has been developed in order to predict the precipitate distribution and the related mechanical properties. At a micro scale, a particle size distribution approach (PSD) [1] is proposed for multi-components alloys to predict the nucleation, growth, dissolution and coarsening of both stable and metastable phases. The model is coupled to thermodynamic computations [2] in order to determine precipitate/matrix equilibrium compositions and associated growth velocity. This microstructural model is applied on a AA 2024 aluminium alloy in order to model both stable (S-phase) and metastable (GPB zones) phases. A DSC (Differential Scanning Calorimetry) calibration procedure has been developed to determine the nucleation parameters (Fig. 1 a). At a larger scale, a macro-structural model is used to define precisely the thermo-mechanical evolutions of the metal parts and thermal evolution inside welds [3]. Then, it is possible to couple the micro/macro models in order to define precisely the precipitate evolutions in nuggets, TMAZ and HAZ domains leaInsert Formula ksnapshotding to heterogeneous mechanical properties (Fig. 1 b). The final mechanical properties are presented and discussed.

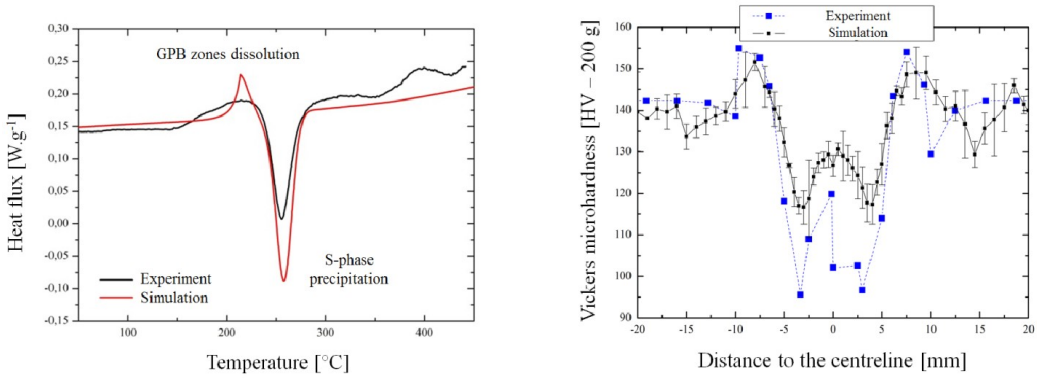

Figure 1: a) DSC analysis of a AA2024 sample / heating rate of 10 °C.min^{-1}. Comparison between experimental (black) and simulated (red) signals. The localization of peaks associated to the GPB zones dissolution and growth of S-phase is shown. b) Prediction of the microhardness (Simulation) and measurements (Experiment).

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FIRST RESULTS OF AN IF-TI STEEL ANNEALED UNDER LASER HEATING

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Keywords: *Laser heating, annealing, IF steel*

IF steels are mainly used in industry for their good balance between formability and good mechanical strength. To obtain drawable sheet, raw steels undergo a series of thermomechanical treatments. The last stage of these treatments consists of a cold-rolling followed by recrystallization annealing, which promotes the formation of a specific crystallographic texture suitable for deep drawing.
Thanks to the recent laser power development, it is now possible to localize the heating and consequently the annealing. it is thus possible to control the recrystallization in the sheet areas which undergo the most critical deformation during the drawing stage.
This work presents the first results obtained on recrystallization of a rolled sheet of Ti-IF steel under static laser heating. The studied material is a sheet of IF-Ti steel, 72% cold laminated. The shape of the laser spot is square, top hat and its surface is about 12x12 mm^2. The laser power tested was low: between 320 W and 400 W.

By measuring the temperature fields with thermocouples, we followed the temperature rate as a function of the laser power. The annealed microstructure was studied both by optical microscopy and EBSD. It shows a gradient of recrystallization between the center and the sides of the laser spots. The problem was that we could observe a big grains size gradient between the center and the side of the recrystallized zone (Figure 1)
To have a homogeneous recrystallized zone, we decided to use the laser in continue and pulse mode. In a first step the laser was used in continue mode to quickly reach the maximum temperature. In a second step, the laser was used in pulse mode to maintain the temperature level a few seconds before cooling by stopping the laser spot.
Finally, we compared these recrystallized microstructures with the microstructures obtained after a “classical” annealing in a furnace. Using EBSD measurement, we studied the grains size and the texture, in particular the area fraction of α and γ fibers. The results show that there are no significant differences between the both processes.

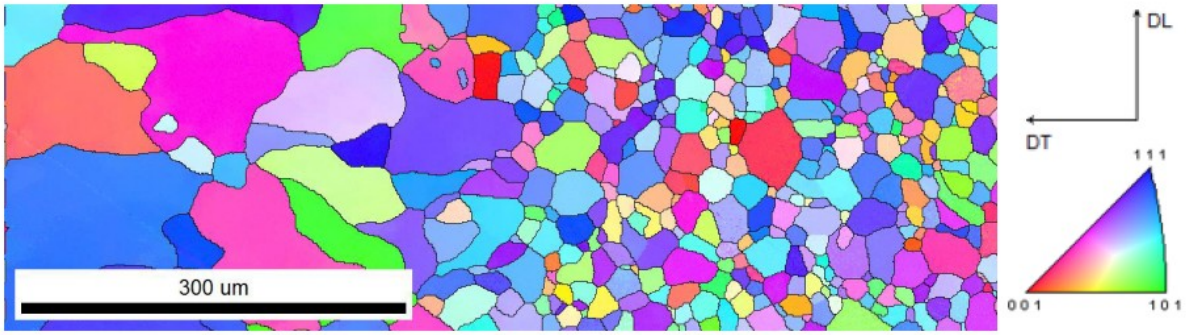

Figure 1: EBSD map of IF-Ti steel annealed by laser heating. The grain size gradient results from the temperature gradient.

LASER SHOCK LOADING TO INVESTIGATE MATERIAL BEHAVIOUR AT EXTREME STRAIN RATES

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Keywords: *laser-driven shock, high strain rates, dynamic behaviour.*

The dynamic response of materials to shock loading is a key issue for many applications, from engineering (safety, armor design, pyrotechnics, hypervelocity impact of space debris, adhesion testing, materials synthesis, high speed machining…) to geophysics (meteoritic impacts…) and transportation (crash worthiness, engine blade loss, bird strike…). In complement to conventional loading techniques based on explosives or plate impacts, high power pulsed lasers allow investigating this response over ranges of *extremely high strain rates*, typically 10^6 to 10^7 s^{-1}, at small spatial (~µm to mm-order) and temporal (~ns-order) scales. They also give access to extremely high pressures (up to ~10^{12} Pa !), relevant for understanding the structure of planetary cores and still out of reach with any other laboratory technique.

Some advantages and limits of laser driven shocks will be illustrated through some selected experiments, in various metals (iron, aluminium, tin…), based on complementary results including time-resolved velocity measurements, ultra-fast transverse shadowgraphy and post-shock analyses of recovered samples. Shock-induced processes governing material behaviour will include yielding, viscoplasticity, polymorphic (solid-solid) transformations, melting, spall damage and fragmentation in both solid and melted states. Finally, how such experiments can be used to develop and test predictive models will be briefly evoked.

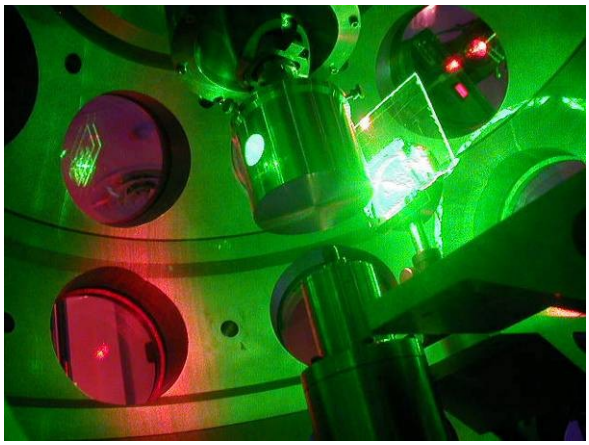

Figure 1: Laser shock experiment inside a vacuum chamber, with a Doppler velocimeter.

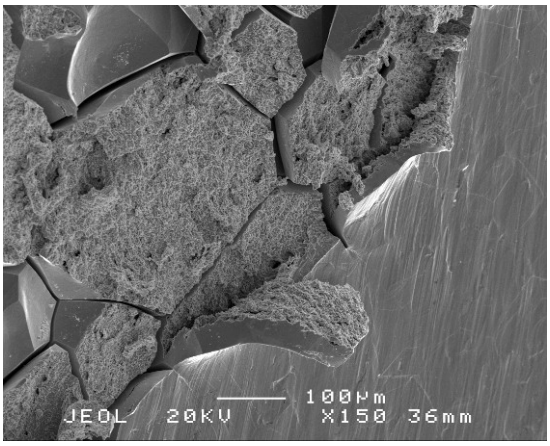

Figure 2: Fracture surface (both trans- and inter-granular) at the edge of a spall crater in the free surface of a laser shock-loaded tin sample.

EFFECT OF SI ADDITION ON FRETTING-WEAR BEHAVIOR OF TI-6AL-4V ALLOY

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Keywords: *Fretting-wear, Ti-6Al-4V alloy, Compliance effect, Wear track area.*

Increasing lifetime of blade-disk contact in aircraft engine is a challenge in aeronautics. The well-known Ti-6Al-4V alloy is well used in this typical assembly due to: High specific strength and high corrosion resistance without any friction process. One might imagine, without any difficulty, that this kind of assembly is submitted to vibrations and micro-displacements, fretting, between every assembly of disk and/or blade, Figure 1.

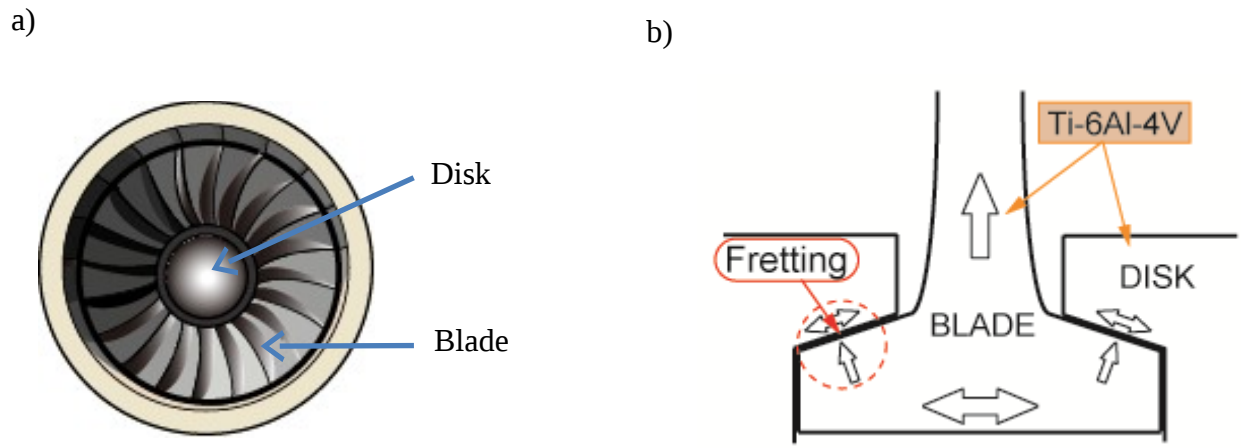

Figure 1: a) fan blade and fan disk related to plane reactor, b) schematic view of fretting location occurring between disk and blade.

Especially during the braking process occurring between the disk and the blade, some fretting contacts may occur. The topic of the study is focused on metallurgy science first, i.e. manufacturing a new typical alloy based on Ti-6Al-4V. Secondly, testing under fretting conditions, actual live mechanisms, in order to highlight either Si has a beneficial effect in the Ti-6Al-4V alloy or not [1]. The conditions are related to fretting test, i.e fretting wear conditions. The contact pressure was about 300 MPa, the sinusoïdal displacement amplitude was +- 40 µm under a frequency of 5Hz. The environemental conditions were dry conditions with Humidity Ratio about 60%. Two types of contact have been compared: Ti-6Al-4V %0Si and Ti-6Al-4V %1Si against Ti-6Al-4V. Under fretting conditions, no effect of Si element on the friction coefficient / dissipated energy, i.e. no ranking process, has been highlighted. However the evolution of the wear volume vs. dissipated energy clearly shows the beneficial effect of Si in the Ti-6Al-4V alloy. Moreover from SEM images of the wear track area, the alloy with Si seems to prevent from cracks initiation in the wear track area. Additional investigations are in progress in order to analyze phases produced after the fretting wear phenomenon.

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INFLUENCE OF THE MICROSTRUCTURE ON THE DURABILITY OF HARD COATINGS OBTAINED FROM METAL POWDERS

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Keywords: *powder metallurgy, microstructure, durability.*

The shutters of the globe valves used in the primary coolant circuits of the nuclear power plants are submitted to important closing pressures. For this reason, the valve seating surfaces need to be coated with a material having high corrosion, mechanical and galling wear properties.
The material chosen and tested in this study is an austenitic-ferritic hypereutectoid stainless steel, the "Norem 02" (24 % Cr, 4% Ni, 4 % Mn, 3 % Si, 2 % Mo, 1,3 % C). This material intends to replace the "Stellite" cobalt-based alloys, which in the primary coolant circuit produce highly radioactive ^{50}Co by radiative capture of neutrons.
The Norem 02 is usually deposited by PTAW (Plasma Transferred Arc Welding). In our study, we compare its microstructure to the one obtained by powder compaction by SPS (Spark Plasma Sintering). The microstructure of the as-deposited PTAW Norem 02 is complex, exhibiting large austenitic dendrites, ranging from about 6 to 20 µm. The interdendritic eutectic phase is composed by Cr_7C_3-type carbides and Mo-rich zones. The chromium content in the matrix is about 18 %, enough to ensure protection against corrosion. On the other hand, the Norem 02 obtained by the SPS process presents a core fine-grained microstructure, composed of austenite, ferrite and $Cr_{23}C_6$-type carbides. A Cr_7C_3 surface layer about 80 µm thick, due to the carbon diffusion from the graphite tools forming the sintering chamber of the SPS press, is observed. Moreover, the chromium content in the matrix is about 13 - 14 %, close to the minimum chromium content required to ensure protection against corrosion.
The nature, size and the distribution of carbides seem to have an important influence on the tribological behaviour [1]. However, in our case, the wear resistance properties cannot be easily predicted on the basis of this criterion, as in the sample obtained by SPS, the core and the surface microstructures are different and are composed of different types of carbides. The height of the carbides present in the core of the material was measured by AFM. No significant difference was put into evidence between the core $Cr_{23}C_6$-type carbides observed in the Norem 02 obtained by SPS and the Cr_7C_3-type carbides in the as-deposited PTAW Norem 02. Nevertheless, as the microstructure also plays an important role in the tribological behaviour, for the Norem 02 obtained by PTAW, the presence of large open matrix areas should negatively affect the wear resistance [1].
The material was aged at 360°C for one week under air. The Norem 02 obtained by SPS shows no significant core microstructure evolution before and after ageing, but the thickness of the surface Cr_7C_3 layer decreases. Carbon and chromium diffuse and redistribute into the matrix, where, after the ageing treatment, the chromium content is about 16 - 17%. Concerning the microstructure of the Norem 02 deposited by PTAW, no significant changes were observed after thermal treatment.
In order to improve the tribological behaviour of the alloy without affecting the corrosion and the mechanical properties, the Norem 02 obtained by SPS was doped with small amounts of niobium and vanadium or Y_2O_3. The first results indicate that, as for the not doped material, a Cr_7C_3 layer forms on the surface of both doped samples. In both cases, the $Cr_{23}C_6$-type carbides were still present before and after ageing. However, the addition of small amounts of niobium and vanadium promotes the formation of niobium carbides and seems to reduce the $Cr_{23}C_6$-type carbide formation. Further investigations are necessary in order to confirm these preliminary results.

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INTERGRANULAR FRACTURE OF STRUCTURAL MATERIALS – AGEING AND ENVIRONMENTAL EFFECTS

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Keywords: *Grain boundary, embrittlement, cracking, Auger spectroscopy.*

In service behaviour of structural materials strongly depends on environmental conditions and materials microstructure, the latter resulting from the processing conditions. One of the most critical types of damage is intergranular fracture that frequently occurs after a limited amount of plastic deformation, well below UTS and sometimes close to the elastic limit.

In this talk we are going to clearly distinguish between intrinsic intergranular brittlness [1] and environmentally induced cracking [2], while assessing 'in service' intergranular brittlness. First, intrinsic intergranular brittlness will be illustrated by thermal aging of a microalloyed steel used for pressurised vessels in nuclear industry. A complete methodology of grain boundary segregation by Auger spectroscopy, including thermodynamical basis of segregation, a special set-up for 'in situ' fractures and quantitative models [3] will be presented. Second, based on the analysis of fracture behaviour of 304H stainless steel, two different mechanisms of intergranular fracture will be introduced : a low-temperature one, usually due to phosphorous segregation and a high-temperature one due to dynamic sulphur segregation. The mobility of these elements towards grain boundaries will be assessed by surface segregation approach based on 'in situ' heat treatments within Auger/XPS spectrometer. Finally, we are going to focus on some cases of intergranular fractures and underline a general sensitivity of high strength steels to hydrogen embrittlement [4]. Two final examples of environmentally assisted cracking of a maraging steel and a supermartensitic stainless steel, both frequently used in 'oil&gas' applications, will highlight not only the sensitivity of these materials to intergranular embrittlement and cracking but also the synergetic effects of microstructure, environment and the level of the applied stress. A perspective on our present research on these synergetic effects will be given.

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UNDERSTANDING THE INFLUENCE OF MICROSTRUCTURE PARAMETERS ON STRESS CORROSION CRACK INITIATIONBY USING ELECTRON MICROSCOPY, FIELD MEASUREMENTS AND FINITE ELEMENT SIMULATIONS

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Keywords: *SCC, Alloy 82, Microstructure, EBSD, DIC, bi-crystal FE simulation.*

Some nickel-based alloys such as mill-annealed A600 are known to be susceptible to Stress Corrosion Cracking (SCC) in Pressurized Water Reactor (PWR) primary circuit. Many components in PWR are joined by welding. Nickel based alloys such as Alloy 182, Alloy 52 or Alloy 82 are used to joint some of these components. This study focuses on the initiation mechanism of SCC for Alloy 82 V-groove welds. A methodology combining mechanical loading in an environment representative of primary circuit and different experimental devices for a multi-scale analysis has been chosen as described in figure 1. Such a choice aims at a better understanding of the role that microstructure and local mechanical loading may play in crack initiation [1][2].

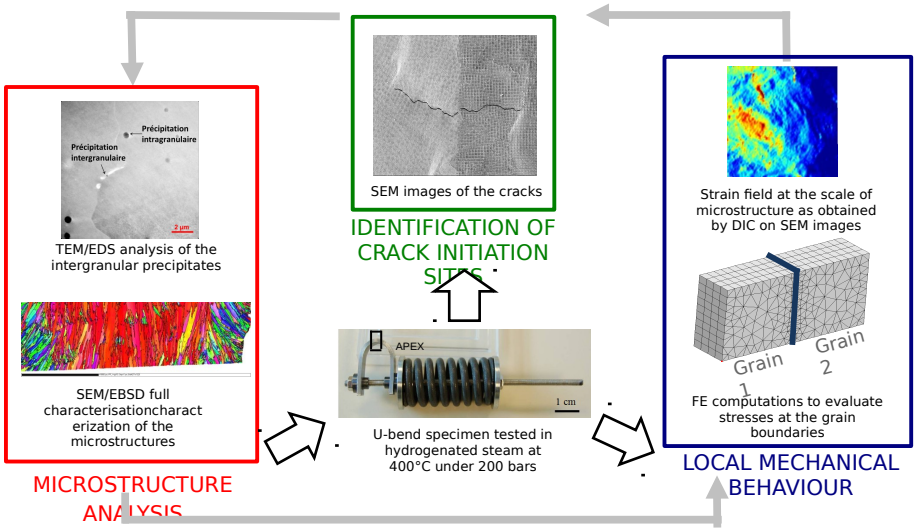

Figure 1: Methodology adopted to identify the critical parameter that governs SCC crack initiation.

SCC occurs when combining three factors: a *susceptible material* under a *mechanical loading* in a corrosive *environment*. The environment is fixed and chosen to be hydrogenated steam at 400°C. Electron Back Scattering Diffraction (EBSD) as well as Energy Dispersive Spectroscopy (EDS) on TEM lamellae have been performed to characterise the material and its grain boundaries. The analysis of strain field at the scale of the microstructure on U-bend specimen coupled to finite element computations of couples of grains enables us to propose a criterion to evaluate the susceptibility of grain boundaries to SCC.

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METALLURGICAL CHALLENGE FOR DRILLING OPERATION IN EXTREME ENVIRONMENTS

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Keywords: *High mechanical properties, high toughness, Sulfide Stress Cracking resistance*

New oil and gas fields are getting scarcer and therefore more difficult to locate with conventional equipment and techniques. Currently, the oil and gas industry is using new extraction technologies and is driving expansion in unconventional spaces: drilling in ultradeep seawater, hydraulic fracking and horizontal drilling technology that require very high mechanical properties. Vallourec group is therefore developing new steel grades combining high mechanical properties with high corrosion resistance and high mechanical properties with high ductility.

Firstly, the susceptibility to Sulfide Stress Cracking is closely dependent on the mechanical properties of the material. This assertion is illustrated in Figure 1: for a given applied stress and for a given corrosive environment, the same steel grade passes up to limiting yield strengths, beyond which the failure occurrence increases dramatically. Such threshold curves define the maximal admissible yield strength and thus the range of yield strengths for a grade.

Secondly, high mechanical properties impact toughness energy and ductility. Most of the time, by adding alloying elements to Fe-C steels there is a clear enhancement of strength but a deterioration of impact toughness, related to the formation of detrimental precipitates (from their nature and/or their size). The optimization of heat treatment allows the control of microstructure, for instance sub-grain and highly disoriented packets, these last ones being keys in the control of crack propagation.

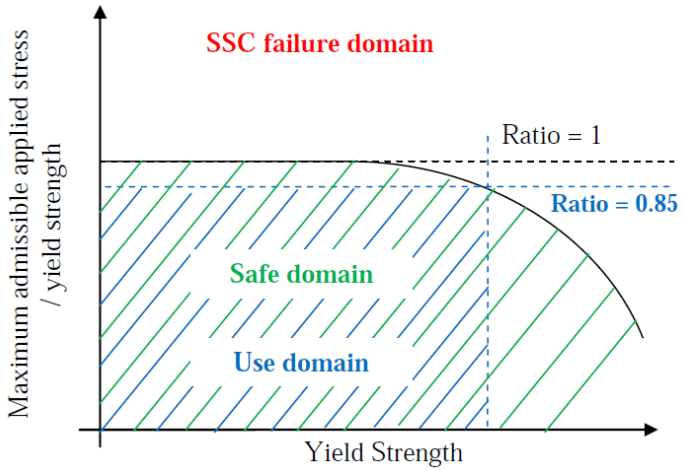

Figure 1: Material characteristic curve for passing or failling to the NACE Method A corrosion test.

ASSESSMENT OF THE SENSITIVITY TO ENVIRONMENTAL ASSISTED CRACKING OF MATERIALS USED IN NUCLEAR WASTE REPOSITORIES

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Keywords: *environmental assisted cracking, carbon steels, nuclear wastes.*

The nuclear industry produces a significant quantity of radioactive wastes, three percent of these wastes, so called high level wastes (HLW), concentrate over 99% of the whole radioactivity. The long term management of these HLW represents a challenge for the scientific community. Therefore, the long term disposal of HLW is studied by ANDRA, investigating steel overpacks buried in deep geological repositories. The repository consists of a multi-barrier system, specifically foreseen for safety purposes. Until 2014, the multi-barrier concept was composed of the HLW cooled in a glass matrix, itself introduced in a stainless steel container emplaced in a C-steel overpack. The disposal cell consists of micro–tunnels drilled in the Callovo-Oxfordian (Cox) claystone, inside which C-steel casing would be introduced. The overpack would then be inserted in the casing.

The carbon steels considered for both casing and overpacks will be in contact with clay and/or with seepage water in equilibrium with clay, first in solutions containing dissolved oxygen, then in anoxic conditions. A maximum temperature of 90°C is expected after the introduction of overpacks. The pH of the solutions varies from slightly acidic to slightly alkaline, and dissolved gas are present (CO_2, H_2 formed by the cathodic reaction of the corrosion processes). The chemical composition is complex, with the presence of carbonates, silicates, sulphates…[1]. Even if the repository conditions do not correspond to known conditions able to trigger Environmental Assisted Cracking (EAC), and could be considered as not critical with respect to EAC, the requirement for extended lifetimes of the overpacks and casing implies an evaluation of the EAC behaviour of these materials [2]. Recently, the HLW reference concept was slightly modified to buffer the potential acidic transient resulting from pyrite oxidation [3]. The injection of a cementitious grout (pH of the poral solution around 11) between the claystone and the casing is expected to reduce considerably the corrosion rate and limit or even suppress the EAC risks in anoxic conditions. However, the evolution of this environment along the sequences of repository exploitation has to be taken into account and EAC risks evaluated in the whole range of anticipated pHs and potentials.

A program dedicated to the evaluation of EAC susceptibility of carbon steels selected for casing and overpacks has been launched including the new reference concept, addressing both initiation and propagation stages. Testing facilities were developed to perform long term EAC experiments (up to 8000 hours) with stressed specimens. The EAC assessment relies on both “technological” long duration tests in conditions representing the evolution of the repository environment and on more fundamental studies relating to the identified mechanisms of EAC damage, in particular stress corrosion cracking induced by the formation of pseudo-passive layers on the carbon steels.

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COMPORTEMENT EN FATIGUE OLIGOCYCLIQUE D'UN ACIER HLE SOUS CHARGEMENT CATHODIQUE D'HYDROGÈNE

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L'endommagement par fatigue résulte de l'hétérogénéité de la déformation plastique au sein des matériaux, de la nature de cette irréversibilité et de son expression en surface. Sous environnements agressifs, et en particulier sous condition hydrogènante, la réactivité des surfaces est affectée. L'adsorption, l'absorption et le transport d'espèces tels que l'hydrogène peut en retour affecter le comportement plastique du matériau. Cette étude s'est intéressée à ces mécanismes sur un acier à haute limité d'élasticité de microstructure trempée revenue. Les évolutions des composantes internes et effectives de la contrainte d'écoulement avec la déformation plastique cumulée ont été analysées par la méthode de Handfield et Dickson, et mises en regard des évolutions de la microstructure à l'échelle des dislocations à partir d'observations en MET et analyses de pics de diffraction RX pour diverses amplitudes de déformation plastique et déformations plastiques cumulées. Ces essais de fatigue oligocycliques ont été réalisés à l'air et sous conditions de chargement cathodique d'hydrogène. Les résultats indiquent que l'hydrogène n'a pas d'effet sur la contrainte interne et la contrainte appliquée, alors que la contrainte effective est altérée. La partie athermique décroissante est interprétée par des effets d'écrantage des interactions à courte distance des dislocations liées au piégeage de hydrogène. Tandis que l'augmentation de la composante thermique est expliquée par des effets de modification du cœur des dislocations vis.

RELATION BETWEEN MICROSTRUCTURE AND LIQUID SODIUM EMBRITTLEMENT OF T91 STEEL

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Keywords: *Liquid Metal Embrittlement, Tof-SIMS, Small Punch Test, Bending Test, Fracture analyse*

The present communication summarizes the results obtained inside a research program devoted to material integrity for the sodium fast nuclear reactor. Though tough and ductile materials are selected, they may become brittle when stressed in liquid sodium exhibiting thus the so called Liquid Metal Embrittlement (LME).
The investigation performed on a 9Cr1MoNb-V martensitic steel (T91) deformed in liquid sodium points out the necessity to perform in depth metallurgical analyses to explain the observed LME. Both effects of microstructure and of experimental conditions have been studied. Small Punch Tests were performed in air and liquid sodium, at different strain rates and different temperatures. Bending tests on notched specimens were performed in air and in liquid sodium. The T91 steel was studied as well after tempering at 750°C (recommended heat treatment) as 550°C. The latter condition made T91 more sensitive to liquid sodium. Indeed, in liquid sodium, T91 tempered at 550°C exhibited a brittle fracture while it was ductile in air. Liquid sodium initiated an intergranular crack which then propagated by transgranular mode in the bulk of the specimen as observed by SEM. However, T91 tempered at 750°C was ductile in air as in sodium. The strong difference between the two tempering conditions has been attributed to the microstructural changes: precipitation (type, partition, localization) and phosphorus segregation. ToF-SIMS analyses (Figures 1 and 2) showed that sodium penetration occurred under loading at prior austenitic grains boundaries and at laths boundaries for the 550°C tempered condition but not for the 750°C one. These metallurgical analyses allowed us to propose a mechanism which explains the sensibility to LME according to the microstructure.

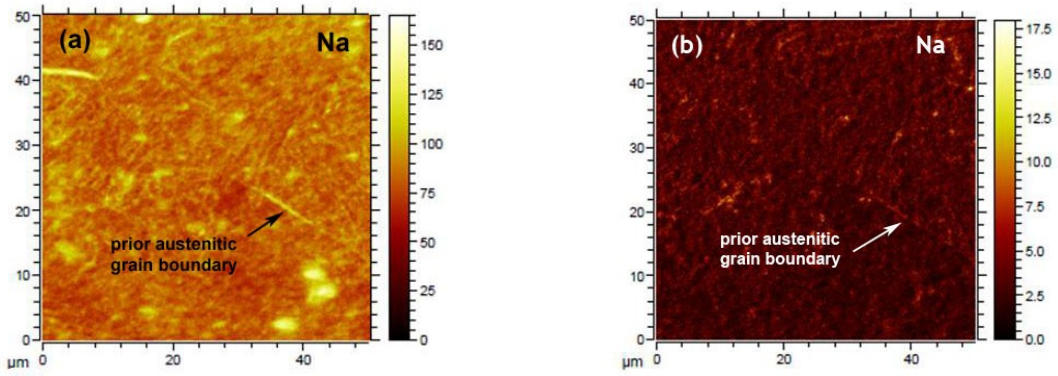

Figure 1: ToF-SIMS images obtained in positive mode after surface sputtering representing Na^+ cartography on plastically deformed specimen: (a) 0.2 µm depth, (b) 0.3 µm depth

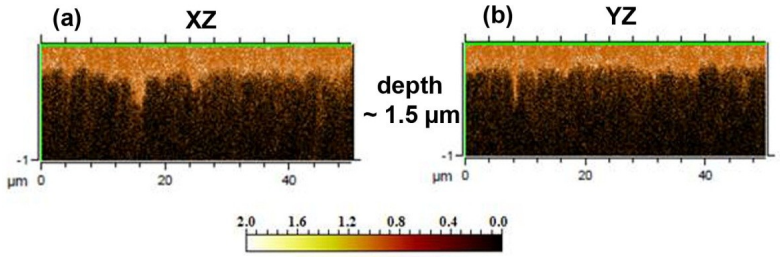

Figure 2: Sodium cartographies obtained by ToF-SIMS of the plastically deformed specimen after pre immersion in sodium for two different cross sections (a) XZ, (b) YZ

UNDERSTANDING OF CORROSION MECHANISMS UNDER IRRADIATION OF ZIRCONIUM ALLOYS - HOW CAN WE STUDY IRRADIATION EFFECTS?

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Keywords: *corrosion, irradiation, zirconium*

In Pressurized Water Reactors, zirconium alloys used as fuel cladding materials are exposed to aggressive aqueous environment (285-325°C, 155 bars, 0.7 to 2.2 ppm Li and 10 to 1200 ppm B). In these conditions, the corrosion kinetics of Zircaloy-4 alloy shows acceleration under irradiation, in comparison with reference autoclave kinetics, the magnitude of this acceleration depending on alloy type, burn-up, irradiation conditions. One of the hypotheses to explain the contribution of irradiation to the enhancement of corrosion rate is the damage due to the neutron irradiation undergone by the materials.

Irradiation can damage many parts of the fuel cladding, its effects on the metallic matrix are well characterized, but it also generate some defects in the oxide layer formed on this material that can accelerate the corrosion by affecting transport properties in this layer. It is possible to simulate irradiation defects through the use of ion irradiation.

This work is devoted to the effects of ion irradiation of the oxide layer and the metallic matrix on the corrosion rate of zirconium alloys.

An original approach based on alternated oxidation steps has been developped to study the irradiation impact on the kinetics of zirconium alloys.

While the ion irradiation of oxides formed on Zy4 results, as expected, in an increase of the corrosion rate[1, 2], the conclusion is on the other hand different in the case of Zr-1%Nb[1]. The incidence of these results on our present understanding of corrosion mechanisms of these two Zr alloys are discussed.

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GAINAGES AVANCÉS POUR LES FUTURS RÉACTEURS À NEUTRONS RAPIDES : DÉFIS ET PERSPECTIVES

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Keywords: *Austenitic steels, ODS, cladding tube, sodium fast reactors.*

Traditionally, the material for the cladding of the fuel rods for Sodium Fast Reactor (SFR) is austenitic steel. These materials are known to swell under irradiation which limits their lifetime. Indeed, vacancies created under irradiation can condense to form clusters and cavities that cause swelling. In the framework of the fourth generation reactors activities, many studies are conducted to develop new alloys and to increase significantly the lifetime of these materials.
The purpose of this presentation is to show the challenges and the latest materials developments led at CEA to address this issue. Different strategies are applied with the use of conventional metallurgy for advanced austenitic steels or powder metallurgy and co-grinding for the development of Oxide Dispersion Strengthened (ODS) ferritic / martensitic alloys.

NANO CHARACTERISATION OF FECR MODEL ALLOYS IRRADIATED WITH CHARGED PARTICLES

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Keywords: *Irradiation, RIS, FeCr alloys.*

High-Cr Ferritic-Martensitic (F-M) steels and Oxide Dispersion Strengthened (ODS) alloys are potential candidates for the cladding and the structural materials of the future GEN IV and fusion reactors because of their excellent swelling resistance, small ductile-brittle transition temperature (DBTT) shift and good thermal properties [1]. These materials, derived from FeCr alloys, will be exposed to neutron fluxes generating matrix damage (dislocation loops and bubbles/cavities) and Cr segregation/precipitation, promoting embrittlement under irradiation. Within this study, model irradiation experiments using alternative irradiation sources have been performed on Fe-Cr model alloys. The high purity FeCr model alloys were elaborated at the Ecole des mines de Saint Etienne. The irradiations have been performed using 2 MeV self-ions and 1 MeV electrons respectively in the Jannus Saclay facility at 773K and in the High Voltage Electron Microscope at the CEA/SRMA at 573K. Matrix damage evolution has been followed by Transmission Electron Microscopy (TEM) while the Cr evolution has been followed by Atom Probe Tomography (APT).
The analyses of the ion irradiated FeCr alloys (3, 5, 10, 14 wt.%) revealed the non-monotonic effect of Cr on the swelling [2]. Furthermore, we show 2 phenomena of radiation induced segregation (RIS) of Cr on dislocation loops at self-ion irradiation at 773K [3] and of radiation enhanced precipitation (REP) of the Cr enriched α' phase in a Fe14%wt.Cr after electron irradiation at 573 K [4] (Figure 1). Comparison of α' microstructures after neutron and electron irradiation revealed that electron irradiation is more efficient to enhance α' precipitation.

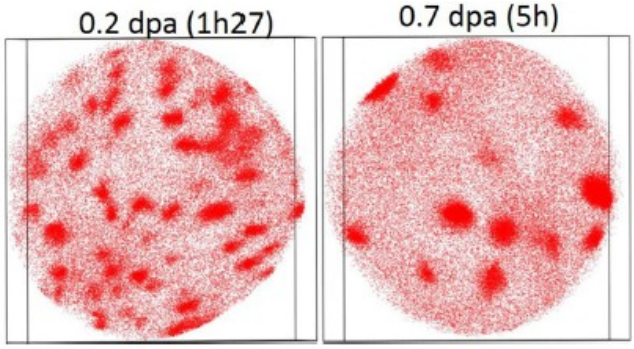

Figure 1: 3D distribution of Cr atoms in the Fe14wt.%Cr alloy irradiated with 1 MeV electron at 573K at 0.7dpa.

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STUDY OF MICROSTRUCTURAL EVOLUTIONS OF THE 6061-T6 ALUMINIUM ALLOY UNDER IRRADIATION

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Keywords: *6061-T6 aluminium alloy, vessel, β" nano-precipitates, ion and neutron irradiations, Transmission Electron Microscopy, Atom Probe Tomography.*

The 6061-T6 Aluminium alloy, whose microstructure at the nanometric scale contains Al(Fe,Mn,Cr)Si dispersoids and hardening needle-shaped (Mg, Si) β" precipitates, has been chosen as the structural material for the core vessel of the Material Testing Jules Horowitz Nuclear Reactor [1]. Because it will be submitted to high neutron flux at a temperature around 50°C, it is necessary to study microstructural evolutions induced by irradiation and especially the stability of the second phase particles. In this work, transmission electron microscopy and atom probe tomography have been used to characterize the evolution of nano-precipitates submitted to in situ and ex situ ion irradiations, as well as neutron irradiations. Dark field microscopy in the [100] orientation reveals that β'' precipitates partially dissolve and tend towards a transformation to cubic precipitates under neutron irradiation at low dose (<12 dpa). Observations in the same dark field conditions show that β'' completely dissolve after 165 dpa ex situ W^{3+} (2MeV) irradiation performed at room temperature at the JANNUS Saclay facility [2]. However a new precipitation of clusters, which have a common direction with [112], occurs. They contain Al, Mg, Si and Cu coming from the matrix and the dissolved nanophases as well as Cr resulting from the partial dissolution of the Cr containing shell of dispersoids [3]. An in situ Au^{2+} (4MeV) irradiation at the JANNUS Orsay facility highlights that both clusters and β'' coexist. In order to understand the mechanism responsible for such evolutions, an in situ Au^{2+} ion irradiation of an untempered 6061 alloy (without β'' initially) has been performed. Dark field observation in the [112] orientation during irradiation shows that clusters precipitate from the solid solution. It is proposed that vacancy drag (like Si-V [4]) or interstitials dumbbell diffusion may play a key role in the irradiation induced precipitation of the new particles which contribute to an increase of the alloy's hardness.

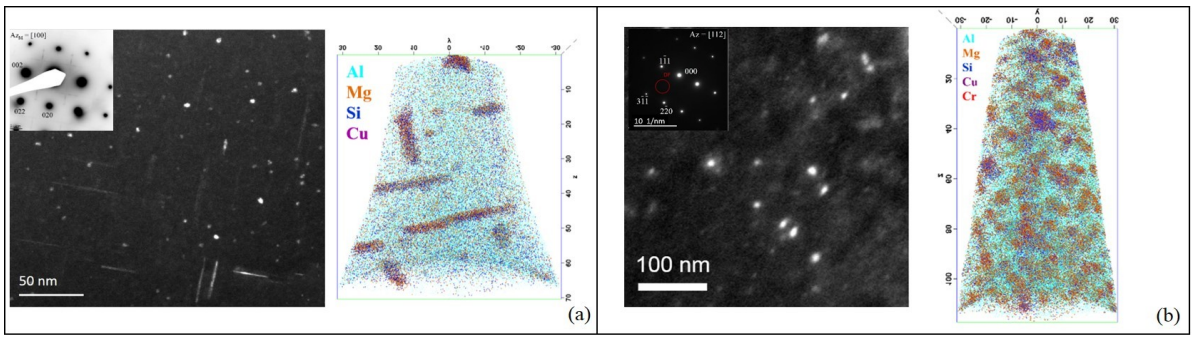

Figure 1: Dark field images and atom probe maps of the 6061-T6 Al alloy (a) before and (b) after high dose ion irradiation (165 dpa).

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THE EFFECT OF OXYGEN CONTENT ON MECHANICAL BEHAVIOR OF A TITANIUM ALLOY. EXPERIMENTS AND MODELING.

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Keywords: *titanium, oxidation, diffusion, oxygen, RFDA, high temperature, creep, finite element.*

Due to their high strength/weight ratio, titanium alloys are very used in components but high temperature oxidation is a key issue above 400°C. At high temperature, under air, two phenomena occur together: oxide scale formation on the surface but also oxygen ingress in the metal [1-4]. The mechanical properties of the oxygen enriched layer differ from those of the base metal. However, titanium alloys are attractive materials for technological improvement for the aerospace industry. It is why the studies of the effect of high temperature exposure on the mechanical behaviour of titanium alloy needs to be investigated and modelled. A considerable amount of work has been done on the effect of testing environment on the creep properties of near alpha and alpha-beta titanium alloys but limited works has been published regarding the effect of prior temperature exposure on the creep properties of Ti alloys.

The effects of oxygen content on the lattice parameters were analysed by X-ray diffraction using synchrotron radiation [5, 6]. Thin specimens with different thicknesses affected area have been tested using RFDA from room temperature up to 600°C. Creep tests have also been realized at 500 and 600°C.

These tests were carried out to quantify the influence of this oxygen ingress on the mechanical behavior. A model is proposed in order to take into account the effect of the local oxygen content in the material during creep. This approach is tested on creep tests realized on specific samples. Creep tests on specific samples were used to compare the proposed modelling and experimental results.

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EXPERIMENTAL AND THERMODYNAMIC ANALYSIS OF DIFFERENCES IN PHASE TRANSFORMATION OFβ-(Ni,Pt)Al COATING DURING ISOTHERMAL AND CYCLIC OXIDATION

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Keywords: *nickel aluminide coating, Ni-base superalloy, oxydation.*

β-(Ni,Pt)Al coatings are used for the protection of Ni-base superalloys against corrosion and oxidation during high temperature exposure. The protective properties of the coating are limited by its transformation into γ'-Ni_3Al phase. It was showed that β-(Ni,Pt)Al→γ'-Ni_3Al transformation occurred differently, as compared for isothermal and cyclic oxidation: while in isothermal conditions the transformation was characterized by a continuous β/γ' transformation front moving from the superallloy substrate towards the surface, the thermal cycling favoured γ'-Ni_3Al precipitation at β-(Ni,Pt)Al grain boundaries. It led to a different coating behaviour during further mechanical tests [1]. Nevertheless, the reasons for such a difference in β-(Ni,Pt)Al→γ'-Ni_3Al transformation in isothermal and cyclic oxidation conditions are not understood.

Since the transformation should be diffusion-controlled, it can be supposed that the diffusion in the β-(Ni,Pt)Al and/or γ'-Ni_3Al phases occurs differently, as compared for isothermal and cyclic conditions. It should result in different composition gradients for the same phase (β-(Ni,Pt)Al and/or γ'-Ni_3Al) for different oxidation conditions. Therefore, in this study a special attention was paid to the measurements of compositions of the phases constituting the coating deposited on Ni-base single-crystal alloy AM1. The oxidation was carried out for different durations in isothermal and cyclic conditions at 1100 °C.

Concentration measurements showed that both β-(Ni,Pt)Al and γ'-Ni_3Al phases became rapidly homogeneous during the oxidation. However, the compositions of β-(Ni,Pt)Al and γ'-Ni_3Al phases were found to be dependent on oxidation conditions. For example, for the shortest oxidation time (N_{min} hours and cycles; duration of each cycle was 1 hour with 45 min dwell at 1100 °C) Pt content in β phase was measured to be 1.3 at. % higher than that measured after the cyclic oxidation (Fig. 1). Further oxidation led to a decrease in this difference (difference in Pt content in β phase reduced to 0.5 at. %). The experimental results were analysed using Calphad approach and several explanations for the observed differences were proposed.

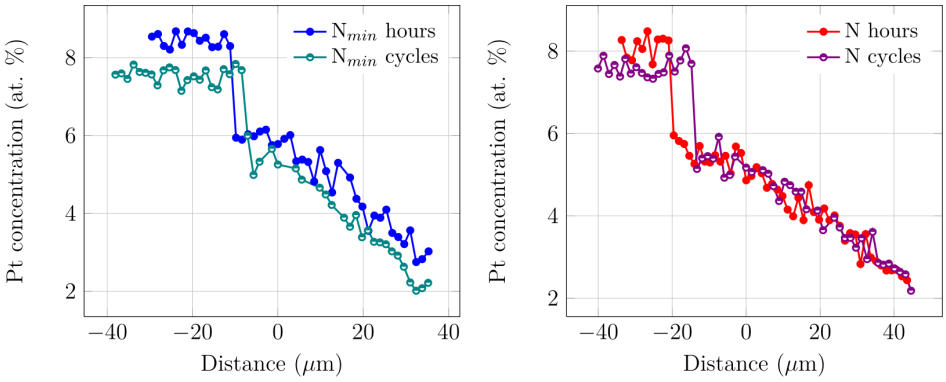

Figure 1: Example of Pt concentration profile measurement by EDS for isothermal and cyclic oxidation and different oxidation duration (Nmin < N): Pt-rich regions correspond to the β-(Ni,Pt)Al

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RELATIONSHIPS BETWEEN MICROSTRUCTURAL PARAMETERS AND TIME-DEPENDANT MECHANICAL PROPERTIES OF A NEW NICKEL-BASED SUPERALLOYS: AD730™

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Keywords: *AD730™, cast & wrought superalloys, high temperature, creep, dwell-fatigue.*

Meeting the higher and higher specifications of mechanical properties in turbine components of advanced aero engines requires the development of new alloys able to withstand these specifications.

AD730™ is a new nickel-based superalloy, designed by Aubert & Duval for turbine disks or seal rings applications. It has been developed to improve the current compromise between high temperature capability and moderate manufacturing cost [1], [2].

Aero engines rotating parts are subjected to several types of loading during service operation, from creep to fatigue, with any combinations of these mechanisms, at high temperature (> 650 °C).

The alloy's properties under these conditions are highly dependent on microstructural parameters, such as grain size, γ' size and distribution, and grain boundaries morphology.

The aim of this study is to evaluate the influence of those parameters on viscoplastic properties of AD730™ in a wide range of temperature (700-850°C). Hence, a fine grain, coarse grain and a single crystalline microstructures have been tested under creep and dwell-fatigue solicitations.

A special focus will be made in this paper on the influence of the applied stress in creep and dwell-fatigue loading conditions, revealing the significance of the unloading phases during dwell-fatigue tests: we can see in Figure below that a "negative plastic deformation" appears during the dwell-fatigue test (long hold time: 300s), slowing down the overall plastic strain rate.

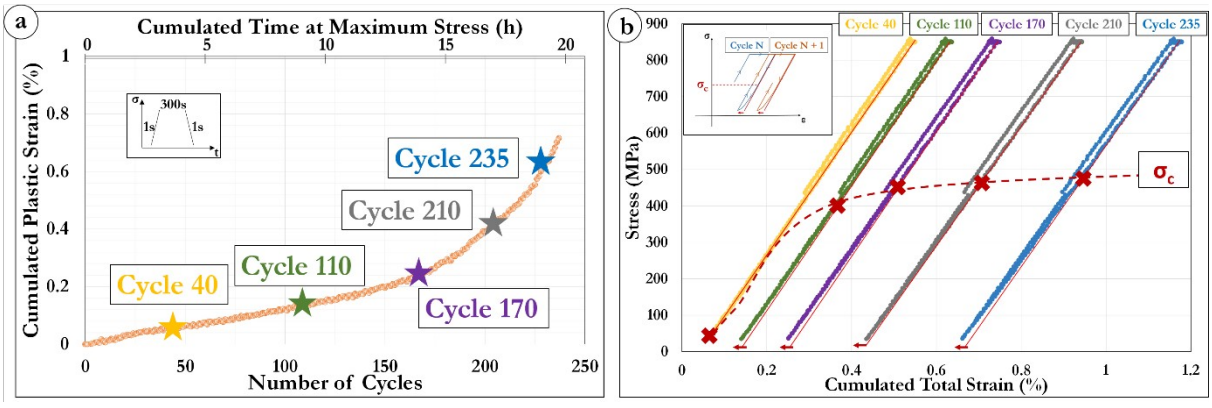

Figure 1: Dwell-fatigue curves from an interrupted test; Fine Grain microstructure; 700 °C - σ_{max} = 850 MPa - Δt = 300 s - R = 0.05; (a) Cumulated plastic strain evolution; (b) Hysteresis loops of several cycles during the test.

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IMPACT OF TEMPERATURE, STRESS MAGNITUDE AND CRYSTAL ORIENTATION ON THE PRIMARY CREEP RESPONSE AND LIFETIME OF NICKEL-BASED SINGLE-CRYSTAL SUPERALLOYS

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Keywords: *superalloys, creep, anisotropy.*

The temperature and stress dependence of the creep behavior of the nickel-based single-crystal superalloy CMSX-4 has been. Tests are performed at a temperature of 850°C and 1050°C along different crystal orientations: [011], [111], [102] and [112]. A strong creep behavior and creep life anisotropy is observed at 850°C, while the anisotropy tends to disappear at higher temperatures (Figure 1).

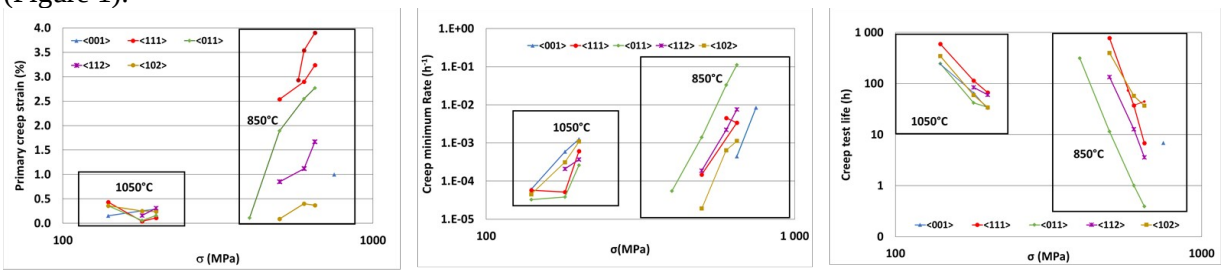

Figure 1: Results of isothermal creep tests. (a) creep minimum rate. (b) primary creep strain. (c) creep test life, w.r.t. orientation, stress level and temperature.

Differences observed at 850°C can be explained by a change in the deformation mechanism, while at 1050°C all specimens deform by {111}<011> slips in the matrix followed by the coarsening and rafting of the hardening γ' precipitates.

At 850°C, creep curves show that the mechanisms activated along the [011] and [112] loading directions are different from those activated when testing along [102] and [111] directions, especially during the primary creep stage. Macroscopic evidences of single slip deformation can be found in these specimens (section ovalisation, slip traces and crystallographic fracture surfaces), suggesting that the difference in creep response with orientation is mainly due to the activation of {111}<112> slips. In fact, in agreement with [1], when conditions are favorable for the activation of such slip systems, high and fast primary creep deformations is generated. For this reason, the directions characterized by this mode of deformation show shorter lifetimes and earlier tertiary creep stages. For a given crystal orientation and temperature, varying the applied stress magnitude shows that a threshold needs to be overcome to trigger such a single slip. This corresponds to the resolved shear stress required for {111}<112> slips' activation. In this presentation, a critical analysis of the deformation mechanisms controlling the creep anisotropy will be performed. In addition, an unusual deformation mechanism (i.e. deformation twinning) in this class of alloy will be detailed and analyzed. It will especially be shown how this deformation mechanism develops and how it controls the damage mechanism for crystal orientation favoring single slip.

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INFLUENCE OF SEPARATE Si AND C ADDITIONS ON MICROSTRUCTURE AND HIGH TEMPERATURE BEHAVIOUR OF THE TNM γ-TiAl ALLOY

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Keywords: *Intermetallics, heat treatment, phase transformation, microstructure, mechanical properties, creep.*

γ-TiAl alloys have been recently introduced as low Pressure Turbine blades into some new aircraft gas turbine engines. However, the currently used alloys are limited to application temperatures below 750°C. Among possible modifications of composition, minor alloying element, such as Si and C, show a significant interest to increase high temperature capabilities of this category of alloys [1, 2].

Small ingots of modified compositions based on the Ti-43.5Al-1Mo-4Nb-0.1B alloy (TNM), Ti-43.5Al-0.7Mo-4Nb-0.1B (TNM 0.7Mo), Ti-43.5Al-1Mo-4Nb-0.3Si-0.1B (TNM+0.3 Si) and Ti-43.5Al-0.7Mo-4Nb-0.2C-0.1B (TNM 0.7Mo+0.2C) were produced by vacuum arc melting to study the separate effects of Si, C and Mo on microstructure and mechanical properties. Ingots were HIPed at 1200°C and subjected to a heat treatment of 3h at 1220°C, followed by 6h at 900°C. Volume fraction of each phase constituent, tensile and creep behaviour have been investigated. Figure 1 highlights the relationships between microstructure of heat treated specimens and creep behaviour at 750°C under 195MPa.

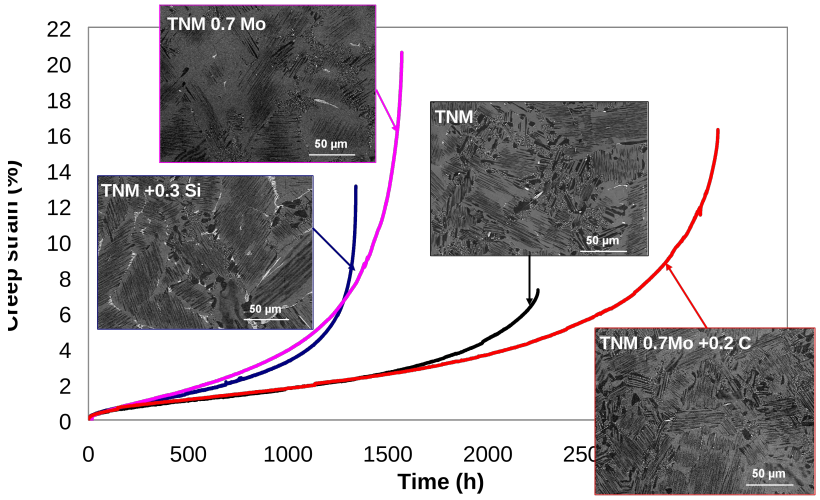

Figure 1: *Microstructure and creep behaviour at 750°C under 195MPa for the studied compositions*

Results indicate that silicon addition does not appear to be beneficial for both tensile and creep properties in β solidifying alloys such as the TNM. On the contrary, addition of carbon results in an increase of yield strength at room and high temperature, a reduction of the secondary creep rate and a longer creep life. It appears moreover that a solid solution effect of carbon is the predominating mechanism in this alloy for high temperature performance.

The research leading to these results has received funding from the European Union's Seventh Framework Programme under grant agreement N° 314366 (E-BREAK project).

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ION CHANNELING ORIENTATION DETERMINATION (ICHORD) : CRYSTALLINE ORIENTATION MAPPING WITHOUT EBSD

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Keywords: *Orientation maps, EBSD, channeling contrast.*

Crystalline orientation maps are usually obtained on a polycrystalline sample by using the Electron Back Scattered Diffraction technique (EBSD) in a Scanning Electron Microscope (SEM). These maps can be complicated to obtain with EBSD because of the special acquisition geometry required by the technique, pseudo-symmetry problems, a too small grain size or the need of large fields of view. We can then wonder if other means can be used to obtain such orientation maps, with a better compromise between spatial and angular resolution and acquisition/indexation speed, if possible.

Channeling contrast is a well-known phenomenon in scanning microscopy, explaining the fact that grains of different orientations appear with different grey levels on an image, as well as the variations of individual grey level for a given grain when the orientation of the sample is modified. The iCHORD method is based on the use of the channeling contrast to determine the crystalline orientation at a given position on the sample surface. An intensity profile is first obtained from a series of images acquired at different sample orientations. This experimental profile is then searched in a database of theoretical profiles. The three Euler angles associated to the closest theoretical profile are then assigned to this position on the orientation map.

Results presented here deal with a ferrite sample and a Ni superalloy sample with gamma prime precipitates. A comparison with EBSD results acquired on the same areas allowed us concluding that all the grains are indexed with an angular error inferior to 5°, for both sample. For EBSD as well as iCHORD, gamma prime precipitates in the Ni superalloy sample are not phase-separated, and are indexed as the Ni matrix. However, the dual acquisition of images using an InLens detector and a Secondary Ion detector for the superalloy sample allowed us separating both phases, with no scan distortions. Experimental parameters specific to the iCHORD method are discussed in this work, as well as their influence on the spatial resolution, the angular resolution and the speed of indexation.

Figure 1: iCHORD maps for (left) the ferrite sample and (right) the Ni superalloy sample.

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ATOM PROBE TOMOGRAPHY IN PHYSICAL METALLURGY: KEEP MOVING

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Over the past 15 years, Atom Probe Tomography has experienced two simultaneous revolutions that amazingly extended its application field. These two revolutions are the implementation of laser assisted field evaporation, opening the technique to low electrical conductivity, even to insulating, materials. The second revolution, which is going to be illustrated in this contribution, is related to site specific specimen preparation methods through correlative microscopy approaches.

Until the end of the last century, specimens for atom probe tomography had to be prepared using conventional electrochemical methods. Consequently, the features contained in the analyzed volumes (typically the final 1 to 200 nm of the tip itself) could not be selected a priori. When a specific region was to be analyzed, a tip had to be prepared, and carefully back polished until the region of interest was brought to the apex regions. Because of extensive transmission electron microscopy examinations, such specimen preparation methods were very time consuming, and the success rate lower than for conventional preparation methods. Site specific specimen preparation methods recently developed are based on lift out procedures using focused ion beam. Sites of interest are located using scanning electron microscopy, the imaging conditions depending on the nature of the site to be located. Lift outs are then welded on pre sharpened tips, and prepared as fine needles using annular milling procedures. The resulting specimen is therefore a tip suitable for atom probe analysis, containing the region of interest within less than 100 nanometers from the tip apex. Interestingly, such specimens did not experienced any electrochemical polishing, making them less prone to premature failure due to the applied electric field stress.

It is now possible to apply atom probe tomography, known for its ultimate spatial and analytical capabilities, to phases which are present in extremely low volume fraction (i.e. a fraction of a percent), and which, most of the time do not produce enough signal to be investigated with other classical techniques. It is also possible to analyze subsurface specimens, such as micron thick nitrided or cemented phases, as well as internal interfaces (such as grain or phase boundaries). When combined with other electron microscopy modes (EDS, EELS, EBSD, TKD….) it gives access not only the location of the boundary, but also its complete crystallographic and chemical nature.

The information provided by EBSD can also be used to prepare specimens with a selected crystalline orientation along their axis. Positioning a superstructure direction parallel to the tip axis allows systematic study of long range ordering, without the prior constrain of using specifically oriented single crystal. When low amplitude fluctuation concentrations are to be investigated (early stages of phase separation for example), specific analysis directions can be selected, limiting possible bias due to peculiar evaporation processes when a low index crystallographic direction is contained in the investigated volume.

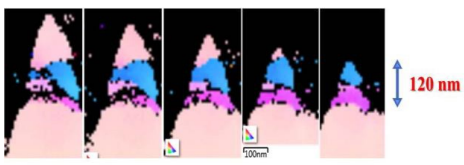

Fig. 1: TKD orientation map of an APT tip containing a ferrite-martensite interface in a model FeMnC alloy. Successive annular milling steps allows bringing the interface in the analysis region.

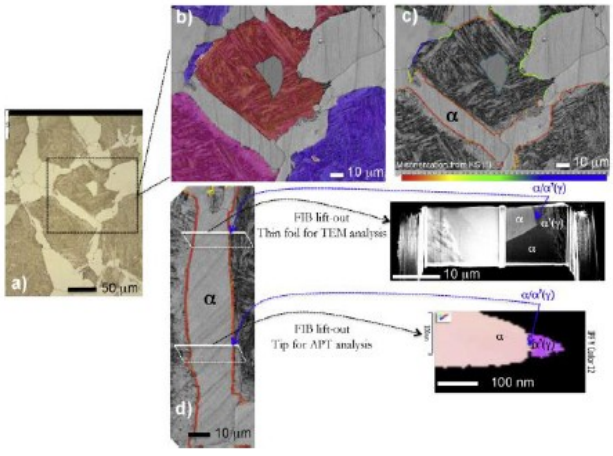

Fig. 2. Correlative microscopy investigation of a ferrite-martensite interface: a) Optical microscopy – b&c) phase identification by EBSD d) TEM and APT specimens

VISCOPLASTIC BEHAVIOUR OF THERMALLY GROWN OXIDE OBTAINED BY SYNCHROTRON RADIATION DIFFRACTION

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Keywords: *high temperature oxidation, chromia formers, stress relaxation phenomena, synchrotron radiation*

This study takes place in the general context of a better understanding of materials degradation mechanisms in extreme environments. In particular, the aim of the present work was to correlate microstructural elements to stress release mechanisms for thermally grown chromia thin films on NiCr alloys. Thus, it was planned to undertake X-ray diffraction measurements in situ during oxidation of the metallic alloys. A 2D detector large enough was used to get sufficient Psi values, to extract the stresses evolution in the chromia films during the course of isothermal oxidation, and also during short plateau just after low temperature jumps (supposed to impose an additional thermal strain).
The main results obtained were the study of the stress release mechanism after the low-temperature jumps. Experiments were carried out in Synchrotron at the ESRF BM02 line. From the intensity and the integral width an accurate description of the microstructure was done. In particular, it was clearly shown that the ceramic grains mainly growth during the very initial oxidation period, while the development of the chromia film is continuous during all the first oxidation plateau of 3 hours. Finally, four chromia ceramic film microstructures have been built at respectively 800, 850, 900 and 1000°C, with corresponding increasing grain sizes (0.2 to 0.8 µm) and film thicknesses (500 nm to 3 µm). After building the microstructures, low temperature jumps were applied, and the internal stress determination was also undertaken from the analyses of the images providing from the 2D detector. An example of the later experiment is shown in Fig.1 for the microstructure initially built at 1000°C. This methodology has been reproduced for the 4 initial microstructures, and from a confrontation with modelling [1], the creep coefficients have been identified.

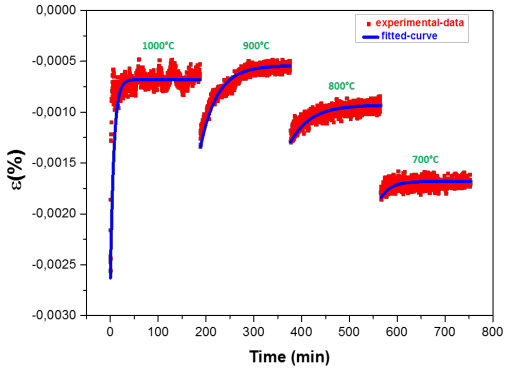

Fig. 1 : Internal strain evolution as a function of the oxidation time, for four subsequent plateaus of 3 hours, at respectively 1000, 900, 800 and 700 °C.

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CORRELATIVE INVESTIGATION OF BORON SEGREGATION AT PRIOR AUSTENITE GRAIN BOUNDARIES USING ATOM PROBE TOMOGRAPHY

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Keywords: *steel, boron, segregation, PAGB, APT, NanoSIMS, EBSD, TKD, FIB*

The advantageous “boron effect” on steel hardenability has been highlighted since the 1920s. Added in small amount, a few tens of ppm by weight, boron addition greatly increases the hardenability of steel. Hardenability refers to the ability of a steel to harden upon quenching, and is related to the range of cooling rate leading to the formation of martensite, the hardest constituent in steel. Using boron addition, martensite, can be typically be obtained for less drastic cooling conditions (or at a greated depth in a component, given a cooling condition).

However, a number of studies have shown that boron effect depends on many factors and may produce the opposite effect of that desired, i.e. reduce the hardenability [1]. This means that boron addition only in steel is not sufficient to get boron effect, and that it is essential to understand the boron action in order to optimize its effect.

Initial studies in this field have shown the importance of boron segregation on hardenability. The commonly accepted scenario is that boron has a strong tendency to segregate to prior austenite grain boundaries (PAGB) [2]. To study this segregation and to quantify the boron level at grain boundaries, these austenitic interfaces must be analyzed down to the atomic scale by Atom Probe Tomography (APT). Analyzing such interface is however rather tricky, because of the large size of grains (30-100µm) compared to the analyzed volume (50x50x200 nm3). In order to do this, several highly technical methods should be employed alternately in an original protocol published soon. The GPM laboratory is one of the few laboratories in world with a fleet of machines allowing this combination of techniques. Because it is necessary to locate the PAGB in the room temperature martensite structure we use NanoSIMS and EBSD (Electron Backscattered Diffraction) associate to crystallographic reconstruction with MERENGUE 2 code [3]. Moreover, APT experiments require the preparation of samples in the form of sharply pointed needles with few nanometers of apex radius. To be analyzed by APT, the PAGB has to be located in the first 50nm of the tip apex thanks to lift-out by FIB (Focus Ion Beam) and TKD (Transmission Kikuchi Diffraction).

Analysis of PAGB was successful thanks to this procedure, and gave access to quantitative information related to boron segregation: depth of the segregated zone, level of boron and other elements, etc. These information are used to investigate the nature of segregation mechanism, i.e. equilibrium or non-equilibrium segregation which can happen at the same time for this phenomenon.

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COMPARISON BETWEEN DIFFRACTION CONTRAST TOMOGRAPHY AND HIGH-ENERGY DIFFRACTION MICROSCOPY

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Keywords: *three-dimensional X-ray diffraction; DCT; HEDM.*

The grain structure of an Al–0.3 wt.%Mn alloy deformed to 1% strain was reconstructed using diffraction contrast tomography (DCT) and high-energy diffraction microscopy (HEDM). 14 equally spaced HEDM layers were acquired and their exact location within the DCT volume was determined using a generic algorithm minimizing a function of the local disorientations between the two data sets. The microstructures were then compared in terms of the mean crystal orientations and shapes of the grains. The comparison shows that DCT can detect subgrain boundaries with disorientations as low as 1° and that HEDM and DCT grain boundaries are on average 4 µm apart from each other. The results are important for studies targeting the determination of crystallographic orientation and volume of bulk grains. For the case of a polycrystal with an average grain size of about 100 µm, a relative deviation of about 10% was found between grain volumes determined with the two techniques. The average disorientation is small of about 0.4°.

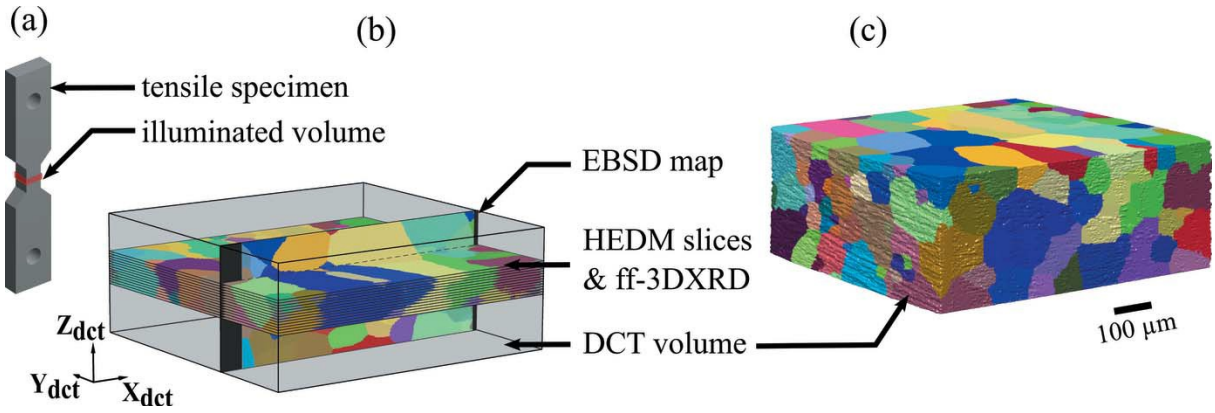

Figure 1: Sample and grain structure imaged with DCT, HEDM and EBSD. (a) The dog-bone shaped specimen, with a height of 14 mm and a gauge length of 1.5 mm. The 350 mm-high illuminated volume is shown in red. (b) The positions of the HEDM and EBSD slices in the DCT volume. Black margins on the EBSD map indicate regions removed by electro-polishing. (c) The DCT volume, which is about 350 mm high. Colour coding is according to the components of the Rodriguez vector (according to Fig. 1 of Ref. [1]).

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4D X-RAY TOMOGRAPHY: A BRIEF HISTORY, APPLICATIONS AND RECENT DEVELOPMENT

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Keywords: *in situ, 4D tomography, hot tearing, high temperature damage, solidification*

X-ray tomography is becoming a standard characterisation in a numerous materials science research field : room or high temperature damage in composites of metals, solidification, sintering …[1] More than 3D images it is possible since several years to perform 4D investigation providing dedicated experimental set up are developed. Since more than 20 years now in situ tomography (4D tomography) has been considerably developed in term of spatial resolution and acquisition time thanks to technological progress. We will present briefly these evolution and the main application that were associated with a focus on some of them. Finally we will present the recent evolution in laboratory tomograph but also concerning in situ nano-tomography.

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PRECIPITATION IN AEROSPACE ALLOYS BY SMALL-ANGLE SCATTERING: FROM KINETICS TO MICROSTRUCTURE MAPPING

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Keywords: *precipitation, small-angle scattering, Al alloys.*

Structural hardening is one of the key tools for alloy design. Classical examples include many aluminium alloys, the latest generations of which are widely used in aerospace industry. A key feature of strengthening precipitates is that, no matter the system, their lengthscale is always of the order of the nanometer. Characterizing these objects is a challenge, and usually implies very localized measurements and small probed volumes.

Small-angle scattering (of X-rays, SAXS, or neutrons, SANS), on the other hand, can characterize the size and volume fraction of these small precipitates on representative volumes (~100µm^3) [1]. The experimental setup is simple enough to allow for a wide range of sample environments for in situ measurements. By scanning the sample with the beam, heterogeneous microstructures (such as in welded structures) can be mapped [2,3]. Of course, this type of experiments in complex systems involves sophisticated methodological developments [4,5].

This talk will illustrate the possibilities of the technique by taking the example of aerospace aluminium alloys (third generation of Al-Li-Cu). In particular, we will show that it is possible to study in situ the complex precipitation trajectories taking place in these multiconstituent alloys, to map heterogeneous microstructures, and to relate local instantaneous precipitate states to mechanical properties of the alloys.

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IN SITU INVESTIGATIONS OF PARTITIONING MECHANISMS IN Q&P STEELS BY SYNCHROTRON DIFFRACTION EXPERIMENTS

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Keywords: *synchrotron, in situ, martensite, austenite, carbon, diffusion, internal stresses*

Q&P (Quenching & Partitioning) is a new annealing route proposed to produce 3rd generation Advanced High Strength Steels. Q&P annealing cycle consists in an interrupted quenching from austenitic soaking to reach a partial martensitic transformation followed by rapid reheating and isothermal holding. During this second step, it appears that carbon can diffuse out from martensite into retained austenite. In common steels, this process is largely inhibited and prevented by carbide precipitation (the so-called tempering process). A judicious choice of alloying elements (Si, Al) and reheating temperatures permits to reduce transition carbides and even cementite precipitation. As a result, retained austenite can be enriched in carbon and can be stable at room temperature. It is the so-called partitioning process. The cycle ends with a cooling sequence during which a certain amount of martensite can form again. Q&P microstructures are thus complex, resulting from the sequence and conjunction of different phenomena (two martensitic transformations, carbon segregation/precipitation/partitioning, possible bainitic transformations …) which are still intensively discussed in the recent literature. The respective kinetics of these mechanisms controls the final microstructures and thus are of prime interest for final properties (TRIP effect).

The Q&P treatment on high C TRIP steel (Fe-0.3C-2.5 Mn-1.5Si-0.8Cr in wt.%) has been studied by the means of *in situ* X-Ray diffraction experiments using a synchrotron source. The experiments have been carried out on the ESRF ID15B line in the ETMT Instron device (powder diffraction configuration in transmission). The high energy monochromatic beam (E = 87 keV) enables high acquisition rates (10 Hz) adapted to study 'real time' processes on bulk samples and for the first time during first reheating and cooling sequences.

The main findings of our study are the following:

- Absence of microstructure evolution during reheating (at about 30°C/s) after first quenching step (below 370°C for the studied steels),
- Significant increase in ferritic phase fraction during partitioning step. In the literature, this evolution is interpreted as a bainitic transformation, as the mobility of martensite/austenite interface during partitioning can be ruled out,
- Evidence of heterogeneous carbon distribution in austenite at the beginning of partitioning step. Carbon enrichment in austenite and bainitic transformations are strongly related.
- High residual stresses in retained austenite at RT which are favorable to a TRIP effect due to eigenstrains resulting from the differences of thermal expansions between austenite and martensitic phases during final cooling,
- Local carbon mass balances permitted by in situ experiments show that a large fraction of carbon remains trapped in martensite laths (segregations on dislocations, carbides). Unfortunately, our experiments have not yet permitted to identify the nature of carbides and to quantify their relative fractions.

This work was supported by the French ANR through the project CAPNANO (ANR-14-CE07-0029). The synchrotron experiments have been realized in December 2014 under the grant MA2305 at the ESRF in Grenoble which is fully acknowledged. The authors would like also to thank the LABEX DAMAS (ANR-11-LABX-0008-01) from Lorraine for their supports.

LATTICE ROTATIONS AND INTERNAL STRESS ANALYSIS IN INDIVIDUAL GRAINS DURING A CYCLIC STRESS-INDUCED MARTENSITIC TRANSFORMATION IN A CUALBE POLYCRISTALLINE SHAPE MEMORY ALLOY

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Keywords: *stress-induced martensitic transformation, lattice rotation, internal stress, synchrotron 3-D X-ray diffraction.*

For better understanding the behaviors related to the stress-induced martensitic transformation, focus has recently been on the study of the relationship between the internal stress and the grains rotation. The bulk properties of Shape Memory Alloys (SMA) such as the macroscopic stress-strain and the superelasticity strongly depend on the reorientation of the embedded grains and their interactions within the polycrystal. It is therefore necessary to consider different scales in a polycrystal such as CuAlBe, instead of oversimplifying it to a single crystal case. At grain volume scale, Three-Dimensional X-Ray Diffraction (3DXRD) experiments on four grains of $1mm^3$ have allowed to carry out influence of the Schmid factor on the crystal rotations [1].

In this work, *in-situ* 3DXRD technique were used to study about hundred grains of 10^{-3} mm^3 (figure 1a) during stress-induced martensitic transformation in the Cu-11.6%Al-0.6%Be(wt.). 3DXRD was performed on the ESRF ID11 beamline and the resulting data were analyzed using the software ImageD11 [2] in such a way several information was obtained for each grain.

The improvements made on the software to process a large number of micron grain lead to more efficient quantification of intra/inter granular rotations (figure 1b), internal strains (figure 1c) and stresses. A different behavior between grains in surface and in volume was pointed out.

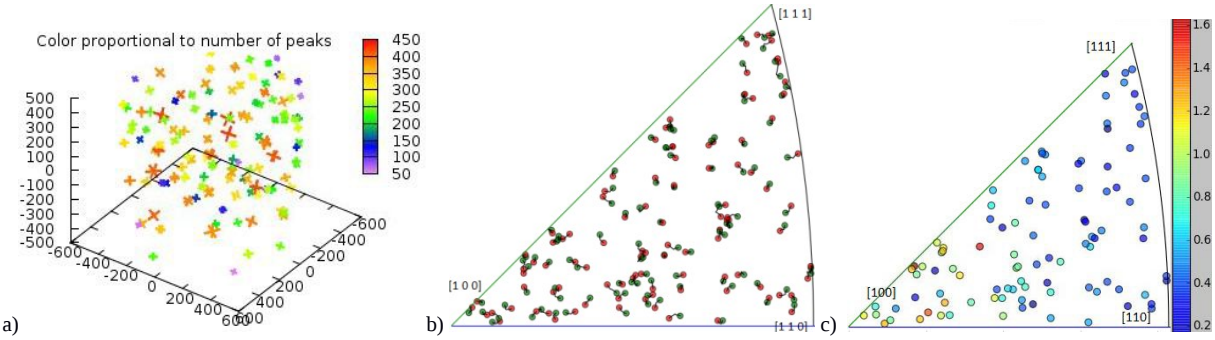

Figure 1: a) Positions of the hundred grains in the analysis volume, b) Inverse pole figure in the tensile direction (TD) of the grain rotation during the tensile test, c) Strain of the grains in the TD at the maximum applied stress.

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STUDY OF FATIGUE MECHANISMS IN ALUMINIUM ALLOYS USING SYNCHROTRON TOMOGRAPHY

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Keywords: *aluminum alloys, fatigue, X ray tomography.*

The selection procedure of aluminium alloys for aerospace components takes into account fatigue properties, which involve crack initiation and growth [1]. In this study, fatigue crack initiation mechanisms (crack sizes below 500 µm) near intermetallic particles are analyzed in alloys 2050-T8 and 7050-T7451.

First, the particles initiating fatigue cracks (Al7Cu2Fe, Mg2Si and Al-Cu-Fe-Mn) are observed in two-dimensions (Scanning Electron Microscope) and in three-dimensions (3D - nanotomography). A voxel size of 50 nm was used at the European Synchrotron Radiation Facility (ESRF) beamline ID22, in order to observe particles in 3D: inside the material and before any fatigue loading, some cracked particles can be detected [2, 3].

The resistance to crack initiation was analyzed through three effects: (i) large particles are more likely to initiate a matrix crack, (ii) particles (Al7Cu2Fe, Mg2Si, Al-Cu-Fe-Mn) do not have the same resistance towards matrix crack initiation – resistance is correlated with the number of cracked particles [3], (iii) the effect of crystallography is second-order.

Second, several samples are cyclically loaded and observed in situ in 3D at beamline ID19. A dedicated automated procedure to detect the crack front shapes is developed in this study. It is used to quantify – at the surface and below – the crack growth rate variations related to crack deviation at the grain scale. A total of 22 cracks in 2050 and 7050 specimen are observed in situ and ex situ [4].

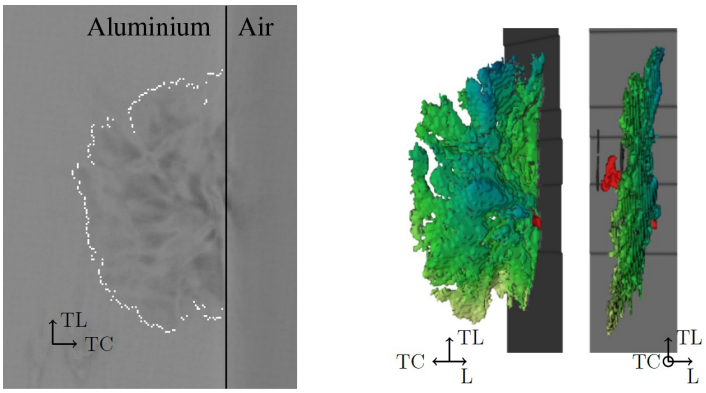

Figure 1: 80 µm diameter fatigue crack after 26 000 cycles (a) observed by tomography (z- projection of grey-level), with crack front detection – white dots (b) visualized after image analysis. Alloy 2050. [4]

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SINGLE AND DUAL-BEAM IN-SITU TEM ION IRRADIATION OF PURE IRON AND FE-CR ALLOYS USING THE JANNUS FACILITY

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Keywords: *JANNuS platform, TEM, Irradiation.*

High-purity iron and Fe–Cr model alloys are of particular interest, as ferritic–martensitic steels are candidate materials for use as structural materials in future fusion reactors. Damage displacement cascades and helium production by transmutation reactions may induce strong embrittlement and swelling.

In order to predict in-service properties of such materials in the various radiative environments, the microstructural evolution under irradiation of model materials (pure Fe and Fe-Cr alloys) using the JANNuS (Joint Accelerators for Nano-science and Nuclear Simulation) platform is studied. Such platform is designed to supply a large range of ion irradiation and implantation conditions, allowing notably in-situ Transmission Electron Microscopy (TEM). Such a facility will play an essential role for multi-scale modelling of irradiation effects in materials. Such work is realized within the European Fusion Development Agreement (EFDA) program.

To understand the evolution of materials under irradiation conditions similar to those in future fusion reactors, high purity iron and Fe-Cr alloys (fabricated at “Ecole des Mines de Saint-Etienne”) are irradiated in-situ (JANNuS Orsay platform) at 500°C in a TEM with dual-beam (1MeV Fe+ ions while simultaneously implanting 15keV He+ ions) and single beam (15keV He+ ions) conditions.

The detailed analysis of the evolution of the material under irradiation at different fluences, notably using sequential irradiation (Figures 1a-1b), reveal that dislocation loops and helium bubbles are present with a heterogeneous bubble formation on dislocation loops: a majority of them are observed inside large dislocation loops. A mechanism explaining the heterogeneous nucleation of helium bubbles within dislocation loops is proposed (Figure 1c).

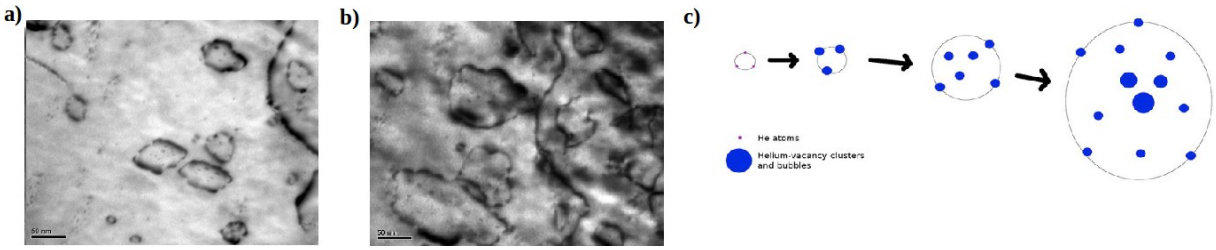

Figure 1: Bright field TEM images: Bubbles and dislocation loops in α-Fe irradiated at 500°C to 1100 appm He on image a) g=[110] (s>>0), positive defocus and later on image b) at 2000appm g=[110] (s>>0),positive defocus. Image c): proposed mechanism for the formation of helium bubbles inside large loops: 1) He is trapped at the dislocation loop cores, 2) Formation of He-V clusters, 3) Growth of loops. 4) Formation of cavities/bubbles.

CHARACTERIZATIONS WITH THE MARS BEAMLINE (SYNCHROTRON SOLEIL) OF MATERIALS IRRADIATED IN NUCLEAR REACTORS

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Keywords: *Synchrotron facility, neutron irradiation, Zr based alloys and ODS*

MARS (Multi-Analyses on Radioactive Samples) is the X-ray bending magnet beamline of the French synchrotron facility SOLEIL, dedicated to the study of radioactive matter. The MARS beamline aims at extending the possibilities of synchrotron based X-ray characterizations towards a variety of radioactive elements (α, β, γ and n emitters). Thus, its specific and innovative infrastructure has been optimized to carry out analyses on radioactive materials with activities up to 18.5 GBq per sample. This beamline, which has been built thanks to a close partnership and support by the CEA, has been designed to provide X-rays in the energy range of 3.5 keV to 35 keV.
Three main techniques are progressively proposed on MARS beamline: transmission and high-resolution powder X-Ray Diffraction, X-ray Absorption Spectroscopy and X-Ray Fluorescence [1].
This presentation deals with recent results obtained on the MARS beamline, thanks to very powerful and useful improvements brought to the experimental set-up of the beamline (figure 1) and to various materials irradiated in nuclear reactors: Oxide dispersion-strengthened (ODS) steels at high doses and also Zr based alloys irradiated in Pressurized Water Reactors up to 7 PWR cycles [2].
Synchrotron radiation analyses bring original results concerning nanosized secondary phases: for example, concerning Zr based alloys, radiation-enhanced precipitation of β-Nb precipitates has been evidence and their crystallographic structure (lattice parameter and Nb content) is reported for the first time, whereas for irradiated ODS, experiences using XAS and XRD provide complementary data.

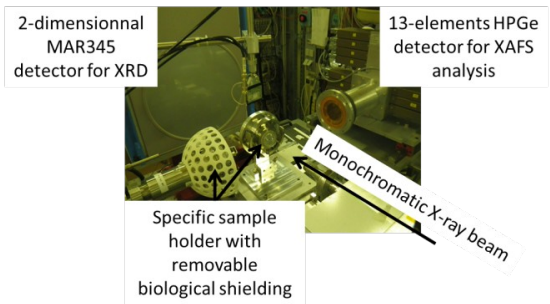

Figure 1: Experimental set-up at the MARS beamline, XRD and XAS for highly irradiated samples.

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DEFORMATION TWINNING IN AN α'' MARTENSITIC TITANIUM SHAPE MEMORY ALLOY

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Keywords: *EBSD; twinning; titanium.*

Metastable beta titanium alloys have been a wide field interest in the last decades due to the possibility to obtain Ni-free superelastic alloys using fully biocompatible elements only. In these alloys, the amount of β-stabilizing elements (such as Nb, Ta, Mo…) is sufficient to retain beta phase or α" orthorhombic martensite at room temperature by quenching from high temperature. Deformation twinning of a full-α" titanium-based shape memory alloy is characterized using Scanning Electron Microscope Electron BackScattering Diffraction (SEM-EBSD) and Transmission Electron Microscope (TEM). α'' is deformed using reorientation and plastic twinning. The selfaccommodating α'' microstructure is shown to be first deformed using reorientation twinning. Plastic deformation occurs further by plastic twinning.

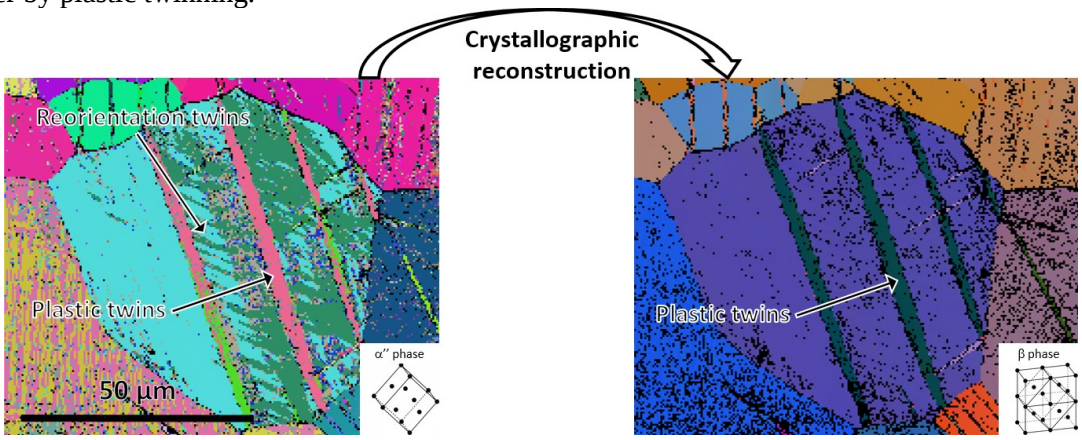

Figure 1: EBSD maps of Ti–25Ta–20Nb alloy strained until 6%: Euler angles obtained from indexation with the α'' phase and from indexation with the β phase.

As shown on Figure 1, crystallographic reconstruction using direct indexation of electron backscattering patterns (EBSPs) [1] is used to make a distinction between reorientation twinning which preserves β orientation from plastic twinning which modifies β orientation. A new plastic twinning system is observed in α'' phase which corresponds to the specific $\{332\}<113>_{\beta}$ twinning system of metastable beta titanium alloys. Maximum lattice deformation calculation is a relevant parameter to predict the variant of martensite that is favored during the reorientation process. Conversely, Schmid factor analysis can be used to predict the selection of plastic twinning variants during deformation. Analogy between α'' and β plastic twinning systems is highlighted. Detailed results can be found in the following reference [2].

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MICROSTRUCTURE TAILORING AND SURFACE FUNCTIONALIZATION OF AN MG-2WT.%CA ALLOY FOR MEDICAL APPLICATIONS AS DEGRADABLE IMPLANTS

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Keywords: *Magnesium, calcium, degradation, mechanical properties, microstructure, functionnalization.*

Degradable implants for bone fixation have been of significant interest since the last decade. Among different materials, Mg-Ca alloys appear as promising candidates due to their unique combination of properties: well tolerated by the body [1], natural tendency for degradation [2] and in addition, a low elastic modulus which can help to reduce stress-shielding effect during bone healing [3]. Due to the variable requirements for degradable implants, it is necessary to know how to tune the mechanical properties as well as the degradability kinetics. In this work a bulk and surface processing approach is proposed: i.e. tailoring the bulk microstructure by thermomechanical treatments and surface functionalization by additive manufacturing.

Extrusion and equal channel angular pressing (ECAP) have been used for bulk processing in order to modify the microstructure of the material: a Mg-2wt.%Ca alloy. It is found that the severe plastic deformation induced by the ECAP process produces the finest grain microstructure and second phase particle distribution. While different microstructural features (dislocations, twins, grain size) can account for the improvement in the mechanical strength, characterization shows that the improvement in the corrosion resistance appears as primarily affected by grain size and second phase microstructure. This influence on the corrosion resistance results from the dispersion of the second phase Mg_2Ca and possibly a more stable oxide layer.

Surface modification is achieved by designing a surface patterning method that uses silver nanoparticle microdeposition to functionalize the material. Indeed, silver is well known for its antibacterial properties [4]. The deposition is followed by a local heat treatment done by a laser sintering process. A series of depositions have been performed to achieve the desired deposition conditions and a reproducible deposition line of 20 µm width and between few hundreds of nanometres and one micrometre thick.

Using this bulk and surface processing approach has permitted to obtain a functionalized alloy of magnesium-calcium with enhanced properties which can be further considered for biodegradable applications.

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MICROSTRUCTURE AND MECHANICAL BEHAVIOR OF EQUIATOMIC CoCuFeMnNi HIGH-ENTROPY ALLOY

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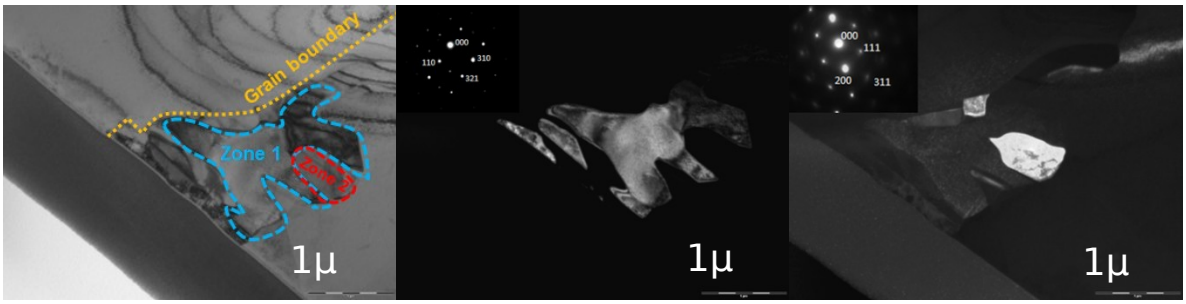
Keywords: *high-entropy alloy, alloy design, eutectic transformation, compressive properties*

High-entropy alloys (HEAs) are a group of recently (2004) discovered materials which draw a big attention of materials scientists, due to their high strength, ductility and high temperature stability [1]. HEAs are always multicomponent (at least 5 chemical elements), what can be true also for conventional alloys, but here all the components are in almost equiatomic proportions [2]. In this case, configurational entropy of the system reaches its maximum, what has a strong effect on many factors, e.g. lattice strain and constituent phases. What is characteristic for HEAs is that they form single-phased structure, despite different crystal structures of elements.

A novel equiatomic CoCuFeMnNi high-entropy alloy was investigated from the structural and mechanical point of view. This composition was considered as a modification of well-known CoCrFeMnNi HEA, where chromium was replaced by copper. According to ThermoCalc calculations, chosen composition should lead to a stable, FCC single-phase structure in a large range of temperatures. The alloy was cold crucible casted (350 g ingot) and investigated in annealed conditions. In compression tests below 600 °C, studied material shows a promising yield strength value of about 400 MPa and it is deformed without cracking. Above 600 °C, a disastrous brittleness is observed in deformation conditions. Microstructural characterisation makes obvious a presence at this temperature of a eutectic transformation, leading to an intergranular liquefaction. TEM analysis let us identify the eutectic phases: FCC rich in Ni, Cu and Mn, and BCC phase enriched mainly in Fe and Co (Figure 1).



(a) (b) (c)

Figure 1: (a) TEM bright field image of phases, (b-c) dark field images with diffraction patterns, (b) BCC, AZ = [001], (c) FCC, AZ = [0-11].

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DEVELOPMENT OF A NEW STEEL TUBE FOR OIL AND GAS

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Keywords: *corrosion resistant alloy, stainless steel, design.*

Corrosion resistant alloys (CRA) such as supermartensitic and duplex stainless steels are regularly used in the Oil&Gas industry in CO_2+H_2S environments where high chloride content and elevated temperature are present.
The design of a new CRA steel that has to meet both customers' requirements (mechanical properties, corrosion resistance, price) and feasibility constraints linked to the manufacturing facilities (Mannesmann rolling process, achievement of the mechanical properties by quenching and tempering heat treatment) is a challenge.
In this presentation, the development of such steel is addressed through the methodology followed during the design and the industrialization of seamless tubular products.

The design started with an ideation process that defines independent concepts. The translation of the technical specification into chemistry and microstructure features, when possible, was used to select the concepts and turn them out into candidates. At this stage, models and computer simulation tools were used.
Material of the selected candidates was produced at the lab scale (small heats), processed (forging / rolling and heat treating) and characterized. Chemistry and process variations of the most promising candidates were then defined for a new design loop until. The learning about the effect of the alloying elements and process parameters on the microstructure and final properties along the iteration loops was used to improve the models and simulation tools and ensures convergence towards valid final candidates for industrialization.
The characterization consisted in forgeability tests, mechanical testing (tensile and impact toughness), microstructure (precipitation, phases balance), dilatometry, corrosion tests (Sulfide Stress Cracking tests at room temperature and mass loss tests at elevated temperature).
At some stage in the design iterations, the hot forming was performed on a pilot piercer to be as close as possible to the industrial process since it was suspected that material properties would be significantly influenced by the hot forming process.

Finally, industrialization of one of the final candidates was launched after a comprehensive risk analysis was performed and risk mitigated as much as possible by tests on lab material. During the industrialization, variations of process parameters that could not be fully investigated during the lab stage were tested.

RECENT DEVELOPMENTS IN THIRD GENERATION ADVANCED HIGH STRENGTH STEELS FOR AUTOMOTIVE

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Keywords: *AHSS, Automotive, TRIP, TWIP, Hot Forming, High formability*

The Steel industry is closely related to the transport sector and steel is indeed the most used structural material in automotive sector. Improvement of passive safety together with lightweightening requirements due to environmental regulation make this market particularly challenging, with new demands in terms of material properties which strongly pull the steel sector in the direction of developing new steel products and new steel solutions.

This paper presents recent evolutions in steel products development for Automotive. Advanced High Strength Steels (AHSS) are now commonly used but the increasing need for safety and weight reduction has led to the development of a new generation of AHSS. Based on an appropriate microstructure design, this new generation provides 10% to 20% weight reduction compared to regular AHSS, while keeping the manufacturing properties at the car makers and allowing to warranty the same safety properties on final vehicles. This goes along with a necessary increase of yield stress and tensile strength properties of the material, that mainly control final crash ability, together with a formability comparable to that of reference steel grades currently used.

When cold formed constituents are targeted, one important possibility is to introduce austenite as a key component in the microstructure, therefore allowing to increase the tensile strength through other microstructural constituents (martensite or bainite) while keeping an equivalent or better formability than the reference materials through the TRansformation Induced Plasticity (TRIP) effect. Several examples will be given, including Q&P (Quenched and Partitioning), CFB (Carbide Free Bainite) based microstructures. Twinning Induced Plasticity (TWIP) effect is also considered to design microstructures and further push the lightening potential.

Another route consists in forming the material at high temperature to take advantage of a favorable rheology, and then quench the structure after forming operation, allowing microstructural control through austenite decomposition under deformation. These hot-formed (or hot stamped) grades constitute another family of new steel products to provide intense weight reduction and safety to the automotive market. We will illustrate recent trends in hot stamping development : increase of tensile strength through the development of martensite with enhanced properties, or increase in the crash worthiness of the final formed material through microstructural design to allow the usage as energy absorption parts.

NEW STEPS TOWARD STEEL SEMI-SOLID FORGED PARTS IN MASS PRODUCTION

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Keywords: *Semi-solid, Steel, Forming.*

The forging of steel parts at semi-solid state aims to reduce the consumed energy and time comparing to classical forging processes. This technology is particularly promising in the field of automotive components manufacturing. Possible substantial gains are expected on forming loads, on the surface quality, on the overall energy balance as well as on the capacity to reach complex geometries.

The road to mass production of components involves to take process specificities into account from design phase to manufacturing control and assessment of life expectancy.

- Product design method is developed to integrate specific process rules in order to define part acceptable geometry with respect to lightweight expectation.
- Micro-Macro physical model as an evolution of past research work considers now poro-elastic behavior, solid fraction influence, and tensile or compressive load dissymmetry.
- Extended experimental campaigns on simple parts (see Figure 1) allowed to consider geometry distribution on large batches, determination of process capability, defects identification and metallurgical control.
- Actions around manufacturing parameters optimization enabled so far to define influence of billet heating cycle, lubrication protocol, and measures to prevent part oxidation.
- Application case studies on engine and power train parts are treated to apply global methodology, to test cooling strategies, to assess repeatability as well to control metallurgical and mechanical properties of products. Feasibility assessment in general.

All the previous depicted aspects are developed in a large research project in association with academic and industrial partners during 4 years. Examples are selected to show potentialities od semi-solid forming of steels in terms of geometries, weight reduction possibilities, such as manufacturing route optimization

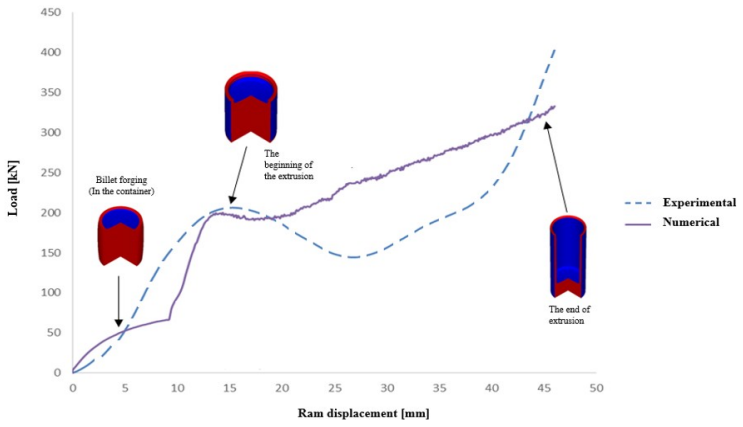

Figure 1: Reverse extrusion of “U shape” parts: Experiment Load versus Numerical Load

Associated Partners

Research are conducted thanks to french PIA program and partners ASCO INDUSTRIES, ATELIERS DES JANVES, SA, FORGES DE COURCELLES, SAS, ECOLE NATIONALE SUPERIEURE D’ARTS ET METIERS, RENAULT SAS, PEUGEOT CITROËN AUTOMOBILES SA

DETERMINATION OF PHASE EQUILIBRIA IN THE FE-C-MN-AL ALLOYS FOR DUPLEX STEELS 3RD GENERATION

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Keywords : *phase equilibria, Fe-C-Mn-Al alloys.*

To contribute to CO2 emissions reduction, a 20 % lightening of automotive weight must be achieved. One of the most promising solutions is the development of a 3rd generation of Fe-Mn-Al-C alloys with duplex structure. These steels possess high-strength and high formability. As a first step, the knowledge of the corresponding quaternary phase diagram serves as a roadmap in the choice of the compositions and the optimization of the elaboration conditions. A thermodynamic database, reliable and precise, is therefore required. However, the literature data on the quaternary system Fe-C-Mn-Al, in the considered domains of composition, are limited.

The objective is to obtain an accurate characterization of the phases in equilibrium, typically ferrite (α), austenite (γ) and carbide $(Fe,Mn)_3AlC$ (κ), their compositions and proportions using an approach coupled by specific experiences and by thermodynamic calculations. To determine the annealing duration for the establishment of the thermodynamic equilibrium at the investigated temperature, the development of phase composition is studied for Fe-0.2C-5Mn-2Al (mass%) at 800°C between 2 min and 4 months. A kinetic model (DICTRA) is proposed to support the experimental evolution of phase fractions and compositions.

Fig.1 shows the evolution of ferrite and martensite phase fraction of this alloy as a function of annealing duration at 800°C. There is no significant evolution of the phase composition by increasing time from 17 to 30 days, evolution also confirmed by EPMA measurement of composition in each phase. Thus minimum annealing duration for the establishment of the thermodynamic equilibrium at a temperature of 800 ° C is considered to be attain after 17 days (stabilization of the composition and phase fraction). In the same way, some selected alloys are characterized, by using X-ray diffraction (XRD), scanning electron microscopy (SEM), field emission gun scanning electron microscopy (FEG-SEM) and electron probe microanalysis (EPMA-WDS), between 700 and 1000°C to specify the areas of stability of γ and κ by the characterization of the α/γ, α/κ, α/γ/κ tie-lines.

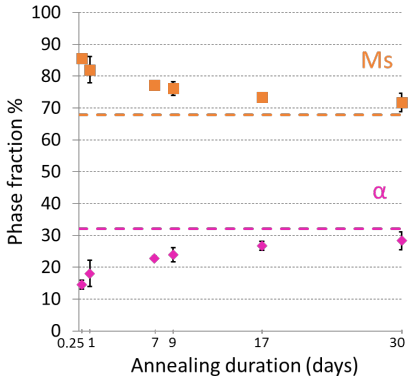

Figure 1: Evolution of ferrite and martensite phase fraction of Fe-0.2C-5Mn-2Al alloy as a function of annealing duration at 800°C. Experimental data (symbols) in comparison with the calculated equilibrium values (dotted lines: database PrecHiMn-03, coll. B. Hallstedt).

Acknowledgements
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LOW DENSITY STEELS

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New lightweight materials for automotive applications are constantly being searched to reduce the weight of cars and by that the levels of carbon dioxide emissions. On this quest a "new" family of steels is introduced; the low density or lightweight steels where important levels of aluminum are added. In comparison to the so-called third-generation steels where the strength is increased while keeping at least the same formability of current concepts, this family brings in addition density reductions higher than 8%. At the levels required, aluminum introduces complex phenomena in steels. Here, some of the effects of aluminum in phase stability and microstructure development are described, the latter in relation with mechanical properties. The potential of lightweight steels for automotive applications is also highlighted.

COMPUTATIONAL DESIGN OF HIGH STRENGTH STEELS CONTAINING UP TO 10 ALLOYING ELEMENTS: 'FROM SCRATCH'

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Keywords: *computational design, stainless steel, improvement*

The complex compositions of current high strength stainless steels for room temperature and high temperature applications are the result of many successive costly experimental programs over a period of over 100 years. However, given our current advanced thermodynamic calculations, metallurgical models and artificial intelligence techniques we should be able to design such complex steels 'from scratch'

In the presentation I will present and demonstrate the results of a 10 year program at the TU Delft in the computational alloy design of UHS stainless steels for room temperature applications as well as creep resistant steels for use at specific temperatures and with pre-defined life times. The model suggests that major improvements in properties of creep resistant steels can be achieved, in particular for PH stainless steels with a martensitic matrix. The design strategy also predicted a new family of W based stainless steels with excellent creep resistance, just before they were presented in Japan as the outcome of a long and costly development program.

While the optimal compositions predicted by the model are probably not the really optimal solution from an industrial perspective, they offer an excellent starting point for much faster and cheaper experimental development programs.

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DESIGN OF ALLOYS BY MULTI-OBJECTIVE COMBINATORIAL OPTIMISATION: PHYSICAL METALLURGY, THERMODYNAMICS, DATA MINING AND GENETIC ALGORITHMS

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Keywords: *alloys-by-design, evolutionary computing.*

Combinatorial metallurgy has traditionally relied on trial-and-error. Nowadays, it makes an increasing use of modelling, simulation and, more generally, of computing tools. In combinatorial terms, its ultimate achievement would consist in being able to predict the properties of all possible alloys; unfortunately, with several tens of potential alloying elements and as many possible concentration levels for each of them, a rough count shows that even if each calculation took the shortest imaginable time (at the extreme: Planck time, i.e $5.39.10^{-44}$ s), then the whole calculation would last several orders of magnitude longer than the age of the Universe… Optimisation methods, like genetic algorithms and their multi-objective counterparts, have been developed to optimise simultaneously several characteristics. These methods are very efficient to explore (partly) the "space of possibles", but they need nevertheless to rely on fast predictive tools (typically, the evaluation of each alloy must take less than a minute). This forbids the use of time-consuming modelling or simulation tools which have to be replaced by models that are both fast and reliable, such as data mining tools (neural networks, Gaussian processes…), computational thermodynamics by the "CALPHAD" method (Thermo-Calc), physical metallurgy analytical models (Hume-Rothery rules, solid solution hardening, dynamic recrystallisation…), or combinations of such models. Examples will be given in the field of nickel-based superalloys [1] and of "high entropy alloys".

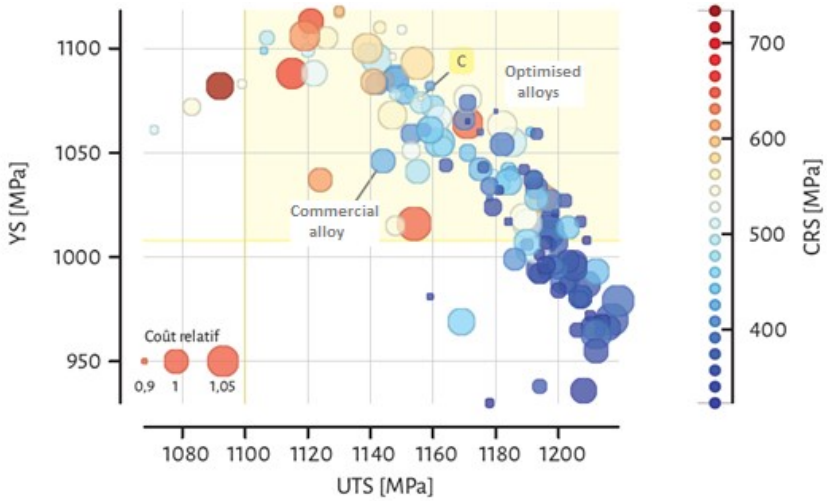

Figure 1: Properties (yield stress, ultimate tensile strength, creep rupture stress and cost) of optimised superalloys compared to those of a concurrent commercial alloy. For instance, alloy "C", although cheaper, is better than the commercial alloy on the three thermomechanical properties.

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NITROGEN STEEL METALLURGY VERSUS CARBON STEEL METALLURGY

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Over the past decade, the authors have developed, in collaboration with ArcelorMittal, nitrogen steel metallurgy. It consists in substituting, partly or fully, nitrogen to carbon in steel. It addresses two major concerns of automotive manufacturers: weight savings and environmental footprint. The two main advantages of nitrogen over carbon highlighted in the present work are its superior solubility in both ferrite and austenite as well as its enhanced reactivity with alloying elements. However, its low solubility in liquid iron requires its introduction in the alloy at the solid state.

Specifically designed thermochemical treatments enabled the successful achievement, in the Fe-N binary system, of most equivalent microstructures known in carbon steel (pearlite, martensite…). The feasibility of nitrogen ferrite/martensite dual phase (DP) steel was also demonstrated (Fig. 1). The concept was subsequently extended to higher-order systems such as the Fe-Si-N ternary system, in which large volume fractions of nanometric silicon nitride Si3N4 can precipitate during the nitriding treatment (Fig. 2). Their density (1.8) is low with respect to the ferritic matrix. These precipitates initially have a cuboidal shape and an amorphous structure stabilized by the excess nitrogen in solid solution in the ferrite matrix. Subsequent removal of the excess nitrogen by annealing in a denitriding medium triggers a transition from the amorphous nitride to one of its crystalline polymorph. This structural transition occurs though the dissolution of the amorphous phase in favor of the nucleation and growth of the crystalline particles, which display a hexagonal prism shape and a slightly increased density (3.3). It was possible to simulate this transition using a purposely-developed numerical model accounting for all the aspects of the precipitation sequence, in particular stress relaxation.

Besides, it has been demonstrated that alloying with trace amounts of aluminum, titanium and vanadium leads to the formation of clusters acting as nucleating agents for the amorphous silicon nitride. Such micro alloying could afford a fine control over the size distribution of the obtained precipitation as well as its amorphous/crystal transition.

This fine precipitation of silicon nitride leads in all cases to a significant hardening to the ferritic matrix and a decrease of the overall density of the material. The resulting composite thus shows promising specific mechanical properties with contained cost implications, making this type of material an attractive alternative for the automotive industry.

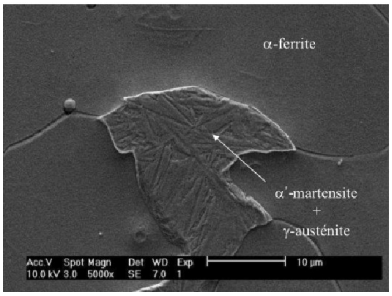

Figure 1: SEM micrograph of an island of martensite and retained austenite in a ferrite matrix in an Fe-N alloy..

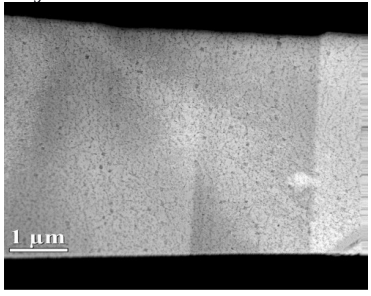

Figure 2: STEM micrograph showing the high volume fraction of Si_3N_4 precipitates in a nitrided Fe-3.3Si alloy.

HOW TO DEFINE HIGH ENTROPY ALLOYS AND WHAT PROPERTIES TO EXPECT?

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Keywords: *high entropy alloys, microstructure.*

High entropy alloys (HEA) are gaining attention since their conceptualisation by Yeh et al. in 2004 [1]. HEA are compositionally complex alloys, in which all elements are very concentrated (from 5 to 35 at.% per element), and form a single solid solution. This alloying design is based on configurational entropy maximization, which is believed to stabilize the solid solution and prevent the formation of intermetallic compounds. Due to these peculiar microstructures, HEA combine high strength and ductility [2]. Our approach consists in the systematic study of these alloys to better understand the chemical and physical phenomena underlying the aforementioned properties. In this context, we focused our work on two alloy families. Concerning the fcc CrMnFeCoNi family, a true solid-solution was observed down to the atomic scale [3], which is apparently present for Ni contents spanning from 100 at.% to as low as 10 at.% [4]. This leads one to believe that the configurational entropy, which is maximized for the equimolar composition, is not a sufficient criterion to define this kind of multi-component alloys. Moreover, the study of bcc TiZrHfNbTa [5-9], and more generally Ti-based HEA, showed that, by tuning the chemical composition using enthalpy maximization calculations or Ti alloys chemical design tools such as the Bo-Md diagram, one could observe tremendous effects on both the microstructure [10] and the mechanical properties [11].

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DEVELOPMENT OF HIGH STRENGTH AUSTENITIC HEA STEELS FROM CoCrFeMnNi FAMILY

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The HEA (high entropy alloys), first proposed in 2004, are formed by "cocktail of metals" and contain at least five elements in near equiatomic proportions. In the huge family of HEA alloys, the equiatomic CoCrFeMnNi system (EA, for "equiatomic alloy") is one of most studied.

In this work, the benefic effects of modifications of chemical composition of EA are discussed. An original composition has been chosen through Thermocalc calculations of phase stability in CoCrFeMnNi system. Called now A3S grade (austenitic super stainless steels), this alloy (in forged state) reaches at RT a yield strength of 800 MPa (twice as high as that of analoguous EA). This high mechanical resistance is associated to a fracture elongation of 35 %. Moreover, the deformed microstructure is stable up to high temperatures, thanks to recovery starting only after 50h at 550°C. Finally, the A3S grade conserves a ductile austenitic structure even at LN2 temperature; in these conditions, shock toughness above 120 J/cm² could be measured.

Different possible mechanisms of plastic deformation are discussed, by comparison between A3S and EA alloys. Several hypotheses could be rejected: no significant fluctuations of local chemical compositions have been observed (APT measurements); no martensitic transformation was identified; no more that the presence of (nano)twins in A3S, while this defect is currently observed in EA. Therefore, as the A3S alloys (in contrast to EA) show a great ability to create and to stabilize nanostructures, this feature is proposed as being responsible for the high mechanical resistance of A3S.

NOUVEAUX ALLIAGES DE TITANE À TRÈS FORT ÉCROUISSAGE : CONCEPTION, MICROSTRUCTURES ET PROPRIÉTÉS MÉCANIQUES

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Bien que connus pour leur excellente combinaison de propriétés mécaniques, les alliages de titane ont généralement des domaines de déformation plastique qui n'excèdent pas 15% et leur plasticité s'accompagne toujours d'une consolidation par écrouissage faible ou nul. Le manque de ductilité de ces alliages constitue un problème récurrent qui limite certaines applications industrielles. En réponse à cette problématique, notre travail porte sur les approches de conception visant à développer des alliages de titane combinant des effets TRIP (Transformation Induced plasticity) et TWIP (Twinning Induced Plasticity) permettant une très forte ductilité (près de 50% à rupture) accompagnée d'un écrouissage important. Nous avons entrepris une démarche de conception d'alliages basée sur les propriétés électroniques régulant la stabilité chimique de la phase β et nous avons formulé des alliages modèle ainsi que de nouveaux alliages de titane industrialisables base Ti-Cr-X (X=Al, Sn) à très fort potentiel.

Les résultats sur ces alliages Ti-Cr-X montrent une combinaison de propriétés mécaniques (Rm=1200MPa/40% de ductilité, très fort écrouissage) non encore atteinte pour les alliages de titane. Les observations en MET ainsi que des analyses synchrotron et en EBSD ont permis de mettre en évidence des microstructures de déformation très complexes combinant la présence de différentes phases précipitées sous contrainte à un maclage mécanique intense du type {332}<113>. Les résultats obtenus sont prometteurs car la combinaison de propriétés obtenue concurrence les aciers les plus avancés. Le travail présenté sera centré sur les stratégies de conception ainsi que sur l'étude fine des relations microstructures/propriétés dans cette nouvelle famille d'alliage.

STRATÉGIES DE DÉVELOPPEMENT DE MICROSTRUCTURES MULTI-ÉCHELLES DANS LES ALLIAGES DE TITANE « PROCHE BETA » : ÉTUDE DES RELATIONS PROCÉDÉS / MICROSTRUCTURES / PROPRIÉTÉS MÉCANIQUES

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Ce travail porte sur la compréhension des phénomènes liés aux séquences de transformations de phases dans la famille des alliages de titane proche-beta (alliages Ti-555 et Ti-18, en particulier), ainsi que leur influence sur les propriétés mécaniques finales des alliages. Nous avons mis au point une stratégie, basée sur des couplages de rampes de chauffage/refroidissement spécifiques et donc sur les cinétiques de changements de phase dans les alliages de titane beta-métastable, permettant d'obtenir une microstructure trimodale comprenant trois échelles différentes de phase a. Nous avons mis en évidence la compétition entre les différents modes de précipitation de la phase α, en particulier vis à vis du rôle joué par la phase ω isotherme sur un régime cinétique « basse température » de la précipitation de la phase α.

L'étude des propriétés mécaniques associées à ces microstructures montre par ailleurs que les échantillons à microstructures trimodales présentent un comportement mécanique très prometteur avec des niveaux de résistance supérieurs à ceux rapportés pour des microstructures bimodales classiques. Le travail présenté sera centré sur la compréhension des modes de précipitation de la phase a et la formation des trois échelles de précipitation de la phase a en fonction des différents paramètres du procédé (vitesses de rampe, températures de traitement) ainsi que la caractérisation des relations microstructures/propriétés mécaniques, principalement par suivi « in situ » par EBSD d'essais mécaniques monotones.

EVALUATION OF AN ALUMINIUM LITHIUM ALLOY FOR AEROSPACE APPLICATION

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Keywords: *aluminium, aerospace, mechanical properties.*

Weight reduction is a key driver for the aerospace industry, linked to increased payload and reduction of the fuel consumption. Use of materials with improved mechanical properties and / or reduced density without compromising neither durability nor safety is a way to achieve lighter structures. Aside from large use of composite (fiber reinforced plastic), better understanding of the metallurgy of more traditional metallic alloys has led to propose new alloys well-tailored for an optimisation of the strength to weight ratio.

This is the case of aluminium lithium alloys: study of the role of each alloying element - with a focus on lithium - has allowed aluminium alloy producers to solve some issues that previous generations of aluminium lithium have faces. In this frame, Constellium is proposing an aluminium-copper-lithium alloy registered as 2050, available in thick plate and part of the Airware® family. This alloy, in T84 temper, is hardened by the precipitation of T1 (Al_2CuLi) phase [1].

Safran Nacelles has evaluated this material for nacelles application and compare the properties with those of incumbents alloys, mostly aluminium zinc (7010 / 7050) and aluminium copper (2219). Tensile properties are in line with other published data [1] and specification [2], with an ultimate strength above 500 MPa in longitudinal and long transverse directions for a thickness up to 150mm.

This represents more then 10% increase in ultimate and yield strength compare to 7010 / 7050 alloys. Nacelles components being close to the engine, material resistance to elevated temperatures is an important property. Tested at elevated temperature, 2050 displays remarkable thermal stability making it a competitor to aluminium alloys specifically developed for high temperatures like 2219 or 2618. Fatigue properties have been evaluated using axial force control standard (ASTM E 466); 2050 shows improved fatigue strength compare to incumbent 7xxx alloys, mainly in low cycle fatigue.

Corrosion resistance is also a key characteristic in aerospace application. For aluminium alloys, this is usually achieved by surface treatment like anodizing and paint application. Ability of 2050 to receive standard surface treatment and resist to corrosive environment, simulated by salt spray, has been checked.

Finally, weight reduction has been assessed for a particular part considering the reduction in density – due to lithium addition, 2050 density is 4% lower than 7050 - and improved mechanical strength.

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PRECIPITATION MECHANISMS DURING AGING OF CENTRIFUGALLY CAST HEAT-RESISTANT STEELS

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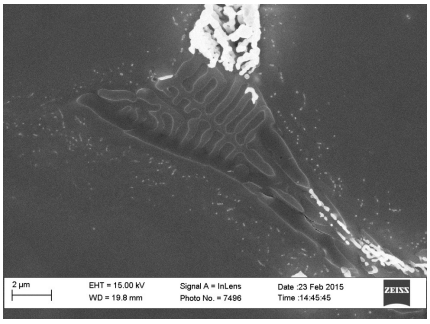
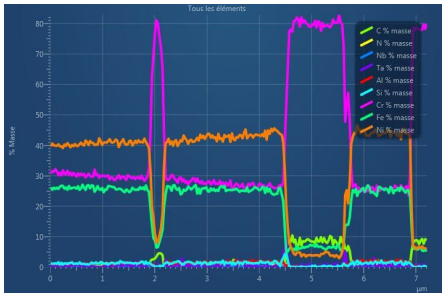
Keywords: *Refractory Steel, Casting, Creep, Precipitation, Diffusion*

This work deals with centrifugally cast heat-resistant steels used in the petrochemical industry for catalytic reforming or vapo-craking with typical service temperatures ranging from 700 to 1100°C. Pipes are usually used in the as-cast state and they should be creep resistant to sustain the inlet pressure and/or their own weight. They should also resist to the hard environment that leads to oxidation, nitriding and carburizing. They contain a significant amount of Cr (25 to 35%) for the oxidation resistance combined with a relatively high amount of Ni (35% to 45%) providing a stable fcc structure. They exhibit a dendritic structure « decorated » by primary carbides (M_7C_3 and MC) [1, 2]. During service, the unstable M_7C_3 carbides decompose leading to the nucleation of the stable $M_{23}C_6$, giving rise to the so-called secondary precipitation [1, 2]. The control and the optimization of this secondary precipitation is an important challenge since it directly affects the creep resistance. Indeed it occurs mainly inside the original dendrites where precipitates may efficiently interact with dislocations and reduce the creep deformation rate.

The exact mechanisms of the secondary precipitation are however not yet fully understood. Thus, the aim of this work was to carry out some careful investigations of the microstructure by SEM and TEM to understand how M_7C_3 carbides are transformed into $M_{23}C_6$, how the excess of carbon and the chromium depletion resulting from this transformation lead to chemical gradients (Fig. 1(b)) and how these chemical gradients are connected to the evolution of the secondary precipitation zone that progressively appears inside dendrites (Fig. 1(a)).



(a)



(b)

Figure 1: (a) SEM image showing large primary carbides (original M_7C_3 dark, NbC bright) and nanoscaled secondary carbides ($M_{23}C_6$) that have nucleated in their vicinity, inside the dendrite. (b) STEM-EDS concentration profile across a primary carbide transformed into $M_{23}C_6$ (right side) and a secondary $M_{23}C_6$ (left side). Ni and Cr gradients are clearly exhibited, the Cr depleted zone (25-30% Cr) is carbide free (see (a)).

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MICROSTRUCTURAL EVOLUTION DURING THERMOMECHANICAL PROCESSING IN A NIOBIUM STABILIZED AUSTENITIC STAINLESS STEEL (316 NB)

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Keywords: *austenitic stainless steel, recrystallization, residual work-hardening.*

Due to their excellent corrosion resistance and appropriate combination of mechanical properties at high temperatures, austenitic stainless steels are widely used in nuclear industry. In 316 Nb thick-walled components, work hardening, dynamic recovery and recrystallization govern hot workability and affect final mechanical properties. Static recovery as well as static or post-dynamic recrystallization can also induce further metallurgical evolution. In addition, solute atoms such as Nb and Mo as well as Nb-rich precipitates may significantly affect recrystallization mechanisms. Obtaining a given microstructure to improve mechanical properties requires deep understanding of the hot deformation behaviour and microstructural evolution mechanisms of this material.

The influence of hot deformation conditions and of initial microstructural parameters, such as grain size and niobium solute content, on dynamic and post-dynamic recrystallization was determined from high-temperature torsion tests. The effect of ultimate annealing on static recovery/recrystallization was then studied. Grain and twin morphology, as well as internal misorientations were determined thanks to optical microscopy and electron backscatter diffraction. Recrystallized fractions were calculated by using the Grain Orientation Spread criterion [1]. The spatial and size distributions of Nb(C,N) niobium carbonitrides was characterized by scanning electron microscopy on carbon extraction replicas.

The extent of dynamic recovery, coarse initial grain size, solute drag phenomenon, and pinning of grain boundaries by fine Nb(C,N) particles strongly hinder dynamic recrystallization which does not significantly contribute to the metallurgical evolution over the range studied, in contrast to literature results reported on Nb-free 316 stainless steel. However, at moderate strain levels, post-dynamic recrystallization occurs by rapid growth of nuclei that depends on deformation temperature, and applied strain and strain rate. A significant residual fraction of non-recrystallized grains is kept after annealing, if the niobium solute content is increased by increasing the soaking temperature before straining. Increasing the initial grain size beyond a (high) critical value also helps keeping residual work-hardening after annealing.

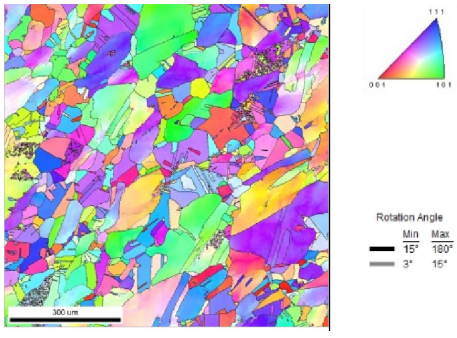

Figure 1: EBSD map (Inverse Pole Figure) showing a partially recrystallized microstructure obtained from a sample with an initial grain size of 250µm strained to $\varepsilon = 0.4$ at 1150°C and $0.05s^{-1}$ then annealed.

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PROPERTIES AND MICROSTRUCTURAL EVOLUTION OF DUPLEX STAINLESS STEELS: INFLUENCE OF CHEMICAL ANALYSIS ON PRECIPITATION AND FERRITE EMBRITTLEMENT

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Keywords: *duplex stainless steel, ferrite embrittlement, precipitation*

Duplex stainless steels (DSS) offer good combination of resistance to pitting corrosion and high mechanical strength that are not easily attainable using conventional single phase austenitic or ferritic stainless steels. The duplex family ranges from super DSS containing large amounts of chromium, nickel and molybdenum to low alloyed DSS with low nickel and molybdenum-free contents in order to develop economical solutions. A serious drawback in using such duplex stainless steels is the limited range of temperature at which the material can be used without risks of failure due to ferrite brittleness. This range of temperature for suitable use goes roughly from -50°C to 300°C.

Below room temperature, low impact energy of the ferrite is critical but the ductile to brittle temperature transition of the duplex can be lowered to some extent by increasing austenite fraction in the duplex or nickel content in the ferrite phase, which explains the superiority of super duplex compared to lean duplex.

Above 300°C, embrittlement is provoked by different phase changes occurring in the duplex grade and usually attributed to the un-mixing of the ferritic Fe-Cr solid solution, either by spinodal decomposition or by nucleation and growth of Cr-rich α' phase. Additional precipitation taking place during aging, like NiSiMo-rich G phase [1]. or Cu-ε, are also known to participate to embrittlement, making the composition dependence of embrittlement kinetic even more intricate.

Above 650°C very low impact energy at room temperature may be induced by inter-metallic precipitation of sigma σ phase and/or Chi χ phase (Fig.1). This precipitation occurs very rapidly when the alloys contain large amounts of chromium and molybdenum [2] which makes super duplex very sensitive to embrittlement in this range of temperature compared to lean duplex.

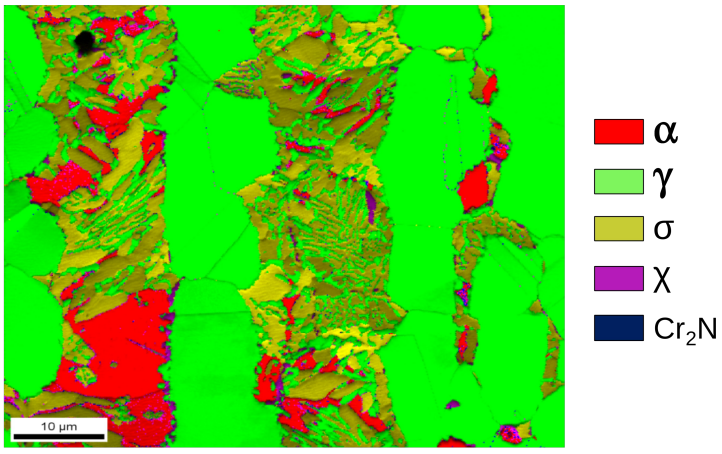

Fig.1 EBSD image of a super DSS after 1h-800°C

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CHARACTERIZATION AND OPTIMIZATION OF TENSILE PROPRETIES OF LEAN DUPLEX STAINLES STEELS FOR BUILDING APPLICATIONS

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Keywords: *duplex stainless steel, tensile properties, TRIP effect.*

Long products for building applications, like rebars or high strength bars, have to satisfy a demanding compromise between strength (Rp0.2 and Rm) and ductility (Agt or A5d). With current lean duplex grades, it is difficult and time consuming to find the right setup for cold forming operations like notching or drawing, to obtain compliant tensile properties. Increasing the mechanical properties, strength and ductility concomitantly, would be helpful to improve productivity and material in-service performance. Four laboratory heats of type 1.4362 with different chemical compositions and hence different degrees of austenite stability were cast and transformed. In situ tensile tests were performed under X-Ray Diffraction at ESRF to monitor the martensitic transformation during straining, the so-called Transformation Induced Plasticity effect. Three markedly different behaviours were observed, see Figure 1. An excessive stability of austenite (alloy A) inhibits the transformation into martensite and necking takes place early, giving a low ductility. At the opposite if austenite is too unstable (alloy D) TRIP effect is consumed at low strain, resulting also to a low ductility. The stability of austenite is thus a crucial parameter to control strength and ductility, and should be finely tuned so that TRIP effect is activated sufficiently and progressively along the tensile test to get a large extent of uniform strain (alloys B and C). Digital Image Correlation was performed on tensile specimen and showed that the increase in the strain hardening rate, while martensitic transformation is active, contributes to fade small strain localizations and postpone necking to larger strains, in agreement with the Considere‘s law. The results obtained were rationalized into a set of three criteria related to ferrite / austenite ratio, austenite stability and yield stress, applicable for alloy design of lean duplex with optimal mechanical properties in terms of strength and ductility. A second series of laboratory castings of lean duplex 1.4062 and 1.4362 were defined according to the latter criteria. The tensile tests confirmed that higher ductility and strength are obtained on the optimised grades compared to reference grades.

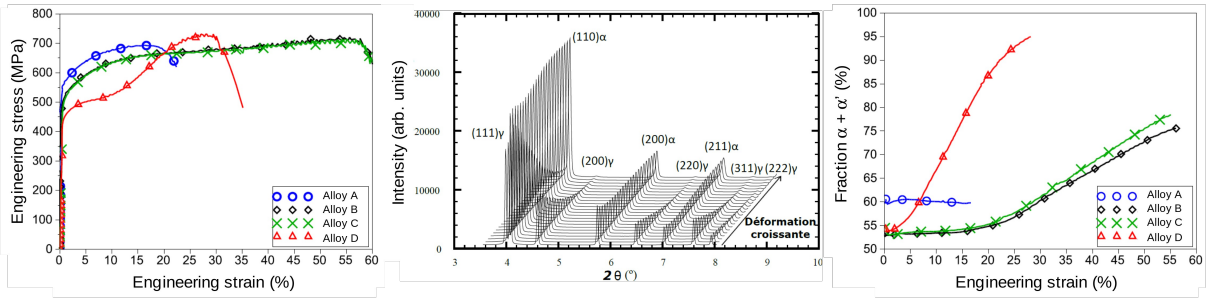

Figure 1: tensile tests performed in situ under X-Ray diffraction at ESRF. Left : stress strain cruves of the four alloys A to D. Middle : evolution of X-ray diffraction spectrum with strain. Right: evolution of ferrite + BCC martensite phase fractions with strain, after Rietveld analysis.

MULTI-OBJECTIVE DESIGN OF NICKEL-BASE SUPERALLOYS BY COMPUTATIONAL THERMODYNAMICS, DATA MINING AND GENETIC ALGORITHMS

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Keywords: *optimisation, Thermo-Calc, neural networks, multi-criteria decision making, Inconel 740H.*

A computing method for the systematic optimisation of the composition of superalloys is presented. Relying on multi-objective genetic algorithms, it allows on the one hand to explore a large compositional space, and on the other hand to optimise the performance of novel alloys. The performance of an alloy is evaluated from predictions of its microstructural and thermomechanical properties. The CALPHAD approach is employed to predict thermochemical characteristics (phase constitution, stability, weldability) [1], whereas Gaussian processes provide an estimate of thermomechanical properties (yield stress, tensile strength, creep resistance) by regression from databases [2]. These tools are integrated in a unique C++ routine which, applied to the design of Ni-base superalloys for power plants, provides numerous candidates supposedly thermomechanically strong and thermodynamically stable [3]. These results suggest that it should be possible to produce, at a reduced cost, alloys with better characteristics than those used at present (Fig. 1).

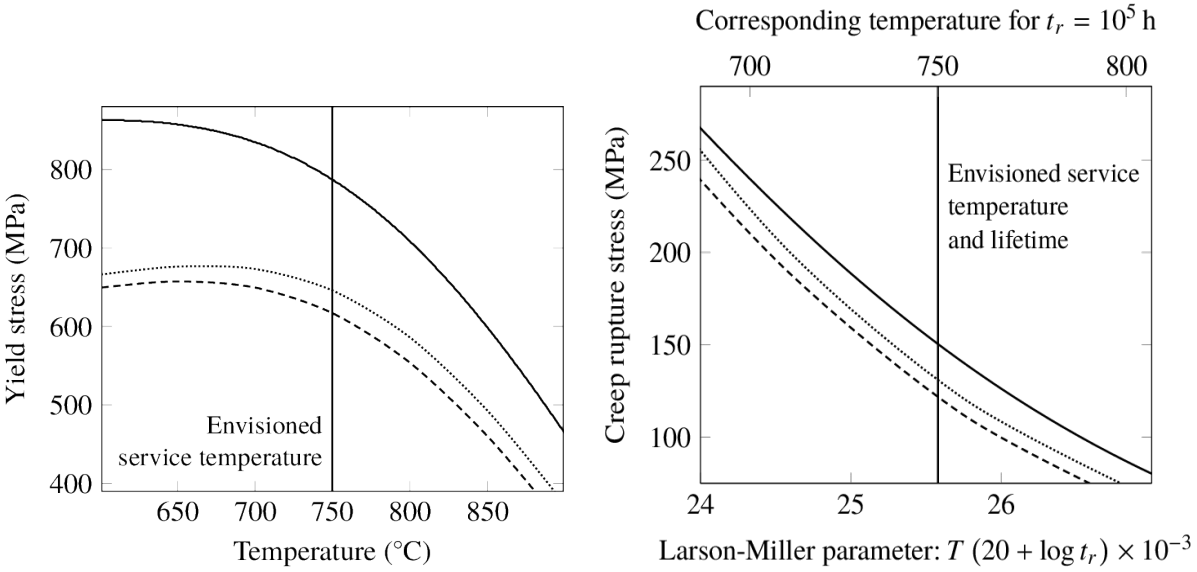

Figure 1: Predicted yield stress as a function of temperature (left) and Larson–Miller parameter plot (right) of Inconel 740H (dashed curves), Haynes 282 (dotted curves) and one of the designed alloys (solid curves). The vertical line marks the planned service temperature (and creep life of 10^5 h) at 750 °C.

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HETERO-EPITAXIAL RECRYSTALLIZATION IN LOW LATTICE-MISMATCH γ-γ' NICKEL-BASED ALLOYS: MECHANISM AND KINETICS

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Keywords: *recrystallization mechanism, nickel-based γ-γ' superalloys, coherent precipitates, EDS-EBSD coupling.*

A new recrystallization mechanism has been discovered in γ/γ' nickel-based superalloys, when deformed in the sub-solvus domain. It is triggered when a microstructure having undergone slow cooling is further hot deformed. It superimposes with the conventional dynamic recrystallization mechanisms that are classically observed in those alloys but leads to very specific recrystallized grains. Indeed, each primary precipitate generates a unique recrystallized grain, which moreover has the same crystallographic orientation. The nucleation of such grains actually occurs during the slow cooling stage applied prior to the hot-deformation stage, during which a coherent γ shell is formed at the rim of all primary γ' precipitates, by a process of "inverse precipitation". During hot deformation, the shells grow under the difference in stored energy with the surrounding deformed matrix. The specific kinetics of this mechanism arises from a complex interaction of driving forces. This mechanism was first observed in the René 65™ alloy [1], but could also be successfully triggered in other γ/γ' nickel-based superalloys [2,3]. A necessary condition is that the alloy must exhibit a close-to-zero lattice mismatch at high temperature [4].

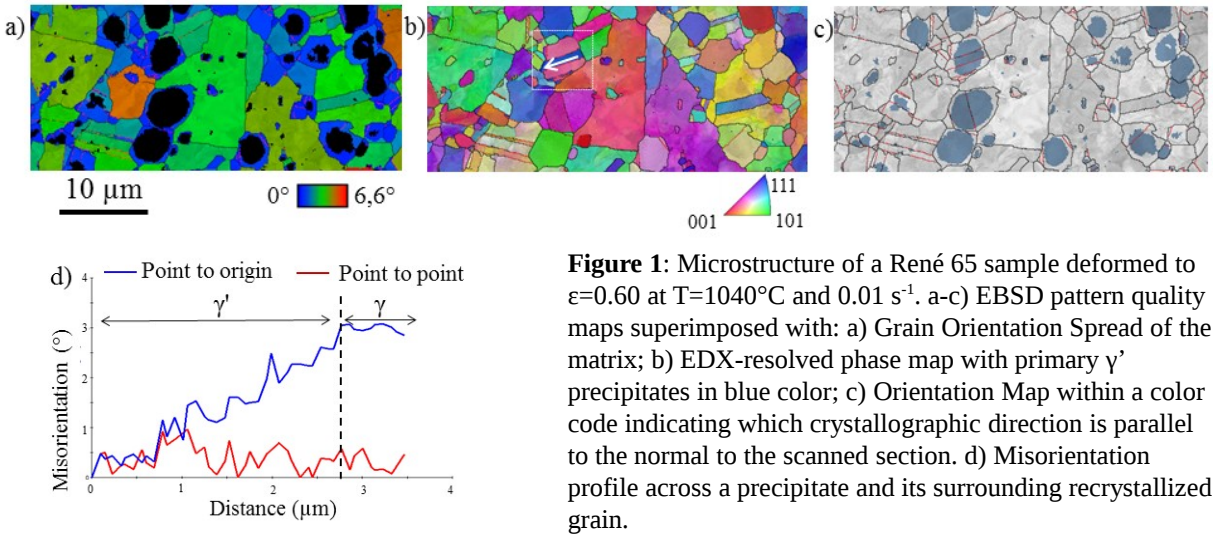

Figure 1: Microstructure of a René 65 sample deformed to ε=0.60 at T=1040°C and 0.01 s^{-1}. a-c) EBSD pattern quality maps superimposed with: a) Grain Orientation Spread of the matrix; b) EDX-resolved phase map with primary γ' precipitates in blue color; c) Orientation Map within a color code indicating which crystallographic direction is parallel to the normal to the scanned section. d) Misorientation profile across a precipitate and its surrounding recrystallized grain.

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EFFECT OF INTERFACE TRAPPING KINETICS ON DIFFUSION IN POLYCRYSTALLINE MATERIALS: HYDROGEN TRANSPORT IN NICKEL

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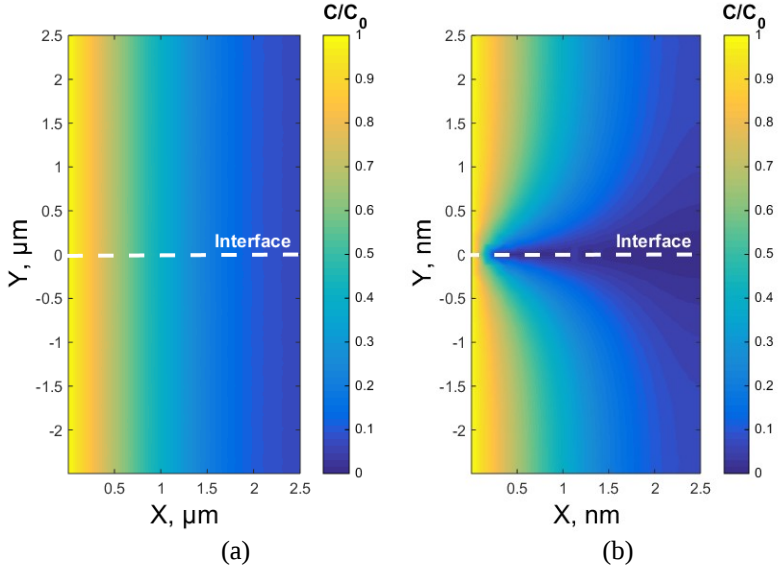
Keywords: *hydrogen, diffusion, trapping, interface, multi-scale method.*

Due to experimental limitations, the solute distribution in polycrystalline materials is difficult to obtain directly, especially in the vicinity of interfaces. We present a multiscale method for simulating diffusion in polycrystals taking into account specific diffusion properties of interfaces. The method connects continuum diffusion and an atomic layer model [1]. The numerical resolution combines finite elements to simulate diffusion in grains, finite differences on a coarse mesh for diffusion along the interface and an atomic layer model for diffusion across it. The proposed method is largely enriched with respect to current continuum models for intergranular diffusion. It can account for segregation, fast or slow diffusion along the interface, provides robust physically-based treatment of stress gradients (and thereby avoids the problem of convergence that can be encountered due to the presence of stress gradients across interfaces [2]) and handles cases where the local equilibrium hypothesis cannot be made: taking into account the trapping kinetics demonstrates the effect of the interface acting as a sink and retarding the diffusion in the bulk (see Figure 1).



(a) (b)

Figure 1: Hydrogen distribution in nickel bicrystal at time $t = 3\ s$ a) without the interface trapping property and b) with interface trapping (segregation energy $\Delta E_{seg} = -0.36\ eV$).

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NOUVELLES APPROCHES POUR LA QUANTIFICATION DES SÉGRÉGATIONS INTERFACIALES DANS LES MÉTAUX

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Keywords: *interface segregation, microbeam techniques, SIMS, EPMA.*

La ségrégation désigne, de manière générale, des écarts locaux à la concentration nominale d'un soluté dans un solvant. On appelle ségrégation « intergranulaire » le regroupement d'atomes de solutés sur les joints de grains d'un matériau, généralement sous la forme d'une fraction de monocouche. Ce phénomène est à l'origine de formes d'endommagement bien connues dans les matériaux métalliques.

La technique de prédilection pour l'étude et la quantification des ségrégations intergranulaires est la spectroscopie Auger sur fracture. Cette technique présente cependant plusieurs inconvénients : préparation d'échantillon difficile (nécessité d'obtenir in-situ une rupture intergranulaire), rareté des instruments, aptitude limitée à l'analyse quantitative. Nous présenterons ici deux nouvelles méthodes quantitatives, complémentaires de la spectroscopie Auger. La première méthode se pratique aussi « sur fracture » et met à profit le rayonnement X du soluté, mesuré grâce à un spectromètre WDS (Wavelength Dispersive X-ray Spectrometry). La seconde est fondée sur la technique SIMS (Secondray Ion Mass Spectrometry), que l'on pratique sur une simple coupe polie du matériau.

A partir de mesures de ségrégation réalisées sur des matériaux « modèles » et industriels, nous présenterons une étude comparative des méthodes de quantification (Auger, WDS et SIMS), pour souligner leurs atouts respectifs et leur complémentarité.

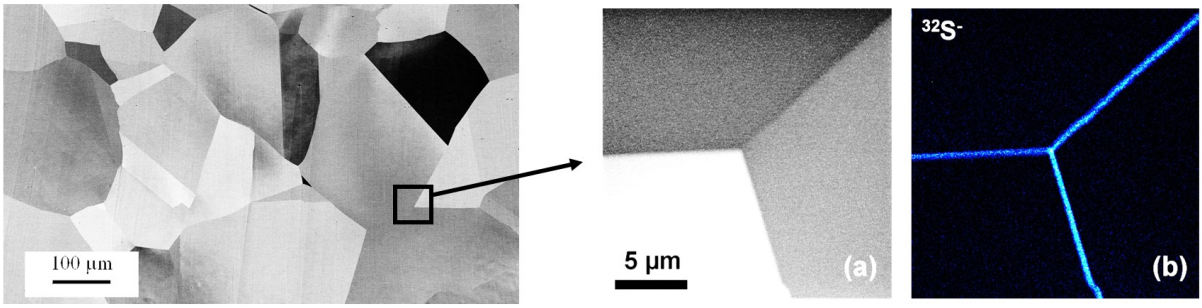

Figure 1: Evidence of sulphur grain boundary segregation in polycrystalline nickel obtained using NanoSIMS. (a) Secondary electron image. (b) $^{32}S^-$ ion map.

SOME FEATURES OF STRAIN-INDUCED GRAIN BOUNDARY MIGRATION (SIBM) IN PURE ALUMINIUM FROM SEM AND AFM OBSERVATIONS

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Features of grain boundary migration induced by plastic straining followed by heat treatment were investigated in high purity aluminum by combining EBSD (SEM) and micrographic 2D (SEM) and 3D (AFM) observations. Polycrystalline Al samples were submitted to sequences of 3% plastic strain followed by heat treatment. Assuming the driving force P for the boundary migration in the usual velocity law V=MP, to essentially be the sum of a curvature term P_c and of a dislocation density jump term $P_{\Delta\rho}$ at the boundary, both contributions are estimated for a set of boundaries. The former is obtained from boundary curvature measurements and the latter one with a crystal plasticity modeling. Firstly, the obtained data are presented under the form of a “migration diagram”. This diagram allows to pointing that if the usual velocity law applies globally, it can apply more or less accurately depending on the precise definition chosen for $P_{\Delta\rho}$. Second, several cases of triple boundaries are examined in details with the help of AFM surface and of SEM sub-surface observations.

We thus exemplify that:

i) boundaries apparently not obeying the velocity law correspond in most of the cases to particular geometries or neighborhoods;

ii) surface thermal grooving does not block migration but slows it down, in proportion to the groove depth;

iii) the velocity of connected boundaries may be ruled collectively rather than individually;

iv) one cannot exclude other mechanisms to contribute to the boundary motion such as grain boundary sliding.

STUDY OF THE MOBILITY OF GRAIN BOUNDARIES NEAR 40° AROUND <111> AND INTERACTION WITH DISLOCATIONS IN FCC METALS BY MOLECULAR DYNAMICS

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Keywords: *Grain boundary, migration, FCC.*

During a thermomechanical loading, grain boundaries (GB) generally migrate through polycrystalline samples. Despite an extensive literature on the subject, understanding the collective behavior of dislocations and GB remains partial, at least on the macroscopic scale. This is due, firstly, to the fact that several driving forces are active simultaneously during the experiments on polycrystals: (i) the surface tension of a curved boundary (ii) the differential elastic energy for elastically anisotropic materials, and finally (iii) the coupling between plasticity and migration. On the other hand, the mobility of such interfaces is closely related to their atomic structure.

This work focuses on understanding the migration of GBs near 40 ° around <111>, which were seen as particularly mobile in the experimental literature [1]. For this, Molecular Dynamics simulations of GB migration are made, using a synthetic driving force [2]. GB mobility is first discussed for various GB planes and temperatures. In a second step, the interaction with dislocations is considered. This interaction could lead to an enhanced mobility by lowering energy barriers required for migration.

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PLASTICITÉ LIÉE AU COUPLAGE MIGRATION / CISAILLEMENT DANS LES JOINTS DE GRAIN

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Les métaux à grains nanocristallins (nc) (≤ 100 nm) présentent une résistance à la déformation plastique largement supérieure au même matériau à taille de grains conventionnelle. Une des spécificités de ces nanocristaux est qu'ils ne contiennent pratiquement pas de dislocations. Plusieurs expériences ont montré que dans ces matériaux, les joints de grains eux-même participaient de façon prépondérante à la déformation plastique [1,2] par un mécanisme de couplage entre migration et contrainte de cisaillement.

Ce mécanisme de migration couplée au cisaillement est mal connu car peu de gens l'ont étudié, de façon expérimentale ou théorique. Nous avons pu montrer qu'il était basé sur le déplacement de défauts linéaires appelés disconnexions qui sont spécifiques aux joints de grains. Comme pour les dislocations dans les cristaux, les propriétés de ces disconnexions semblent guider le mécanisme de déformation par couplage. Pour arriver à cette conclusion, nous avons à la fois mené des expériences en MET in situ et des simulations en dynamique moléculaire à l'aide de la technique NEB.

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MICROSTRUCTURAL EVOLUTION PREDICTION DURING FORMING PROCESSES: TOWARDS A MODELLING BY INDUSTRY

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Keywords: *microstructural evolution modelling, mesoscale computations, level-set framework.*

Mechanical and functional properties of metallic materials are strongly related to their microstructures, which are themselves inherited from thermal and mechanical processing. Microstructural evolutions during thermomechanical treatments or thermal treatments are thus of prime importance for the control of the final in-use material properties (mechanical strength, fatigue limit, crack resistance, stress corrosion resistance...). Being able to accurately predict the microstructure obtained after complex processing routes recently became crucial for the metallurgy industry.

Macroscopic and homogenized metallurgical models are widely used in the industry, mainly due to their low computational cost. If this mean field framework is quite convenient, it can be synonymous for a given material of a large amount of experiments with advanced laboratory devices. Moreover, the homogenization of the microstructure does not permit to capture some very local phenomena.

In order to overcome these issues, lower-scale models (called full field models) have been developed over the last decades with the aim to simulate explicitly the microstructural evolution. The idea behind these “mesoscale” simulations is that the morphology and the topology of the grain boundary network play a non-negligible role in the evolution of the microstructure. In these approaches, simulations are performed on Representative Volume Elements (RVEs) where the microstructural features are explicitly represented. Boundary conditions applied to the RVE are representative of what suffered a material point at the macroscopic scale (thermal or thermomechanical cycle).

Recently a new full field approach, based on a Level Set (LS) description of the interfaces in a finite element (FE) context has been introduced to model primary recrystallization, including the nucleation stage, and has been extended to take into account the grain growth stage of monophasic polycrystalline structures [1]. Moreover in this LS context, Smith-Zener pinning (SZP) phenomenon can be taken into account in a natural way [2]. Modelling at the mesoscopic scale can thus be a help for understanding complex microstructural phenomena such as abnormal grain growth ; it can also be used to optimize/calibrate higher scale models (like mean field models) through numerical experiments leading to a real multiscale approach.

These full field approaches are generally associated with an elevated computational cost making them hardly usable in 3D. Moreover they require many numerical parameters whose calibration is not straightforward. Recent major developments and improvements addressed these issues [3] making possible the use of these approaches in an industrial context through the development of the DIGIMU® software.

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EVOLUTION OF THE INTERFACES DURING HIP-BONDING OF AUSTENITIC STAINLESS STEEL – FULL FIELD MODELING OF GRAIN GROWTH MECHANISM

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Keywords: *HIP-bonding, 316L, grain growth, level-set framework.*

Hot Isostatic Pressing-diffusion bonding of grooved plates is a potentially attractive technique for manufacturing compact heat exchangers. During the process, microstructural changes must be controlled and groove deformation must be minimised. Firstly, plastic deformation processes result in collapse of the surface asperities, the interfacial void closure is then achieved by diffusion controlled mechanisms such as creep and grain boundary diffusion, bonding is finally achieved by grain growth. The process parameters must be accurately calibrated in order to ensure the disappearance of the initial diffusion welding plane while controling the final grain size distribution. Modeling at the microscale in then a precious tool to reach these goals. A new full field front capturing framework based on a level-set (LS) description of the grain boundary network is currently developed in order to provide the *ad hoc* numerical tools. The following figure illustrates the 3D modeling of the last step of a HIP-bonding process (grain growth phenomenon) for 316L thanks to the LS approach, the color code corresponds to the grain size and more than 20,000 grains are considered in the simulation. Perspectives of improvement of these first simulations will also be discussed.

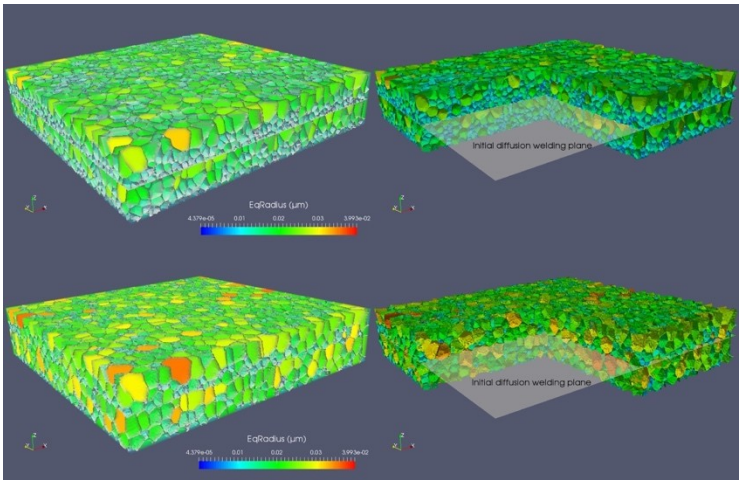

Figure 1. 3D modeling of the last step of a HIP-bonding process (grain growth phenomenon) for 316L thanks to the LS approach, the color code corresponds to the grain size. In the left side, the white surface corresponds to the grain boundary network. In the right side, a cutting view of the grain boundary network is described with the bonding plane (defined by the middle Z-plane). More than 20,000 initial grains are considered in the simulation. (Top) Representative microstructure at the beginning of the grain growth mechanism (where T-junctions are then present in the bonding plane). (Bottom) Evolution of the microstructure after 600s at 1100°C.

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MEAN FIELD MODELS FOR DYNAMIC AND POST-DYNAMIC RECRYSTALLIZATION: ASSESSMENT OF THE GRAIN SIZE DISTRIBUTION

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Keywords: *Discontinuous dynamic recrystallization, Mean-field modeling, Grain size distribution.*

A mean field model for discontinuous dynamic recrystallization (DDRX) has been chained with a post-dynamic recrystallization (PDRX) model to predict transient and steady-state flow stresses and average grain sizes during deformation and subsequent recrystallization. Numerical results are compared with experimental data obtained on a 304L stainless steel yielding to a good agreement in terms of average grain size. However an unrealistic grain-size distribution is observed after DDRX, which affects the results of PDRX model. It is proposed to highlight consequences of chaining DDRX and PDRX mean field models on average grain size and grain size distribution.

The mean field model employed here has been described in detail elsewhere [1]; A spherical grain interacts with an equivalent homogeneous matrix whose properties are the average properties of all the grains. This leads to the following grain growth equation:

$$\frac{dD_i}{dt} = 2M\tau\left(\bar{\rho} - \rho_i\right) \qquad (1)$$

The grain size distribution predicted during steady state from the numerical implementation is presented in Fig.1. The theoretical density tends to infinity for the larger grain size, while the experimental distribution roughly exhibits a log-normal shape. This distribution impacts the coupling of DDRX and PDRX models. The evolution of the average grain size during PDRX is shown in Fig. 2 [2]. The offset observed is related to the time required to inverse the initial grain size distribution shape into a more realistic distribution. When t=0, driving forces are low because most of the grains have similar dislocation densities and sizes (i.e.and).

The unrealistic grain size distribution is a consequence of the deterministic Eq. 1 which can be modified by introducing stochastic aspects with a simple connectivity between grains.

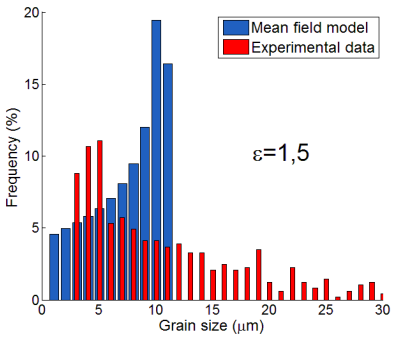

Figure 1: Grain size distributions during DDRX

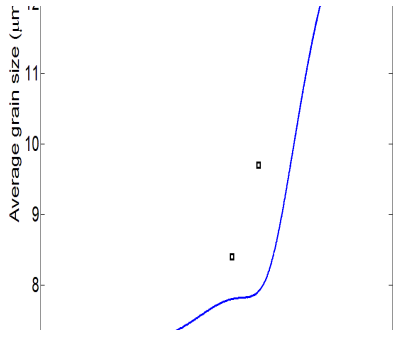

Figure 2: Average grain size evolution during PDRX

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FULL FIELD MODELLING OF LAMELLA SPLITTING AND LATH SPHEROIDIZATION IN A/B TITANIUM ALLOYS

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Keywords: *spheroidization, surface diffusion*

The α/β titanium alloys are materials that receive considerable attention due to their attractive mechanical properties and their numerous industrial applications. Spheroidization of the α laths occurs in α/β titanium alloys during thermomechanical processing and it is a very important mechanism for the microstructure control. The spheroidized microstructures indeed show enhanced strength and ductility. This study is focused on Ti-64 alloy, with an initial microstructure made of colonies of α lamellae in former β grains. During spheroidization, the α lamellae split into smaller α laths, which subsequently undergo spheroidization. The phenomenon is considered here under isothermal conditions only.

The initiation of this phenomenon occurs during thermomechanical processing with the appearance of grooves along the α lamellae. The subsequent splitting and spheroidization is driven by the minimization of the total interfacial energy. According to the current knowledge of the phenomenon, at a given temperature (and thus constant phase volume fraction), the governing mechanisms are surface diffusion at the α/β phase interfaces, α/α interface motion driven by capillarity, and coarsening, in addition to crystal plasticity [1].

In the present work, a method to simulate the global phenomenon as it occurs during and after hot deformation is being developed. So far, the main focus has been placed on the effect of interface diffusion and interface motion driven by capillarity on the splitting of the lamellae and on spheroidization of laths. During deformation, local misorientations develop inside α lamellae, which result in the formation of α/α sub-boundaries (see Fig. 1). Atomic scale processes are initiated near the intersection of α/β interface and α/α sub-boundary, which result in the appearance of grooving inside α lamellae. Basically, the α/β interfaces are initially controlled by surface diffusion while the α/α interfaces are driven by capillarity forces [2].

A new level set - finite element framework is used to describe that interfacial kinetics [3]. Furthermore, several mesh adaptation techniques have been tested in order to efficiently describe the shape evolution of α laths and to reduce the calculation time.

Additionally, hot compression tests have been carried out on Ti-64 double-cone samples at 950 °C in order to get an accurate experimental description of the involved physical phenomena and to produce suitable data to be compared with simulation results (example shown on Figure 1).

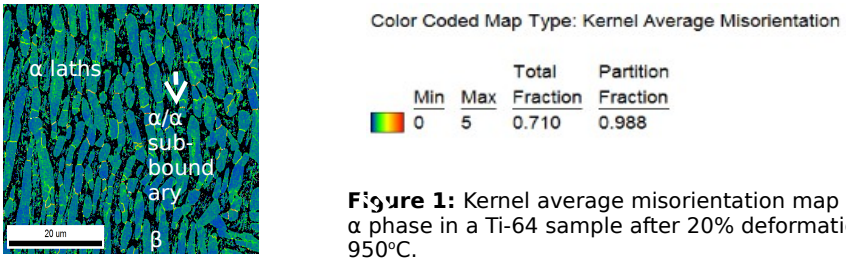

Figure 1: Kernel average misorientation map of the α phase in a Ti-64 sample after 20% deformation at 950°C.

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MODÉLISATION DE LA RECRISTALLISATION PAR CHAMPS MOYENS : AVANCÉES RÉCENTES, LIMITES ET PERSPECTIVES

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L'évolution microstructurale par recristallisation durant la déformation à haute température des métaux est étudiée par des modélisations à champs moyens, en association avec une méthode d'optimisation inverse. Cette technique permet l'identification des paramètres inconnus du matériau comme la mobilité des joints de grain, ou encore la fréquence de nucléation des grains. Il est alors possible de quantifier l'effet des éléments d'alliage sur la microstructure, et d'effectuer des comparaisons avec les métaux purs. Les comparaisons avec des données expérimentales jettent un nouvel éclairage sur les mécanismes à l'origine des transformations microstructurales, notamment sur l'effet de l'auto-échauffement et des évolutions post-dynamiques. L'ajout de mécanismes supplémentaires ne se traduit pas forcément par une plus grande complexité, mais améliore grandement les prévisions du modèle à haute vitesse de déformation. Cependant, le manque de précision concernant la distribution de taille de grains rappelle les limites des modèles à champs moyens. De nouvelles perspectives sont à envisager à travers un modèle unifié de la recristallisation continue et discontinue, avec des considérations topologiques.

DISCONTINUOUS DYNAMIC RECRYSTALLIZATION: EXPERIMENTAL ANALYSIS, ASSISTED BY CLOSED FORMS MODELLING, OF GRAIN-BOUNDARY MIGRATION AND NUCLATION OF NEW GRAINS

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Keywords: *Discontinuous dynamic recrystallization, Grain-boundary mobility, Nucleation rate.*

The present abstract is the result of a work of *circa* 20 years for the development of a mesoscopic model for discontinuous dynamic recrystallization. Each grain is considered in turn as an inclusion, embedded in a homogeneous equivalent matrix, the properties of which are obtained by averaging over all the grains. The model includes: (i) a grain-boundary migration-equation driving the evolution of grain size *via* the mobility of grain boundaries, which is coupled with (ii) a single-internal-variable (dislocation density) constitutive model for strain hardening and dynamic recovery, and (iii) a nucleation equation governing the total number of grains by the nucleation of new grains.

All the system variables tend to asymptotic values at large strains, in agreement with the experimentally observed steady-state regime. With some assumptions, both steady-state stress and grain-size are derived in closed forms, allowing immediate identification of the mobility of grain boundaries and the rate of nucleation.

An application to Ni–Nb-pure-binary model alloys and high-purity SAE 304L stainless steel with Nb addition is presented. More specifically on one hand, from experimental steady-state stresses and grain sizes, variations of the grain-boundary mobility and the nucleation rate with niobium content are addressed in order to quantify the solute-drag effect of niobium in nickel. And on the other hand, the strain-rate sensitivity and the activation energy of both the mobility of grain boundaries and the nucleation rate of new grains are evaluated by both empirical fitting and theoretical derivation. This leads to the introduction of two Derby exponents [1] varying separately the strain rate or the temperature:

$$a_T = -\left(\partial \ln \sigma_s / \partial \ln D_{sd}\right)_T = -m / m = 1.2,$$

$$a_{\dot{\varepsilon}} = -\left(\partial \ln\left[\sigma_s/\mu\right] / \partial \ln D_s\right)_{\dot{\varepsilon}} = \left(Q_\mu - m_\sigma Q_\sigma\right) / \left(m_D Q_D\right) = 0.9.$$

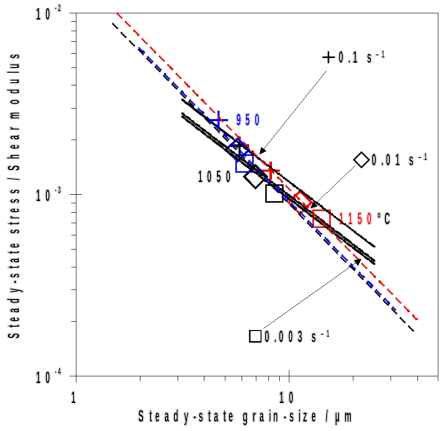

Figure 1: Introduction of two Derby exponents

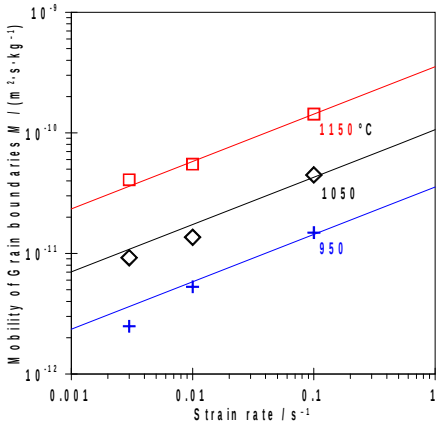

Figure 2: Grain-boundary mobility vs. strain rate

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FULL FIELD MODELING OF DYNAMIC RECRYSTALLIZATION IN A LEVEL-SET FRAMEWORK

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Keywords: *DRX, Crystal Plasticity, Level-Set.*

The optimization of industrial forming processes relies on the understanding of the material behaviour under the deformation conditions. During hot working, the energy is mainly released through heat dissipation. However, a small part of the energy remains stored in the material under the form of crystallographic defects, principally dislocations. Many mechanisms can then be activated to minimize this energetic potential, such as recovery, grain growth or recrystallization. It is essential to simulate these microstructural evolutions with regard to the final in-use properties. From a numerical point of view, the full field methods have a great potential. They rely on an explicit description of the polycrystal, which enables to localize the microstructure evolutions.

The formulation presented here relies on a front capturing approach concerning the description of the grain interfaces thanks to the level-set method [1]. A polycrystal RVE is generated with a Laguerre-Voronoï algorithm to respect any given grain size distribution. Then the RVE is immersed onto the finite element mesh where grain interfaces will be known from the signed distance functions, called LS functions. LS methods allow to track the evolution of grain boundaries without adding any numerical parameter. Such evolution results from the resolution of the partial differential equations systems concerning DRX grain boundary motion. The energetic state of the grains is obtained from the concomitant plastic deformation using crystal plasticity finite element method (CPFEM) within a unified numerical framework. This numerical framework enables to deal with high strain level and to handle topological events, such as the appearance and disappearance of grains during recrystallization and grain growth. This numerical strategy is applied here to simulate DRX in 304L stainless steel. The results are discussed comparing the RVE numerical simulations with 2D experimental data from previous studies [2], [3].

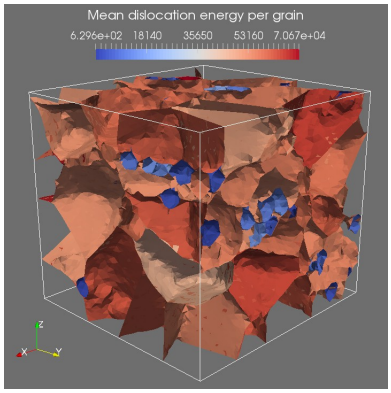

Figure 1: Mean stored energy field ($J.m^{-3}$) on grains and nuclei during dynamic recrystallization

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EFFECT OF INTRAGRANULAR STRAIN HETEROGENEITY ON RECRYSTALLIZATION KINETICS ASSESSED BY NUMERICAL SIMULATION AT THE MESOSCOPIC SCALE

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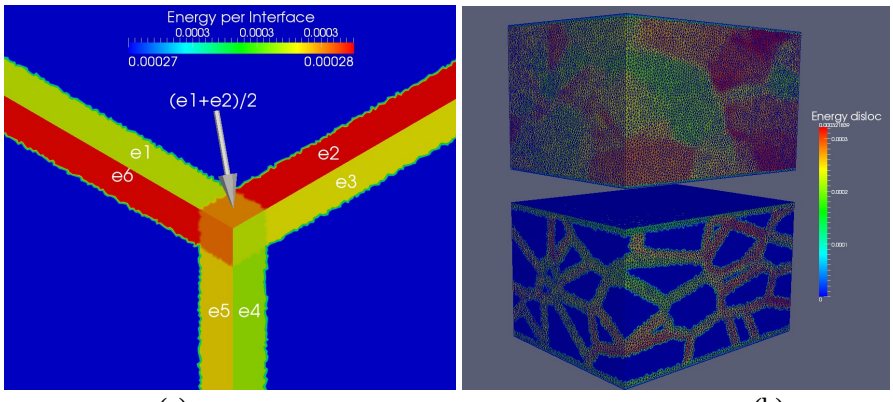
Keywords: *Recrystallization, strain heterogeneity, interfaces, Level set method.*

In addition to the curvature driven grain boundary motion, the stored energy gradient contributes to the driving force of the recrystallization affecting its kinetics and, hence, determines the evolution of the microstructure in the material under thermomechanical processing. The Level Set (LS) method, characterized by implementation simplicity and ability to automatically handle topological changes, was successfully applied for the full field modelling of the static recrystallization with nucleation and Zener pinning phenomenon in two and three dimensions while averaging the stored energy per grain [1,2]. In the present work, the effect of a heterogeneous intragranular strain field on recrystallization in single phase FCC alloys is assessed by numerical simulation using the LS method coupled with a crystal plasticity model in a finite element framework. Limits and difficulties of the numerical treatment to obtain the stabilized resolution of convection-diffusion equation in the LS approach are considered. Recrystallization kinetics are compared with the results of modelling with averaging the stored energy per grain, per grain periphery and per interface. For the computation of stored energy averaged per interface, a technique for detecting neighbouring phases has been developed based on the extension of the phase index field up to the thickness of interface zone (see Fig.1). After computing the energy for each interface, additional averaging is performed in the multiple junction zones (shown in Fig.1) which enables to make use of velocity smoothing in the recrystallization calculation proposed in [1]. The first results show that the recrystallization kinetics are faster in the simulation with averaging the energy per grain periphery and per interface in comparison to the approach in which the energy is averaged per grain.



(a) (b)

Figure 1: a) Schema of stored energy averaging in 2D for a triple junction and b) stored energy in the deformed 3D aggregate computed by the crystal plasticity model (top) with corresponding energy per interface (bottom).

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FINITE ELEMENT MULTIGRID AND ADAPTIVE MESH APPROACHES TO NUMERICAL SIMULATIONS OF THE COUPLED PHENOMENA RELATED TO MAGNETO-THERMOELECTRIC EFFECTS IN SOLIDIFICATION OF METALS

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Keywords: *Magneto-thermoelectric phenomena, alloys solidification, FEM simulation.*

During solidification of a liquid metal which is usually performed with high thermal gradients, some properties like electrical and thermal conductivity and thermoelectric power change between the liquid phase and the solid phase, which induces a thermoelectric current in both media [1]. Superimposition of a static magnetic field leads to the appearance of thermo-electro-magnetic forces (TEM forces) both in the solid and in the liquid which may alter the solidification structure [2], [3]. Complete simulations of the coupled phenomena which include eddy current and convective heat transfer which occur in the liquid, transport of the solid particle and, in addition, phase transitions, remain challenging. Although one can find several publications on the subject [4], [5], the models there are usually simplified. The problem arises because of a large difference in characteristic scales of coupled phenomena: small scale for the thermoelectric and eddy current and large scale for convective transport. To overcome these difficulties in our simulations we applied a FEM-based approach with adaptive meshes and a FEM-based approach with multiple superimposed grids. The first one allowed us to demonstrate a restrictive effect of the eddy current appearing in the liquid. Because of it, a straightforward increase of the imposed magnetic field does not allow one to reach arbitrarily large TEM force in the system, on the contrary, the thermoelectric current decreases. With a multigrid approach we studied displacement of the particle under the action of the TEM forces in a solidification domain (fig.1).

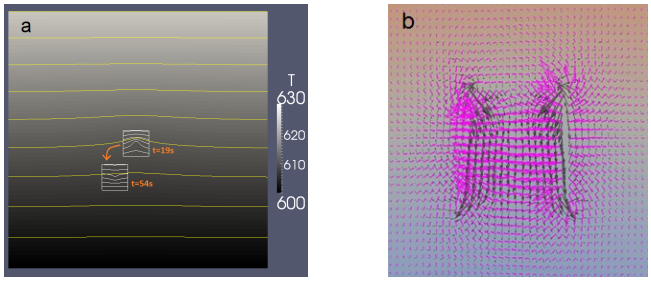

Figure 1: (a) Displacement of a metallic particle in the liquid due to the action of the TEM force; (b) zoom on the particle: vectors of the thermoelectric current (gray) and the TEM force acting on the particle (magenta)

Acknowledgments
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MULTIPHYSICS AND MULTISCALE MODELING OF SOLIDIFICATION PROCESSES

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Keywords: *solidification, defects, modelling, multiphysic, multiscale*

In the metallurgical production chain, the solidification of the molten metal during casting sets the basic crystalline microstructure of the material. The modification of this structure during subsequent processing steps is limited. During solidification several types of defects form, such as heterogeneities of chemical composition: microsegregation at the microstructure scale, macrosegregation at the scale of the product; heterogeneities of structures in terms of grain orientation, size and morphology [1]. The reduction of these defects is an important issue for the industry because they impair the final properties of products.

Process-scale models of solidification processes need to account for couplings of different physical phenomena across several length scales. This requires a multiscale description of crystal growth, of formation of dendritic growth morphologies, and of multiphase heat and mass transfer and fluid flow across several regimes of multiphase transport. Microscopic phenomena can be incorporated into macroscale models by various homogenization techniques; the volume averaging method is often used. This requires a careful simplification and averaging of the microscale phenomena and interactions [2]. Moreover, efficient and robust algorithms that can solve a large number of strongly coupled partial differential equations are needed for the solution of the models [3]. In our presentation we introduce the existing models and their fundamental principles. We present the most recent applications of these models to industrial solidification processes (casting of steel ingots, direct-chill casting of aluminium alloys). We demonstrate their ability to help in the understanding of complex phenomena, such as the competition between nucleation and growth of crystal grains in the presence of convection of the liquid and of grain motion, and we discuss their predictive capabilities. Finally, we address the key remaining questions for future research.

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MICROSTRUCTURE EVOLUTION DURING SEVERE PLASTIC DEFORMATION OF METALS; EXPERIMENTS AND MODELING

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Keywords: *Severe plastic deformation, ultra fine grains, microstructure, texture*

During severe plastic deformation (SPD) crystalline metallic materials undergo a grain fragmentation process. Extreme small grain sizes can be reached by SPD techniques, down to 100, or even 1000 times smaller then the initial ones. The immediate effect is an increased yield strength of the metal, due to the Hall-Petch effect. There are also tremendeous changes in the disorientation distributions and in the crystallographic textures.

Experimental results will be reported on the fragmentation process of several material classes, including fcc, bcc and hcp metals. The techniques used are Equal Channel Angular Extrusion, High Pressure Tube Twisting and High Strain Rolling. A steady state grain size regime is obtained after a large amount of strain. It will be shown that the evolution of the microstructure is strongly related to the disorientation distribution of the first neighboring grains. This distribution correlates also with the GND (geometrically necessary dislocation) density.

A grain fragmentation model is presented which is based on the lattice rotation induced lattice curvature. The model is able to account for, quantitatively, in a single modeling frame, for the refined grain size distribution, the texture evolution, the disorientation evolution and strain hardening. The modeling results are compared to the experiments with good agreements.

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EVOLUTION OF DEFORMATION MICROTEXTURES IN ALUMINIUM: EXPERIMENT, SIMULATION AND MODELLING

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Keywords: *microtextures, crystal plasticity, EBSD, finite element method, aluminium*

The evolution of the lattice orientations of 182 individual grains of an aluminium polycrystal deformed in hot plane strain compression was analysed by a “microtexture tracking” experiment. The experiment consists in following by EBSD the orientations and rotations of grains on the internal surface of a split sample. This was carried out in successive steps to obtain orientations maps of the grains at strains of 0.2, 0.4, 0.8 and 1.2, as well as the initial, underformed state [1]. At each strain, several thousands of orientation measurements were obtained. The orientation maps are provided in Fig. 1a.

In parallel, the deformation was simulated using the crystal plasticity finite element method, on a polycrystal with the same orientations than in the experiment [2]. The polycrystal was represented by a 1000-grain Voronoi tessellation whose 182 central grains were assigned the experimental orientations. The polycrystal was finely mesh to enable intra-grain orientation distributions to develop. At strains of 0.2, 0.4 and 0.8, the polycrystal was fully remeshed to correct excessive element distortions. The simulation was run up to a strain of 1.2 and provided orientation maps (as in the experiment) but also mechanical data (slip rates, …). The orientation maps are provided in Fig. 1b.

The experimental and numerical results were compared using different metrics [2]. First, the average disorientations were found to grow rapidly at the beginning of the deformation and then to saturate at large strain. Second, the orientation distributions were found to exhibit some anisotropy. The principal axes of the orientation distributions were found to distribute about TD at low strain and RD-ND at large strain, in both experiment and simulation. Theoretical models are proposed to explain this trends: the low-strain, TD preferential disorientation axes are properly reproduced by considering the effect of the stress distribution associated to dislocation and/or grain interaction effects (assumed isotropic) combined with the non-linear slip behaviour. The large-strain, RD-ND preferential disorientation axes can be related to the first principal direction of the gradient of the reorientation velocity field about the stable orientation fibres. These mechanisms can be combined into a “microtexture prediction model”.

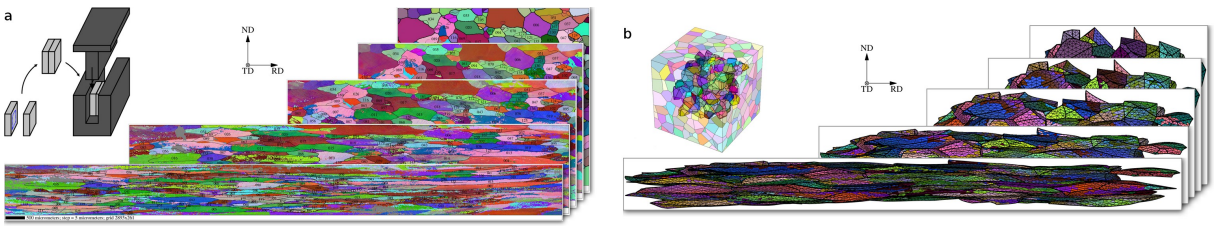

Figure 1: (a) Microtexture tracking experiment and (b) crystal plasticity finite element simulation.

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NUMERICAL SIMULATION OF ELECTROMAGNETIC PULSE SURFACE TREATMENT

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Keywords: *Surface treatment, multiphysic simulation, residual stress.*

Peening methods like shot peening and laser shock peening are used to introduce superficial compressive residual stresses in mechanical parts. These residual stresses prevent the initiation and growth of cracks, and hence improve the fatigue life of mechanical components. Electromagnetic pulse peening (EMP) is a similar process of surface treatment that could be used to introduce compressive residual stresses in metals by the generation of a high transient electromagnetic field [1]. The system of EMP is composed of a flat spiral coil placed near the workpiece surface and connected to a pulse generator. The pulse generator produces a current pulse by the charge and discharge of a capacitor bank. The current travels through the tool coil and a variable electromagnetic field is created between the coil and the workpiece. When the electromagnetic pulse penetrates a conductive material, eddy currents are generated and the workpiece is subjected to Laplace forces. These internal forces, which depend on the characteristics of the current (maximum intensity and frequency), can deform plastically the material superficial layers if its yield strength is overcome. Compressive residual stresses are created after the recovery of the elastically deformed sublayer. The aim of this study is to build a numerical model in order to predict the residual stresses resulting from an electromagnetic surface treatment to study the feasibility of the process.
A sequential coupled electromagnetic-mechanic, 2D axisymmetric model has then been set up with the commercial finite-element software SYSWELD. The total calculation time is subdivided into many steps and, at each step, electromagnetic and mechanical calculations are performed. During the electromagnetic calculation, the transient magnetic flux density and forces are calculated by solving Maxwell's equations. These forces are then used as loading conditions for the structural analysis by FEM. According to the deformation of the workpiece, the geometry of the model is updated for the next time-step calculations and so on until the end of the computation. After validation, the model has been used to simulate a case of introducing residual stresses in a nickel-based alloy 690 thick sample by electromagnetic pulse. The residual stress profile obtained under the coil turn is shown in figure 1. One can see that compressive residual mean stress as high as 50% of the material yield strength, is actually introduced in the metal alloy. Furthermore, the depth of the compressive layer is equal to 5 mm, which is very deep in comparison with conventional peening techniques.

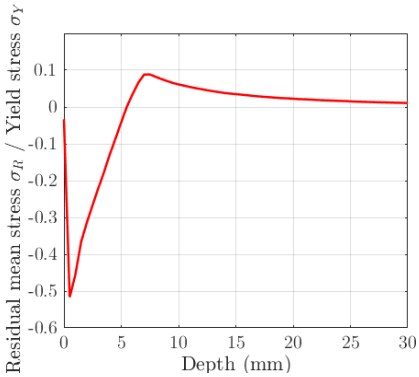

Figure 1: Residual mean stress (normalized by the yield strength) profile under the coil turn

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MECHANICAL BEHAVIOUR AFTER THERMAL TRANSIENT: APPLICATION TO THE CASE OF THE AGE HARDENED SUPERALLOY INCONEL 718 FOR TIG WELDING

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Keywords: *Inconel 718, precipitation hardening, yield strength modelling, TIG welding.*

Inconel 718 is a nickel based superalloy mainly strengthened by the precipitation of intermetallic phase γ'' () D022 tetragonal). The presence of γ'' phase increases the yield strength from 300 MPa for a γ solute solution, to 1000 MPa in the presence of γ''.

It is therefore essential to study the precipitation of γ'' to better understand and predict the mechanical behavior of Inconel 718 alloy. Moreover during welding a partial dissolution of the γ'' phase is observed. In this paper we study how welding affects the precipitation state and mechanical behavior of Inconel 718 alloy. To solve this problem, the link between thermal treatment and mechanical properties via microstructural evolution is investigated.

The numerical model of Kampmann and Wagner (KNW) is used. It is based on the Classical Nucleation and Growth Theories (CNGTs) that have been adapted to model accurately the precipitation of plate shaped particles. This model is implemented in a 'Lagrangian-like' class management software (PreciSo) developed in Mateis and LaMCoS laboratories [1]. Validation has been made with the experimental mean radius evolution (Fig. 1.a)

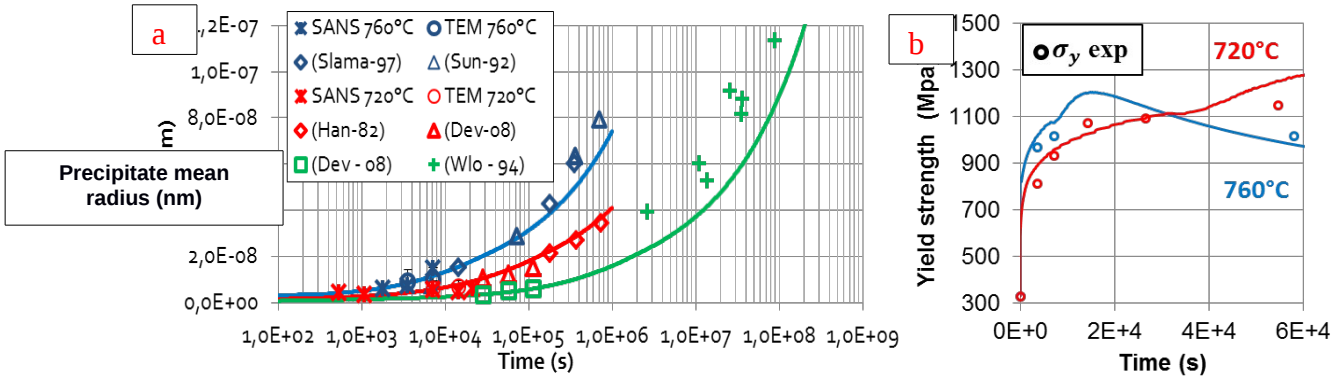

Figure 1: a. Precipitate mean radius evolution with time: precipitation model vs. experimental data. b. Yield strength evolution with time : precipitation+yield strength model vs. experimental data

A model base on the interaction between dislocations and microstructure is proposed in order to predict the yield stress. The classical formulation originally proposed by Friedel [2] has been improved by Kocks [3] and Deschamps [4]. This semi-phenomenological model accounts for solute atoms in the matrix hardening and precipitation strengthening contribution. Validation has been made with experimental data from tensile tests performed on isothermally treated samples (Fig. 1.b) for two temperatures of interest.

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METALLURGICAL MODELS FOR NON-ISOTHERMAL TREATMENTS OF A 6061 ALUMINUM ALLOY

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Keywords: *aluminium alloy, cyclic behaviour, physical metallurgy, welding.*

In age-hardening alloys, high-temperature processes, such as welding, can strongly modify the precipitation state, and thus degrade the associated mechanical properties. The aim of this contribution is to present a coupled approach able to describe precipitation, associated yield stresses and kinematic/isotropic hardening for non-isothermal treatments of a 6061 aluminum alloy. A thermomechanical machine has been specially designed to perform fast and controlled non-isothermal cycles representative of different positions along the weld [1].

The precipitation state (in terms of volume fraction and precipitate size distribution) is modelled thanks to a recent implementation of the classical nucleation and growth theories for needle-shaped precipitates (see [2]). The precipitation model is validated through Small-Angle Neutron Scattering (SANS) and Transmission Electron Microscopy (TEM) experiments. The precipitation size distribution is then used as an entry parameter of a micromechanical model for the yield strength and kinematic/isotropic hardening of the alloy (see [3]).

The major contribution of the present study is to propose a cyclic hardening model that accounts for the whole microstructure distribution, typically both sheared and by passed precipitates, therefore able to describe the post-welding mechanical properties at any point of the weld.

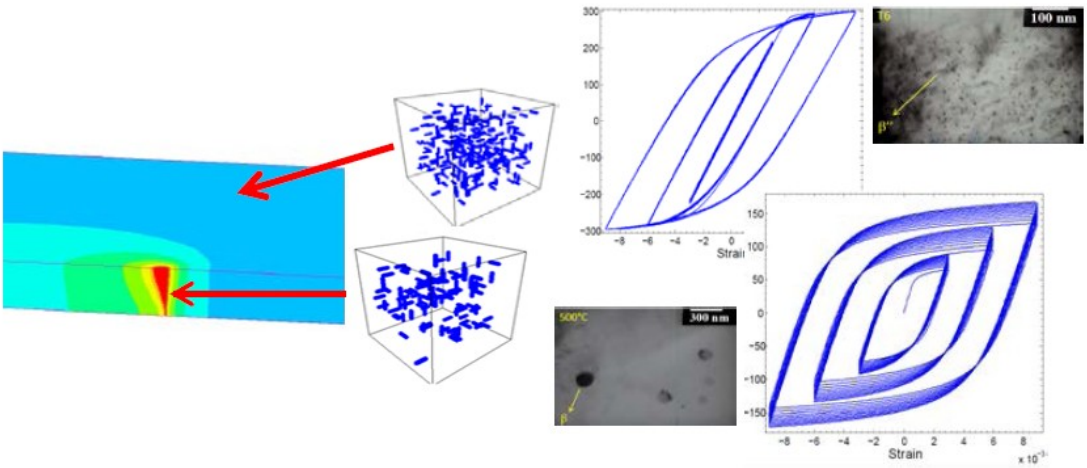

Figure 1: Prediction of the post-welding precipitation state and then cyclic behaviour of the AA6061

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INFLUENCE OF ELASTICITY ON COARSENING KINETICS IN NI-BASED SINGLE-CRYSTAL SUPERALLOYS

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Keywords: *single-crystal superalloy, precipitation, microstructure, elasticity, modeling, pattern formation, phase diffusion*

High pressure turbine (HPT) blades are made of single-crystal nickel-based superalloys, because their specific microstructure provides outstanding mechanical properties at high temperature. This two-phase microstructure consists of a high fraction (typically around 70%) of cuboidal coherent precipitates of the $L1_2$-ordered γ' phase roughly aligned along the cubic directions of the FCC γ matrix.

During gas turbine operation, the thermo-mechanical loadings generally induce changes in the size, morphology, and spatial arrangement of the γ' precipitates, which deteriorate the mechanical properties of the blade. Thus, the optimization of the service life of HPT blades requires an accurate control of the microstructure evolution, which could be achieved by the prediction of relevant models at the macroscale.

However, the development of such models constitutes a great challenge, mainly because elasticity plays a major role in the evolution of this microstructure. Indeed, the crystallographic misfit between the γ' precipitates and the γ matrix gives rise to long-range and anisotropic elastic interactions between the precipitates [1], leading to attraction, repulsion, alignment and merging of the precipitates. Therefore, the precipitates shape and their spatial arrangement are fundamental ingredients of the evolution process, thus preventing the use of the conventional precipitation models [2].

Hence, the improvement of models at the macroscale requires the introduction of relevant parameters to describe the spatial organization of the precipitates, and the development of evolution equations for these parameters.

Within this scope, we introduce parameters derived from pattern formation theory [3], called phases, for the topological description of the γ/γ' microstructure. The elasticity-driven evolution of the system during coarsening may then be investigated through that of its phases, which display slow variations in space and time. To better understand how elasticity affects coarsening kinetics through the spatial arrangement of the precipitates, we carry out phase field calculations of ideal and perfectly regular microstructures undergoing slight perturbations of the precipitates arrangement, during an isothermal heat treatment. The response of the system against Eckhaus and zigzag perturbations is discussed and quantitatively analyzed by means of the calculated phases of the pattern.

Our work shows that the slowly varying phases are relevant microstructure descriptors for Ni-based single-crystal superalloys and could therefore be used to develop a microstructure modeling to the macroscale.

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STATIC AND DYNAMIC STRAIN AGEING IN METALLIC ALLOYS

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Keywords: *Portevin - Le Chatelier effect, Lüders band, visco-plastic instabilites, strain localisation*

Many metallic materials exhibit serrations on their tensile stress – strain curve. The so-called Portevin - Le Chatelier (PLC, [1]) effect occurs in a given range of temperature and applied strain rate. This effect is due to strain ageing defined as the interaction between mobile dislocations and solute atoms [2]. Under certain conditions, this effect can induce a negative strain rate sensitivity of the material in a given range of temperature. The strain ageing phenomenon can be simulated using a phenomenological model denoted KEMC proposed by Kubin, Estrin and McCormick [3] based on two internal variables: the cumulated plastic strain rate, and the ageing time related to the interaction between dislocations and solute atoms.
Strain ageing is also responsible for the well known yield peak and Lüders band phenomenon that occurs in some metallic alloys at the elastic to plastic transition. The KEMC model is also suitable to simulate this effect during finite element simulations of tensile specimens.
The KEMC model has been used in many study to simulate Lüders and PLC effect in many metallic alloys such as aluminium, steels, titanium, zirconium, nickel and cobalt based superalloys,... Some numerical difficulties have to be overcome since the model in very unstable by nature [4]. The work presented here is a review of the main experimental and numerical features of strain ageing phenomena, based on some examples observed and simulated on the metallic alloys aforementioned.

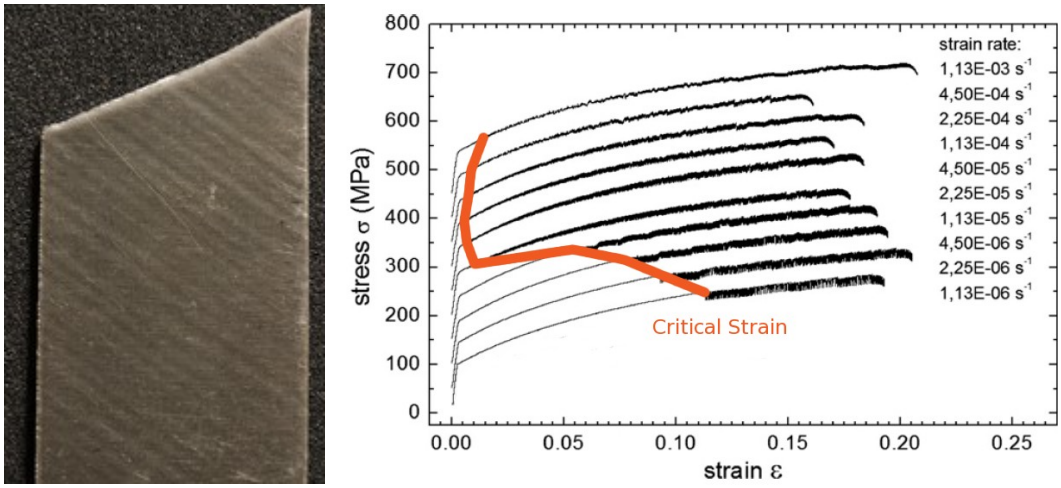

Figure 1: Portevin - Le Chatelier effect in an alumium alloy

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MODELING OF PHASE TRANSFORMATIONS IN TITANIUM ALLOYS

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Keywords: *Ti alloys, phase transformations, Widmanstätten structures, phase-field models, elasticity.*

Titanium alloys are commonly used in the aerospace industry for their excellent specific mechanical properties. These properties, relying on particular two-phase microstructures, are achieved by complex heat and thermo-mechanical treatments. To optimize these treatments targeting at the best properties, it is mandatory to have a deep understanding of the microstructure evolutions ensuing from the β (bcc) to α (hcp) phase transformation.

Among the different microstructures resulting from this transformation, the Widmanstätten morphology (Fig.1a) is of particular interest from both technological and fundamental points of view. It displays specific features that are shared by similar microstructures in other metallic alloys such as steel or brass.

(i) It is composed of parallel plates of the same crystallographic variant gathered in colonies.

(ii) Colonies appear at former grain boundaries of the mother phase.

(iii) The plates grow at a constant rate under isothermal conditions.

Although numerous investigations have been devoted to explain the features listed above, a number of puzzling issues remain to be solved. In particular, there is currently no satisfying theory able to predict the growth kinetics of such structures.

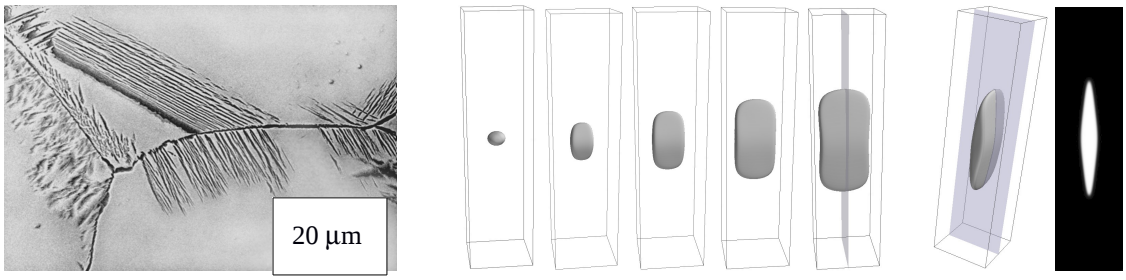

Figure 1: (a) SEM micrograph of Widmanstätten colonies in β-Cez Ti alloy (courtesy of E. Aeby-Gautier); (b) Growth of a Widmanstätten plate in Ti predicted by the phase field model (snapshots at increasing times in a $0.1\times0.1\times0.4$ µm^3 box). (c) Side view and section across the plate exhibiting the aspect ratio observed in (a).

It is usually assumed that the Widmanstätten plate tips adopt a parabolic shape, because it is one of the few that are preserved during diffusion controlled growth in a supersaturated matrix if the interface is a concentration level set. In that case, by solving the diffusion equation, the supersaturation can be related to the product of the tip radius by the lengthening rate, and the selection of a unique radius must rely on some extra ingredient that is not accounted for. Recently, phase field calculations have shown that the selection is ensured by the anisotropy of the elastic energy generated by the mismatch between the crystal structures of the coexisting phases [1].

In the present contribution, we will substantiate this striking conclusion by additional 2D and 3D phase field calculations and additional analyses. In particular, we will show that the tip shape and radius correspond to the equilibrium morphology determined mainly by the elastic energy.
Finally, we will show that the 3D growth process of Widmanstätten plates may involve different stages (Fig.1b) and complex scenarios depending on the initial conditions.

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REPAIR BY ADDITIVE MANUFACTURING FOR AIRCRAFT PARTS MADE WITH LIGHT ALLOYS - INFLUENCE OF ARC WIRE PROCESS

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Keywords: *additive manufacturing, arc wire processes, aluminium alloy 6061*

This study aims to compare different arc wire processes used reloading with light alloys. Representative sections of the part to repair have been refilled using different welding processes MIG. Several derivatives were tested MIG: classical MIG with smooth current, MIG with pulsed current MIG Cold Metal Transfer (CMT)®. The different results were compared to result from TIG process. Aircraft part to repair was in 6061 in T6 state. We had to find the charging process and the recharging strategy which affected the least the substrate to recharge. The Figure 1 present the influence of the different processes on the mechanical properties of the substrate.

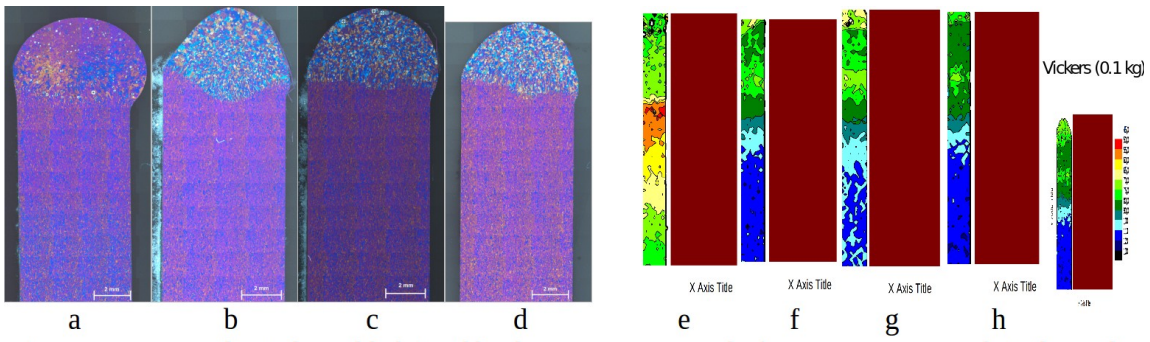

Figure 1: macrography (polarized light) and hardness map respectively for TIG process (a and e), classical MIG process (b and f), pulsed MIG process (c and g) and MIG CMT process (d and h).

We determined by infrared multispectral thermography the fields of temperature and we linked them to the microstructural changes and mechanical properties.

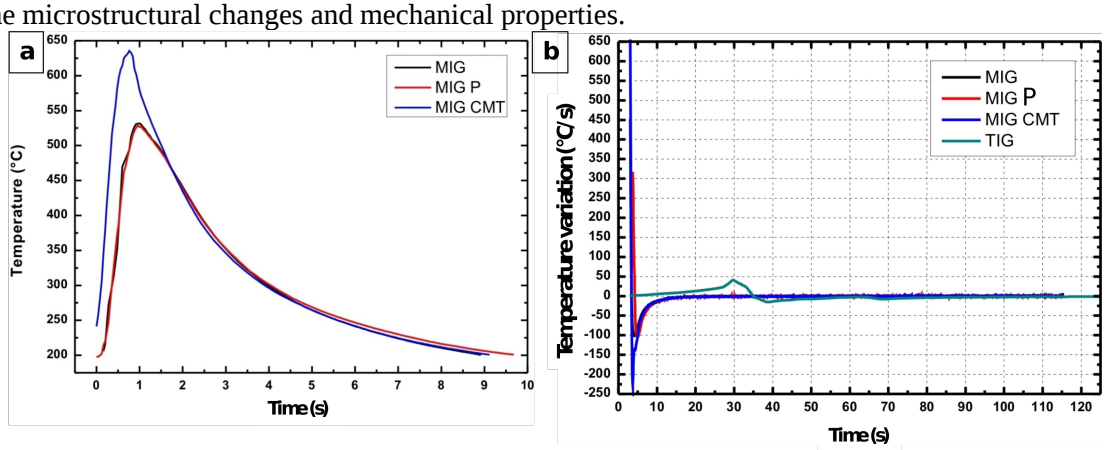

Figure 2: thermogrammes (a) and heating and cooling speeds for the different processes. The measurement position is at 1 mm under the top surface of the substrate.

We were able to determine the charging parameters for the best process that allowed to keep the integrity of the substrate while giving a repair without defects.

DISSIMILAR METAL JOINING BY FRICTION STIR WELDING BETWEEN TITANIUM AND ALUMINIUM

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Keywords: *FSW, aluminium, titanium, intermetallic.*

Friction Stir Welding (FSW) is an assembly technique between two similar or dissimilar material parts. Unlike the classical welds, FSW occurs without additional metal and does not reach the melting point of the metal [1]. It creates a joint between the pieces, strongly flanged placed on an anvil, using a tool which pierces and stirs the interface. Since FSW development, this technology easily finds fields of application such as automotive, railway and maritime industries. It also attains crucial field as air transport, aerospace, mass transport and nuclear industries. However, the industrialization is mainly found for similar metals and mostly for aluminium.

The objective of this research is to understand the junction method on dissimilar materials, to observe the interface and the behaviour of the matter. Previous preliminary works demonstrated that, concerning the couple stainless steel/aluminium, the creation of intermetallics, and Al river shapes strongly influence the mechanical properties of the welding. The river formation phenomenon strengthens the cohesion and acts on the mechanical strength. Besides, the process quickness enables to create one or two kinds of ultra-thin intermetallics [2].

In order to deepen the knowledge in the field of dissimilar FSW and intermetallic growth, the study focuses now on the Al/Ti couple [3]. Tensile tests, microscopy, SEM, EDS are used to characterize the weld. The aim is to find optimal parameters for heterogeneous welding and to link it with the (Ti, Al) intermetallic nucleation and growth (fig. 1). First results show that it is possible to compare and correlate the materials behaviour during the procedure, especially at the metallurgical interfaces.

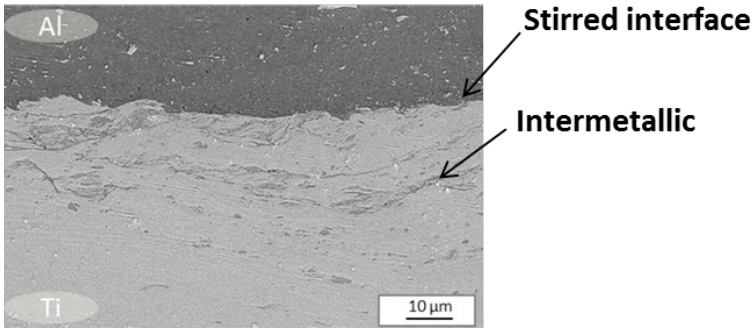

Figure 1: SEM observations of Ti-Al welded interface.

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SYNTHESIS OF NANOCRYSTALLINE NICKEL VIA SPARK PLASMA SINTERING FROM A STARTING NANOSTRUCTURED POWDER OBTAINED BY BALL-MILLING

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Keywords: *ball-milling, nanostructured nickel, SPS.*

The specific properties of nanocrystalline materials have enhanced the study of the different ways to synthesize them and the impact of the processing techniques on their microstructure and their properties. These methods include electrodeposition, equal-channel angular pressing, high pressure torsion or Powder Metallurgy (PM). In PM there are different possibilities and Spark Plasma Sintering (SPS) is a very interesting option [1], [2] due to its capacity to increase sintering kinetics and synthesize nearly fully dense samples with low residual stress.

In this research, ball-milling experiments were performed to find the optimal parameters (speed, time, mass of methanol) to obtain a nanostructured nickel powder. This was followed by the study of how different SPS parameters, mainly sintering temperature and holding time at maximum temperature, change the microstructure of the samples (Figure 1). Both of these processes, ball-milling and SPS sintering, imply many variables that have a big impact on the resulting product.

From ball-milling, activated powders with high defect density are obtained. These properties can be beneficial to increase the driving force that controls the sintering process. The resulting powders were studied by SEM and XRD showing a nanometric microstructure. After SPS, the evolution of the microstructure was studied by hardness measurements as well as XRD. It was shown that the resulting samples exhibit a grain size of several hundreds' of nanometers with very low residual stress levels.

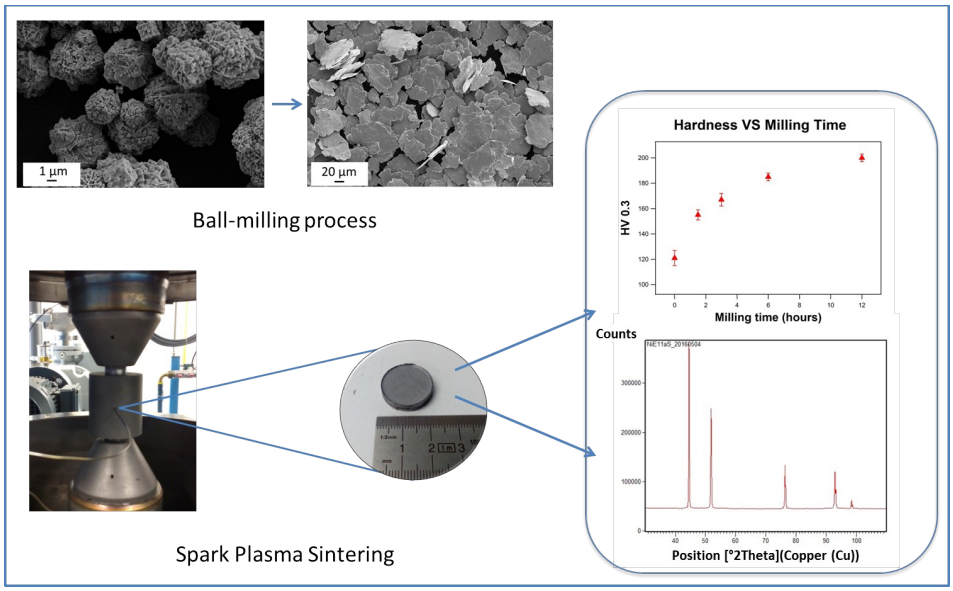

Figure 1: Synthesis route towards nanocrystalline nickel processed by SPS

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SIMULATION OF STATIC RECRYSTALLIZATION AND GRAIN GROWTH PHENOMENA BY MEAN FIELD AND FULL FIELD MODELING

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Keywords: *Grain growth; Mean field modeling; Full field modeling; Level set*

The improvement of material properties is a permanent concern in the industry. Metallurgists have long observed that the macroscopic material properties such as ductility, strength and hardness are strongly related to the microstructure and especially to the mean grain size <R>. Thus, understanding the phenomena of static recrystallization (SRX) and grain growth (GG) is crucial when it comes to optimize the microstructure and the final in-use properties of the material. It is commonly accepted that single-phase fully dense polycrystals are described by a log-normal grain size distribution (LN-GSD) [1] that is driven by <R> and a standard deviation σ. LN-GSD can lead to a quite homogeneous microstructure composed of similar grain sizes (for low values of σ) or heterogeneous microstructure (for high values of σ). Consequently, the σ value has a significant influence on the grain boundaries curvature and thus on their kinetics.

In the present study, we propose to quantify the influence of the initial grain size distribution (GSD) on different mean field models predictions in the contexts of 3-D GG and SRX. For this purpose, the predictions of the Burke and Turnbull (B&T) [2] and the Hillert [3] models for GG and the Johnson-Mehl-Avrami-Kolmogorov (JMAK) [4] model for SRX are confronted with full field (FF) numerical simulations of 10.000 grains [5]. Several initial GSDs are considered, either log-normal or bimodal. To our knowledge, the impact of the initial GSD has never been investigated in 3-D, as other authors generally consider a unique initial GSD [6, 7] or a 2-D framework [8].

Based on the FF simulation results, new formulations of these MF models have been proposed. Contrary to the initial one, the new B&T formulation takes the initial grain size dispersion into account. It has been proven able to predict accurately the evolution of <R> for any LN-GSD, regardless of σ. We also confirmed that the Hillert model is more versatile because it considers interactions between grain classes. Nevertheless, the inherent assumptions in this model lead to the existence of a first-order parameter which needs to be finely calibrated. Numerical FF investigations have permitted to propose a new value for this parameter, which remains globally constant for all initial distributions. Thus the new Hillert formulation predicts accurately <R> and the evolution of the GSD. Finally, the JMAK law has also been shown to be imprecise in many cases of SRX.

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ELECTRO-THERMAL CHARACTERIZATIONS OF WELDING PROCESS – PARAMETERS INFLUENCE ON THE WELDING OF ALUMINUM ALLOY WITH STRUCTURAL HARDENING

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Keywords: *welding process, multiphysics characterisation.*

Welding is a joining technology which covers a broad field of science: electricity, magnetism, fluid mechanics, materials chemistry, metallurgy, mechanics, ...
Welding processes are often referred to as multiphysics processes. Therefore, to study these processes of assembly should be set up a certain number of characterization tools during the welding operation covering all scientific areas mentioned above.
We have developed a welding bench, all processes electric arc equipped with a high-speed optical camera and a fast camera Infrared Multispectral (Figure 1). On the bench, we have synchronization between the electrical parameters of welding, visualization of the movements on the surface of the melt and the transfer of filler metal (Figure 2) and thermal field (Figure 3) on the surface of the assembled part.

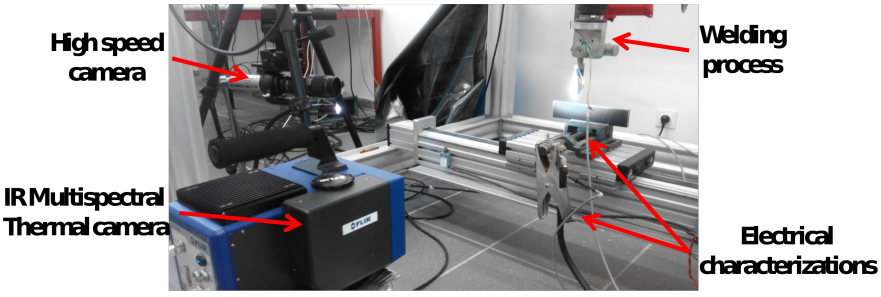

Figure 1: visualisation of the installation.

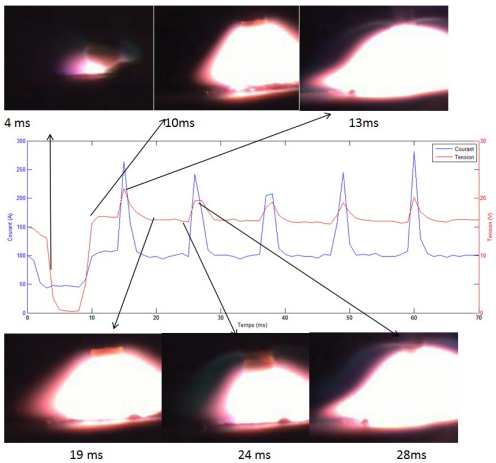

Figure 2: synchronisation between electrical parameters and arc visualisation for MIG CMT Pulsed.

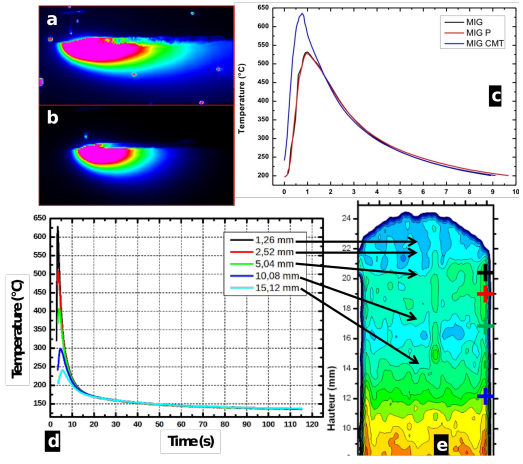

Figure 3: IR visualisation for MIG CMT (a) and Pulsed MIG (b); temperature variation for different welding process (c); relation between temperature and mechanical properties (d).

Thanks to the data obtained, we can predict the properties of use of welded joints but these data are invaluable for validating models and multiphysics simulations of welding processes.

COMPARATIVE STUDY ON MICROSTRUCTURES AND MECHANICAL PROPERTIES OF THE ALUMINUM-SILICON CASTING ALLOYS IN AS-CAST AND AFTER HEAT TREATMENT.

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Keywords: *Microstructure, Casting, Precipitation, System **AL**-**SI**-Cu-Mg*

Abstract: In the first part of this study, the influence of casting processes of gravity die casting (GDC) and high pressure die casting (HPDC) in AlSi10.6CuMg alloy were investigated with the aim to provide a comparative study on the variation of the microstructures and the mechanical properties in as-cast condition and after the heat treatment T6. Among the techniques used for the identification of the all phases in the microstructures obtained by the two types of casting, the SEM equipped with EDX and the X-ray diffraction analyses. In the second part, we are interested in the effect of alloying elements on mechanical properties of aluminum alloys and the various precipitation hardening phases. We present in this part the observations of light microscopy and identification SEM of the different phases of alloys AlSi10.8Cu0.3 and AlSi7Mg0.6. Micro-hardness measurements were carried out after several solution heat treatments at 525 ° C and 500 ° C from 5 minutes to 16 hours and holding for various aging times at 210 ° C of 10 minutes until 50 hours. Following the results of DSC and XRD, a study pushed by the MET was made in order to identify accurately the precipitates responsible for the hardening of these alloys. The experimental results in the first part have allowed affirming that the HPDC has a high and relatively rapid age hardening compared to the GDC process. In the second part, the results determine the best combination of the temperature of solution heat treatments and the time of aging leading to maximum hardness of the studied alloys.

Introduction

Aluminum-silicon (Al-Si) casting alloys are widely used in automotive applications on account of their high strength to weight ratio and ability to be cast into complex shapes. The largest casting processes are gravity and high pressure die casting. The rapid solidification under high pressure die casting produce a consistently dense and highly refined surface structure with excellent mechanical properties [1]. During the solidification process, the impurities and alloying elements in Al-Si alloys partially form various constituent particles including θ-Al_2Cu, Q-$Al_5Cu_2Mg_8Si_6$, β-Al_5FeSi, π-$Al_8Mg_3FeSi_6$, β-Mg_2Si, α-Al(Mn,Fe,Cr)Si and/or other particles under different conditions [2,3].

Results

HPDC decreases significantly the SDAS, refined and modify the form and distribution of the eutectic Si and the (α and β)-Fe intermetallic phases. In this processes, the distinction and identification of the π-$Al_8Mg_3FeSi_6$, Q-$Al_5Cu_2Mg_8Si_6$ and β-Mg_2Si phases is very difficult compared to the GDC. In the second part, the most important element for strengthening in AlSi10.8Cu0.3 and AlSi7Mg0.6 alloys are θ''-AI_2Cu and β''-Mg_2Si respectively.

Conclusion

The values of micro-hardness after the treatment T6 are relatively higher and faster in HPDC than GDC for the same alloy AlSi10.6CuMg. The hardness peak of AlSi7Mg0.6 alloy is remarkably higher than AlSi10.8Cu0.3 alloys, this peaks occur at 8 h of solution treatment followed by 40 min of aging and 15 min of solution treatment followed by 2 h of aging respectively.

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INVESTIGATION OF MECHANICAL PROPERTY GRADIENTS ON HYPER-DEFORMED SURFACES: NANO-MECHANICAL TESTING IN PURE α-IRON

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Keywords: *Mechanical surface treatments, Tribologically Transformed Surfaces (TTS), Gradient of mechanical properties, Nano-indentation, In situ micro-compression testing.*

In the industry, there are several techniques which improve the service lifetime of materials by increasing the local mechanical properties in the near-surface. In the case of mechanical surface treatments (such as impact-based), the material is exposed to repeated mechanical loadings, producing a severe plastic deformation in the surface, and then leading to a local refinement of the microstructure into the affected zone (Tribologically Transformed Surfaces - TTS). The microstructure's transformation is characterized by a progressive increment of the grain size from the surface until the bulk material. Consequently, very interesting physical properties such as high hardness and better tribological properties are exhibit in these mechanically-induced transformed surfaces. Nowadays, it is well-known that the grain size gradient generated provokes an evolution on the mechanical properties in the impacted zone over a few tens of microns. However, a simple micro-hardness test is not quite enough to quantify precisely the engendered variation of mechanical properties due to the heterogeneity of the transformed surface. The main issue of this work is to assess and describe precisely the elastic-plastic behavior and the distribution of mechanical properties on deformed zones of a model material (pure α-iron). In our project, a characterization of the transformed microstructure, as well as a statistics measurement of the grain size distribution on the cross-section of the sample is presented firstly. Afterwards a methodology based on nano-indentation tests (Figure 1) and in-situ micro-pillars compression tests (Figure 2) is implemented to quantify the evolution of mechanical properties starting from the near-surface. A relation between the mechanical properties gradient and the microstructure evolution (grain size, dislocation density, etc.) is established. A comparison between the properties measured by both techniques is also discussed.

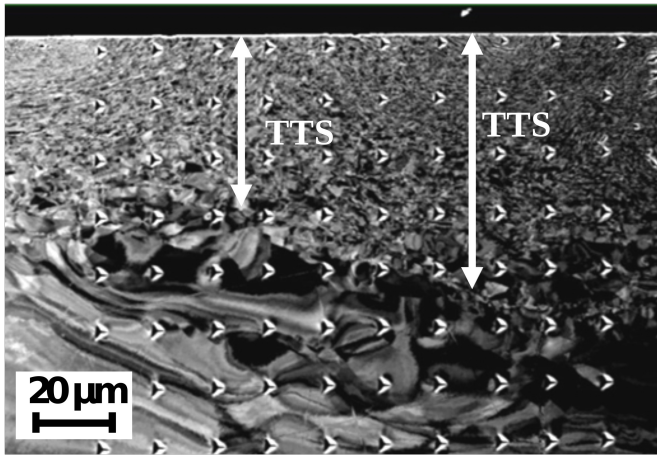

Figure 1: Nano-indentation tests (Berkovich) on a TTS zone in pure α-iron. Cross-section view of the sample.

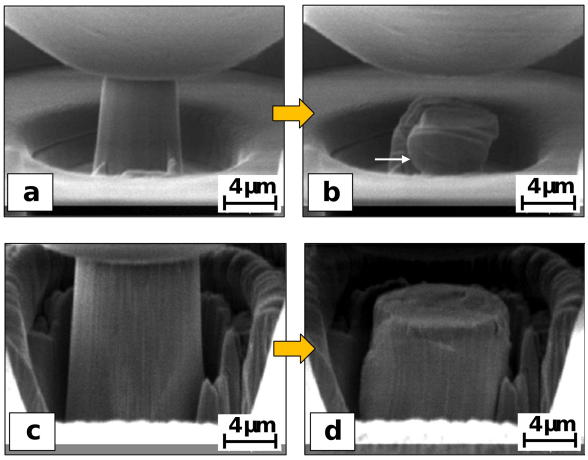

Figure 2: Micro-compression tests in pure α-iron of a single crystal pillar (a,b) and a TTS pillar (c,d), using a flat punch of Φ 15 µm.

QUANTIFICATION OF THE DISLOCATION DENSITY IN NICKEL-BASED SUPERALLOYS

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Keywords: *Nickel-based superalloys, dislocations, intragranular misorientations, EBSD.*

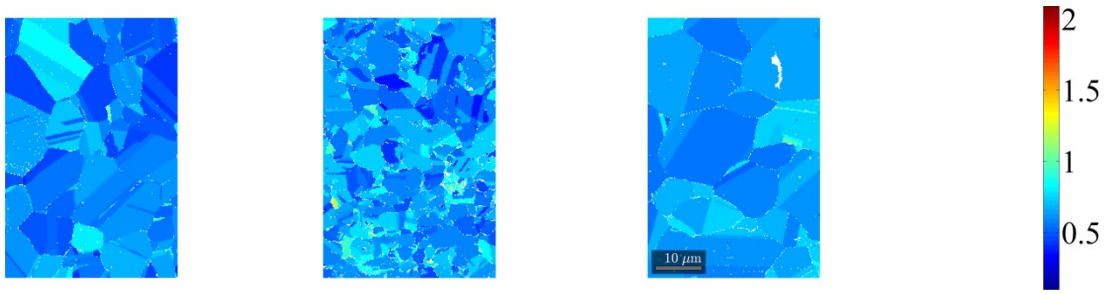
Dislocations greatly influence microstructure evolution during thermal treatments of Nickel-based superalloys, notably with regards to recrystallization and precipitation kinetics. A method is evaluated in the present work for quantifying the distribution of the geometrically necessary dislocations density stored in the material during deformation.

The method consists in analyzing local misorientations in EBSD maps of size 228 µm by 171 µm and a minimum of 10000 grains analyzed with the MTEX [1] Matlab® toolbox.

The post-treatment method used by Moussa [2] and inspired from Kamaya [3] is applied to estimate the dislocation density at each acquisition point.

Eventually, the dislocation density field is characterized following statistical analysis principles. The dislocation density at a given acquisition point is defined as a random variable whose are calculated the unbiased estimators of the expectation and of the mean deviation , and a confidence interval for the probability . The values of and provide orders of magnitude of the mean level and of the scattering of the distribution, respectively. The confidence interval , for example if p = 95 %, provides a range where most values of the distribution lie. Note that no probability law was chosen to represent the distribution in order to preserve the raw nature of the obtained data.

The studied material is the Inconel® 625 superalloy. Samples corresponding to different thermomechanical paths were selected : (a) a solutionized sheet, (b) a solutionized sheet shear spinned then annealed, and (c) an industrial part (aircraft engine nacelle), formed by 10 % cold deformation.



(a) (b) (c)

Figure 1: Grain average misorientation (GAM) maps in degrees and estimated statistical values , , and in materials (a), (b) and (c)

GAM maps and estimated values on Figure 1 show that:

- material (b) presents small grains and a value similar to (a), explainable by its recrystallization during annealing due to the stored energy accumulated during shear spinning,
- material (c) presents slightly higher intragranular misorientations than (a) and a slightly higher value than (a) and (b), which is consistent with the fact that this material was 10 % cold deformed.

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EFFECT OF PROCESSING CONDITIONS ON MICROSTRUCTURE AND MECHANICAL BEHAVIOUR OF SELECTED HEA ALLOYS FROM COCRFEMNNI FAMILY

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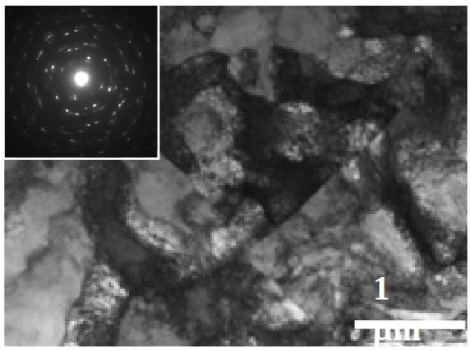
Keywords: *High Entropy Alloys, HEA, CoCrFeMnNi*

One of the latest top subjects in materials science is High Entropy Alloys (HEAs). A general definition of HEAs says that these are solid solutions that contain five or more principal elements in equal or near equal atomic percentage, usually between 5 at% and 35 at% [1]. Often some slight changes in content are made or alloying components are added in small quantities to influence the properties [1,2]. Many of the promising compositions are chosen by modelling and use of thermodynamic calculations softwares, like Thermo-Calc or CALPHAD, even though the calculations are limited by databases built on the idea of interactions between a major element and solutes. First work to analyse multi component alloys was done by Cantor et al. in 1981, where compositions containing from 5 to 20 elements in equiatomic ratios were analysed. As a result they obtained one alloy whose structure was single phased: the $Co_{20}Cr_{20}Fe_{20}Mn_{20}Ni_{20}$. Independent work was made by Yeh et al. whose publications were the first to explain the concept of HEAs by providing experimental results and related theory [3].

In this work two original alloys from the CoCrFeMnNi family with low Co content have been chosen by Thermo-Calc calculations and analysed. Microstructure development and mechanical properties have been described and compared for two series of casts: prepared by cold crucible melting with ultrahigh purity of raw materials in EMSE laboratory and VIM ingots with typical industrial residuals (O, N, C, S) done in the APERAM research center. The ingots have been processed by forging or rolling (hot or cold). For both series of alloys, a single FCC phase has been obtained. Complicated structure at the nano scale level has been revealed after forging (*Figure 1*), which might be at the origin of high mechanical resistance. Grain growth kinetics (after cold rolling) has been shown to slow down for the casts containing impurities. Tensile tests of the as-forged alloys at liquid nitrogen temperature have proved an increase of both strength and ductility without martensitic transformation. New non-equiatomic HEA grades have been proved to present very good mechanicla properties at a wide range of temperatures and favourable phase stability. Prospect for further study is highly promising.

a) 

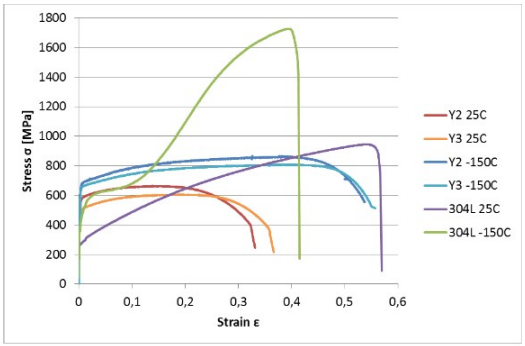
b) 

Figure 1: a) TEM image of microstructure after hot forging; b) stress-strain curves after traction in room temperature and -150°C, comparission between as firged new compositions Y2 and Y3 with classical SS 304L

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CORROSIVE ATTACK OF NICKEL-BASE SUPERALLOYS AND THEIR OXIDES BY ELECTROCHEMICAL METHODS: INFLUENCE OF COMPOSITION, TEMPERATURE AND CHLORIDE

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Keywords: *alloy design, high temperature oxidation, corrosion, superalloys.*

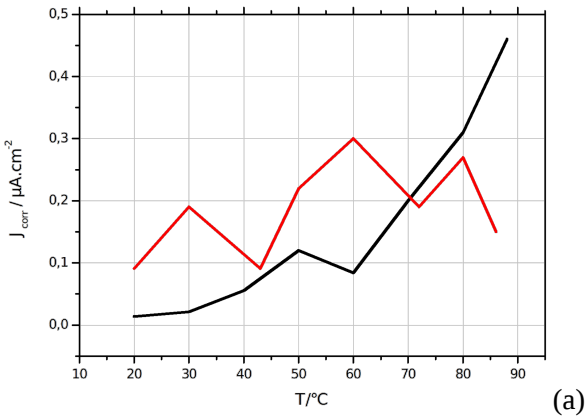
Nickel-base superalloys are known for their good resistance to corrosion in basic or in non-oxidising acidic media; in oxidising atmospheres, they can resist to temperatures even higher than 1000°C. This performance originates from their aptitude to self-protect by forming an oxide scale, and from the ability of this scale to resist a subsequent dissolution in the case of wet corrosion (passivation) or, in the case of high temperature oxidation, to remain sound and well attached to the metallic substrate. However, there is so far no global description giving the environmental resistance of a superalloy as a function of its composition. Our work consisted in using electrochemical methods to study the resistance of materials to a corrosive attack in the presence of chloride ions: alloys were immersed in a solution of NaCl at 2M, and a Taffel test was performed every 10°C until reaching the critical point (Fig. 1a). We have characterised alloys of different compositions:

(i) either in the raw metallic state (polished surface),
(ii) or in an oxidised state following an exposure of one week at 900°C in air.

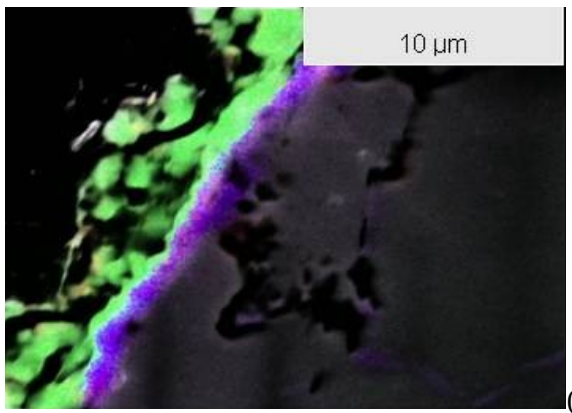
There is a double objective:

(i) in the first case to link wet corrosion resistance to alloy composition,
(ii) in the second case to estimate the quality of the oxide scale that was formed at high temperature, and to link it to alloy composition.

Significant differences were found between alloys, as well as compositional trends. A systematic characterisation of many different alloys and their oxide scales by this method should allow to correlate the performance of alloys (resistance to wet corrosion or to high temperature oxidation) to composition, and to propose environmental resistance criteria in view of a multi-objective optimisation design of novel alloys [1].



(a)



(b)

Figure 1: Illustration of results for alloy 718: (a) corrosion current density as a function of temperature for the polished alloy (red curve) and for the oxidized alloy (black curve). (b) EDS map of Cr near the surface of the oxidised alloy: formation of the Cr_2O_3 scale (in green) depletes the alloy in Cr below the oxide scale, which may explain the decrease in corrosion resistance when testing temperature increases, as measured in (a).

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MECHANO-CHEMICAL SYNTHESIS OF NANOMETRIC REFRACTORY ALLOYS POWDERS

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Keywords: *Mechanically induced chemical reaction, refractory metallic alloys, nanopowders.*

The synthesis of Nb-W, Nb-Mo, Mo-W and Nb-Mo-W stoichiometric refractory alloys has been performed by inducing the reduction of their respective oxides (Nb_2O_5, WO_3, MoO_3) by magnesium, in excess, the reaction being triggered using high energy planetary ball milling, according to the following reaction :

$$WO_3 + 0.5Nb_2O_5 + MoO_3 + 8.5Mg \rightarrow WNbMo + 8.5MgO$$

As these reduction reactions (so-called thermites or more specifically magnesio-thermitic reactions) are extremely exothermic, with a final temperature that may exceed 3500K, adjusting the product final temperature is necessary to avoid extensive melting. This has been done by adding an inert component, namely sodium chloride, to the mixture. Using a ball-to- powder ratio of 15:1, a mill velocity of 500 RPM and tempered steel milling media, the reaction starts within 3 to 12 minutes, depending on the composition, and is characterized by a brutal temperature and pressure increase inside the milling jar.

Products were then leached using 2M hydrochloric acid to remove NaCl and MgO, filtered, rinsed and dried before full characterization. SEM observation revealed a final microstructure of agglomerated particles with a mean diameter within the 80-150 nm range (Figure 1). BET specific surface analysis, X-ray diffraction analysis have also been performed (Figure 2); we will show how it is possible, through an adjustment of our synthesis parameters, to obtain a final product with a single stoichiometric composition, with a near perfect distribution of the 2 or 3 atom types within the alloys, at the nanometric scale.

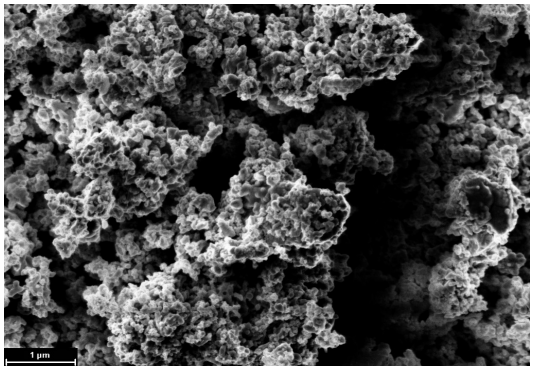

Figure 1: SEM image of the MoNbW powder obtained by mecanosynthesis

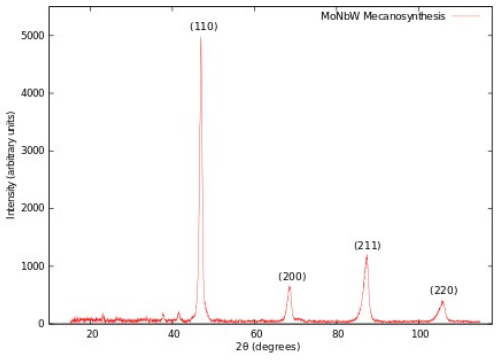

Figure 2: XRD pattern of MoNbW ternary system obtained by mecanosynthesis

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QUANTITATIVE ANALYSIS OF THE MICROSTRUCTURAL HETEROGENEITIES IN AD730™ NICKEL-BASED SUPERALLOY BILLETS

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Keywords: *nickel based superalloy, billet, microstructure.*

The AD730™ new nickel-based superalloy has been designed by Aubert & Duval in order to achieve a better compromise of mechanical properties at high temperature (up to 700°C) and production cost, compared to the currently used superalloys for turbine disk manufacturing [1].

For any alloy strengthened by high contents of many elements, the conversion of the cast ingot does not allow for the removal of all the microstructural heterogeneities in the billet and the resulting microstructure is often only partially recrystallized. The billet is also massive enough to observe microstructural variations along the radius, as a result of thermo-mechanical fluctuations within the volume. Characterizing billet heterogeneities is essential to decide about the further die-forging operations which will lead to the turbine disk. In fact, at the end of the forging process, the disk microstructure has to be fine-grained and homogeneous so as to guarantee optimal mechanical in-service properties. The present work aims to describe both qualitatively and quantitatively the heterogeneities in two industrial AD730™ billets with different diameters (8'' and 12''). The 8'' and 12'' billets are respectively fine-grained and coarse-grained but both present three types of local microstructure which are: large elongated recovered grains (R in Fig.1), equiaxed grains (E) and sub-structured areas (S). While recovered grains are mainly located in the inner billet, sub-structured areas are more frequent at the periphery and especially at the periphery of the 8'' billet. After solution heat-treatments, the three local microstructures are still visible and equiaxed grain sizes are little changed (even if few abnormal grains are noticed); however, intragranular precipitation is modified in each microstructure, consequence of a decrease in the γ' phase fraction. Recovered grains also appear to statically recrystallize in some places; heteroepitaxial recrystallization [2] is observed in recovered grains and sub-structured areas.

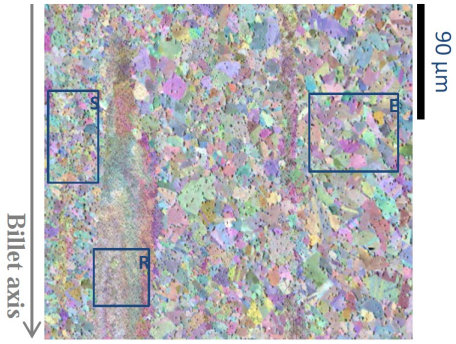

Figure 1: FSD image – Microstructure of the 8'' billet, Mid-radius**.**
E, R and S stand for Equiaxed grains, Recovered grain and Sub-structured area respectively

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FORGING ALONG THREE ORTHOGONAL SUCCESSIVE DIRECTIONS

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Keywords: *cross-forging, recrystallization, texture.*

Forging metals successively along three orthogonal directions at high temperature is a common practice in a variety of contexts. In industrial plants, it breaks up the coarse as-cast structure of ingots. In laboratories, it is used to impose severe plastic deformations to test-pieces. In certain conditions, their shape is not altered at the end of a cycle of three orthogonal passes. In such experiments, the aim was mostly to obtain ultrafine-grained materials. In the Georges Friedel Laboratory (LGF) in Saint-Etienne, the process, also called triaxial or multipass forging , has been applied to several alloys. The aim was to obtain equiaxed and possibly recrystallized grains and to contrive new forging sequences [1] with the following materials:
i) aluminium alloys: a novel, home-made equipment was designed (see Figure 1). Several compressions and upsettings of the test-piece can be performed without intermediary quenching, machining and re-heating. Some grades of aluminium, like AA 7475, are reported to undergo grain-size reduction after multipass forging. This was not the case with the Al-3Mg-Sc(Zr) alloy prepared in the LGF and triaxially compressed to an accumulative strain close to 3. But a very fine sub-structure appeared and the resulting crystallographic texture was a characteristic mix of three major Cube orientations, each rotated by 45 ° around the axes of forging.

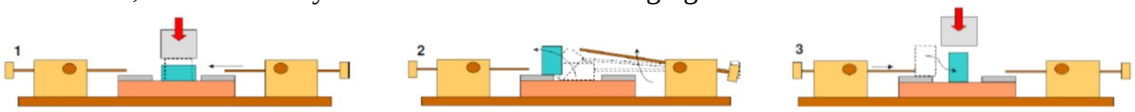

Figure 1: Upsetting of a test-piece by means of alumina rods. They flip the sample on a lower tool in the multipass forging equipment of the LGF

This triaxial equipment does not work above 450 °C. This is not enough for titanium and zirconium alloys which are bcc at high temperature and hcp in usual conditions. With them, an important problem is to break, or globularize, the lamellae which appear during quenching. So they were studied with a classical thermomecanical machine in which the temperature is raised before each compression. The test-piece is quenched after each pass. This quenching is very quick, being performed in-situ [2]. It was applied to:
ii) Zircaloy-4. This alloy is single-phased below 810 °C. It was shown that between 650 and 750 °C, three orthogonal compressions suffice to create a fine (around 20 µm) globular microstructure. No protracted strain hardening is necessary. The results are particularly good when the strain rate is increased (to $4s^{-1}$ for example), because the recrystallization is then almost complete.
iii) TA6V, which exhibits two phases at room temperature. Three compressions in the range of 800 -900 °C followed by cooling produce a mixed microstructure with lamellae and nodules. Their respective proportions can be adjusted to produce materials which are at the same time fatigue and creep-resistant.
Efforts are made now to design an appliance able to perform passes at high temperature without the effects of thermal inertia linked to re-heating. It would allow studying the effect of annealing, which seems most efficient between two compressions, as shown by preliminary attempts.

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NEPER: A FREE SOFTWARE FOR THE GENERATION AND MESHING OF COMPLEX, LARGE-SCALE MICROSTRUCTURES

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Keywords: *Numerical microstructures, polycrystal, meshing, finite element method.*

*Neper is a software for the generation and meshing of polycrystalline microstructure*s [1, 2]. Neper provides inputs for simulations in a wide range of applications, such as materials science, micromechanics, but also magnetism and rock mechanics, using resolution methods such as FEM, FFT, DEM, … [2].

Neper can generate microstructures of different complexities. First, *an advanced model using Laguerre tessellations in an optimization context enables to generate microstructures from experimental data.* For example, grain size and shape distributions can be provided (Fig. 1a). Another possibility is to use synchrotron X-ray diffraction (or FIB-SEM) data, such as grain centroids/volumes (obtained by 3DXRD) or full grain images (obtained by DCT or FIB-SEM). Second, *multiscale microstructures can be generated, which are characterized by one or several subdivision(s) of the grains*, as typically found in steels or Ti alloys. These are modelled by applying tessellations in the cells of a previous tessellation, an arbitrary number of times (Fig. 1b). Finally, the two capabilities can be combined for controlling the morphological properties at all microstructure scales.

Neper meshes the microstructures with controlled element sizes and shapes, for small-strain or large-strain simulations. This involves 3 steps: *(i) regularisation*, which consists in cleaning the microstructures of its smallest details, *(ii) multimeshing*, which consists in meshing each grain independently using several algorithms until a minimal mesh quality is reached, and *(iii) remeshing*, which consists in constructing a new mesh from a deformed mesh.

Neper can be downloaded from http://neper.sourceforge.net.

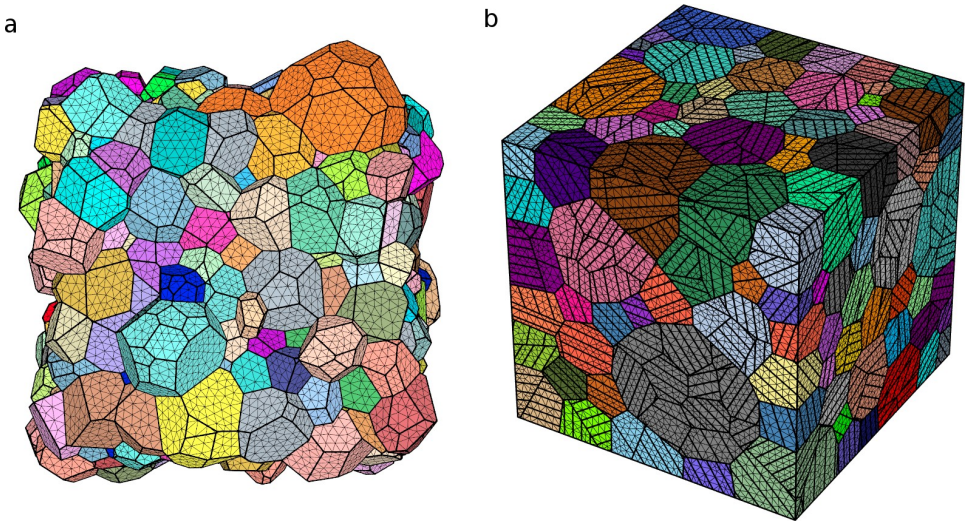

Figure 1: (a) Microstructure generated from experimental grain size and sphericity distributions. (b) Multiscale microstructure representing a bainite microstructure (uniform colour for the primary grains). On both images, a finite element mesh is shown.

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SCRAP ALUMINIUM BASED COMPOSITES THROUGH COMBINED METHOD OF POWDER METALLURGY AND THIXOFORGING

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In general, the design problem of damping materials is how to work the damping mechanisms associated with the stress-induced movements of the general microstructural defects. Principally, certain structural defects such as the dislocation and the grain boundary defects are more extensively employed owing to their relatively high specific damping level. However, it is difficult to find a high damping capacity and good mechanical properties at the same time in the same materials structures. In the literature, it was shown that the damping mechanism of point defects will not weakens the mechanical properties contrary improves the damping capacity of the materials. Additionally, Aluminium alloys generally show a moderately low damping capacity. Metal Matrix Composites (MMCs) techniques can provide adjustments to the microstructure of the matrix and also interfaces reinforcement/matrix. For this reason, Aluminium based composites very often used as MMCs are reinforced with certain particulates for improving damping capacities. Some commonly used particulate reinforcements, such as Graphite - Gr, Al_2O_3, TiC, etc., can improve the damping capacities of these composites.

In the frame of the present work, scrap aluminium chips (A2014 & A7075) were used as matrix and certain reinforcements were added to improve damping capacity of the composites designed as alternative composite materials. Mechanical properties, such as impact behaviour, microhardness evolution were analyzed. Fine microstructural evaluation as well as interface analyses by Scanning Electron Microscopy (SEM) was studied.

SILICA AND TIO2 PARTICLES REINFORCED HYBRID COMPOSITES FROM SCRAP ALUMINUM CHIPS

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Metal matrix composites (MMCs) are largely used in the aerospace industry for different applications very often as joining elements. Al-based (MMCs) are used generally for their high-strength, hardness, and high tribological properties. Certain reinforcements such as silica, TiB2 and/or TiC are also progressively used in the aluminium composites as substitute materials for cylinder heads, liners, pistons, and brake disks in different industrial applications for the high-wearing resistance, light weight, and specific strength in order to reduce the costs of manufacturing parts.

In the frame of the present work; a special manufacturing technique was applied for the processing of the hybrid composite by combining powder metallurgy and thixoforming. As the reinforcement elements silica and TiC and/or TiB2 were added in the matrix obtained from fresh scrap aluminium (AA2014 + AA7075) chips. Impact resistance, mechanical and physical properties were studied by microindentation tests.

RECYCLING OF ALUMINIUM CHIPS FOR LOW COST COMPOSITES BY MEANS OF SINTERED -THIXOFORMING PROCESSES

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Aluminium chips are generally recycled via foundry methods. In this research, the use of a combined method through powder metallurgy techniques + thixoforging has been proposed as a more effective low cost and alternative processing for recycling aluminium chips. AA7075 Al chips and Al2O3 spherical ceramic are mixed as matrix and reinforcement, respectively, and other additives - pure boron powder and TiC in different proportions, are used as well to optimize the composition. The effects of the different additives in the microstructure, mechanical properties and impact choc values are compared. Finally, effects combined processes are also evaluated.

PREDICTION OF DEFORMATION OF A 316L HEAT EXCHANGER DURING DIFFUSION WELDING: INFLUENCE OF GRAIN GROWTH AND ANISOTROPY

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Keywords: *316L, high temperature mechanical behaviour, grain growth effect, anisotropy.*

Stainless steel (316L(N)) compact heat exchangers are developed at CEA for a sodium-cooled fast breeder reactor (Generation IV). The manufacturing process uses the Hot Isostatic Pressing (HIP) technology to assemble sheets of metal by diffusion welding. The high pressure, temperature (up to 1100°C) and long time (several hours) imposed during the process may lead to important deformation of the channels (up to 15%).

To predict the deformations, simulations are developed using ABAQUS. First studies, on simplified exchangers geometries, showed that a Chaboche model [1] (Norton model with isotropic hardening) is well adapted to describe the viscoplatic behavior of 316L. The current gap between experimental and numerical results is less than 20% but still too important for our problematic. The gap is supposed to come from a lack of data on the parameters of the Chaboche model (2 parameters for creep + 3 parameters for isotropic hardening).

A better knowledge of those anisothermal parameters from 20°C up to 1000°C, their dependence on time (effect of grain growth) and on the solicitation direction (anisotropy of the metal sheets), is required to improve the simulations.

Creep properties up to 700°C and short time mechanical properties (high strain rates) up to 1000°C of 316L are well known. However, the considered HIP process involves low strain rates ($< 10^{-4}$ s^{-1}) above 1000°C.

Moreover, grain growth in 316L metal sheets has been highlighted above 1010°C [2]. It is known that an increase of the grain size decreases the tensile properties, according to the Hall-Petch relation, at room temperature [3], up to 750°C [4]. At higher temperatures, an opposite effect might occur [5]. Concerning creep resistance, the grain size effect is still uncertain but in many cases an increase of grain diameter enhances the creep resistance [6].

As a consequence, improvement of the Chaboche model for 316L sheets requires new data.

Investigations on 3 mm thick sheet above 900°C will focus on :

- Creep and tensile properties at low strain rates ($< 10^{-4}$ s^{-1})
- Grain growth effect
- Anisotropy of the metal sheets

According to the results, a grain growth and anisotropy dependence of the Chaboche model should be introduced.

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MECHANICAL CRITERIA OF FORMATION OF SEVERELY DEFORMED SUB-SURFACE LAYERS UNDER TRIBOLOGICAL STRESSES

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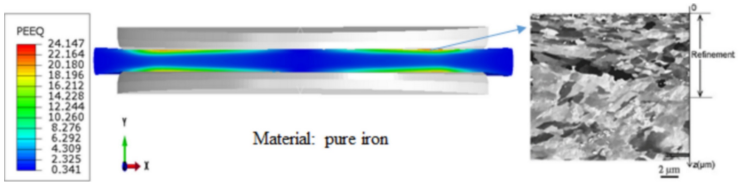
Keywords: *friction, severely deformed microstructure, finite element modelling.*

Wear process result from mechanical and physico-chemical phenomena. During contact's life, different deformation paths lead to the formation of severely deformed microstructures in a thickness ranging from few nanometer up to a dozen micrometer in the near-surface layer (tribological transformed surface, TTS [1]). Tribological stresses produce TTS in contacts, a confined medium. To overcome the problem of the contact instrumentation, both experimentally and numerically, the idea is to (re)produce TTS in a simplified configuration with a better control of the contact conditions. High Pressure Torsion (HPT) configuration permits not only the possibility to reproduce similar mechanical conditions (high pressure and shear) but also to follow the evolution of the deformed microstructure with shear strain. HPT tests have been performed with pure iron and a perlitic steel [2, 3].

In the aim to identify strain/stress history leading to high deformed microstructures, the HPT test configuration using different controlled strain paths will be coupled to a finite element model of the tests, developed with commercial software ABAQUS, using a mesh adaptivity method based on an Arbitrary-Lagrangien-Eulerian formulation. A low coupling, accounting for the strain gradients, will be introduced between the microstructural evolution model (continuous dynamic recristallization, CDRX model [4], developed at EMSE for high deformation in the framework of this project) and the FE model through an iterative process.

The enriched FE model will be used to investigate in a sufficiently accurate way the strain paths, in relation with the developed microstructures and their localization. Mechanical criteria of extremely deformed microstructures formation will be deduced from the coupling of the experimental approach to strain and stress fields estimated by the numerical modeling of tests.



(a) (b)

Figure 1: HPT test, (a) EF modeling and (b) microstructural consequences (from [3]), of a plastic shear flow in the sample sub-surface.

Acknowledgments

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MODELLING OF HYDROGEN DESORPTION TO INTERNAL CAVITIES DURING COOLING OF A LOW ALLOY STEEL

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Keywords: *Manufacturing process, Hydrogen diffusion and precipitation, Numerical Method, low alloy steel.*

Hot formed shells in low alloy steel used in pressurized vessels contain chemical overconcentration areas, further called “Segregated Band” (SB), linked to the solidification process [1,2]. Under some specific cooling conditions, internal hydrogen can diffuse through these SB towards MnS inclusions, concentrate at MnS/matrix interfaces [3] and result in cavities formation. These cavities might grow due to hydrogen recombination at the solid-gas interface [4].

The scope of this poster is to present an effective internal hydrogen diffusion model to describe pressurization of cavities during heat treatment. The estimated H_2 internal pressure may be used wherever possible to identify critical steps and consider cavity growth.

Experimental observations have been performed to define the relevant study cases and therefore to envisage a reduced model. A diffusion model which handles heterogeneities has also been presented. This model has been developed to calculate hydrogen redistribution among the three ‘phases’ of the low alloy steel: matrix, SB, internal cavities (assumed to form at the SB/MnS interface). The related computational model has also been described including three boundary conditions: zero flux at external boundaries due to the condition of symmetry and continuity of flux at both solid-solid interface, solid-gas interface.

Simulations underline that under standard cooling conditions for heavy forgings, the redistribution of hydrogen between matrix and SB is discontinuous. To be more accurate, several values of interfacial concentrations were calculated. The amplitude of the discontinuity increases with the temperature drop. Concerning the solid-gas interface, a pressurization of the cavity resulting from desorption driven by Sievert’s equilibrium was observed [5]. With decreasing temperature, the internal pressure increases, which is due to the solubility drop. Consequently the pressure is maximized at room temperature.

Thermodynamic equilibria are reached for narrow domains (typically millimetric scale), while large boxes (centimetric scale illustrating real components) lead to important kinetic effects.

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PERFORMANCE OF MATERIALS EXPOSED TO EXTREME CONDITIONS: HYDROGEN EMBRITTLEMENT, STRESS CORROSION CRACKING AND CORROSION FATIGUE – A COLLABORATIVE NETWORK, ECOLE DES MINES DE SAINT ETIENNE, IFPEN, INSA LYON, INSTITUT DE LA CORROSION

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Keywords: , *stress corrosion cracking, hydrogen embrittlement, corrosion fatigue.*

Most often, material selection relies on the mechanical performance of materials for a dedicated application, the environmental conditions being, if not ignored, neglected. However, in various industrial sectors, the materials are exposed to complex environments, which may induce premature failure of equipment due to the combination of applied stresses/strains, static or cyclic and the environment. The assessment of material behaviour in such conditions, requires not only capacities to reproduce aggressive conditions but also experiments dedicated to the understanding of corrosion processes involved in material damage.

Collaborations have been engaged since several years between the different laboratories cited above, to study material behaviour in complex environments, such as those encountered in the oil and gas industry, involving hazardous gas, high temperatures and pressures. The neighbourship of the laboratories is an advantage for the development of joined projects.
If the test equipment allowing to carry out studies in harsh conditions is not available in academic research laboratories, the EMSE and INSA teams can suggest collaborative work, involving experimental resources belonging to either IFPEN or Institut de la Corrosion (IC), to industrial companies.

Similarly, the capabilities of the academic laboratories regarding deep investigations, in situ and ex situ, monitoring, analyses of damage mechanisms, are complementary and often essential to support the more technological work performed at IFPEN and IC. These complementarities offered by the partnerships strengthen the relevance and the interest of the proposed projects.

Currently collaborative actions are running through PhD students, industrial projects involving one or several industrial companies, ANR projects, demonstrating the efficiency of the network. The partnerships were naturally developed based on the competencies of each laboratory, the attractiveness of the group being related to the ability to propose both fundamental and technological approaches.

MÉCANISMES DE FISSURATION AU COURS DU SOUDAGE DES MÉTAUX D'APPORT DE L'ALLIAGE 690 : EXAMEN DE LA FISSURATION DITE PAR "CHUTE DE DUCTILITÉ"

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Keywords: *Ductility dip cracking, inconel, filler metal.*

Inconel alloy 690 replaces the inconel alloy 600, for the fabrication of certain kind of the primary and secondary circuit components of the first generation of nuclear power plant. This alloy, and its welding filler metals (52 and 152), are sensitive to hot cracking during welding operation. This type of cracks is called "ductility dip cracking". The aim of the thesis is to identify the mechanism which leads the ductility dip cracking (grain boundaries sliding? secondary carbides precipitation? Impurities segregation?). To do this, 3 grades are tested with a hot crack sensibility test (Varestraint test). Those results are compared to hot tensile tests performed in Gleeble machine in association with advanced characterisation tests means : metallography, SEM, EBSD, WDS, Auger spectrospcopy.

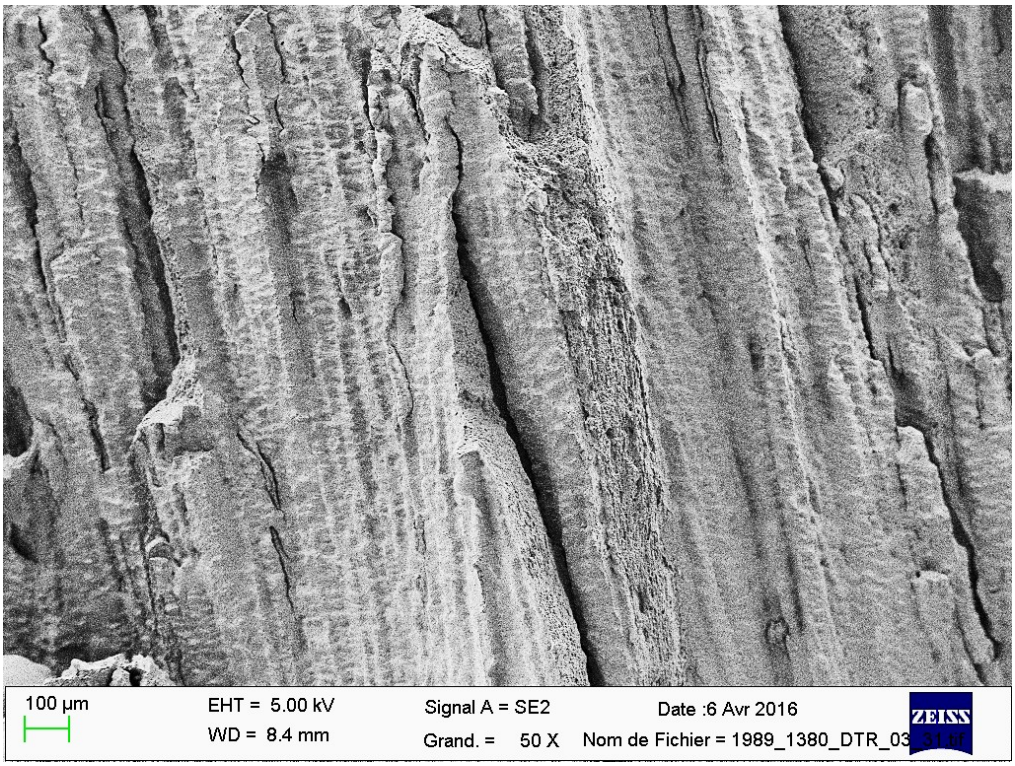

Figure 1: Gleeble test fracture surface on 52M alloy at 870°C on cooling

MÉCANISME DE TRANSPORT ET DE PIÉGEAGE DE L'HYDROGÈNE DANS UN ACIER MARAGING

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Keywords: *Hydrogen Embrittlement, Kelvin Probe Microscopy, Maraging Steel.*

Les aciers maraging, combinant structure martensitique et aptitude au durcissement structural, présentent une résistance mécanique exceptionnelle et trouvent de nombreuses applications dans le domaine de l'aéronautique et du militaire. Nous nous intéressons ici à l'acier maraging 350. Cet acier présente une limite d'élasticité pouvant atteindre 2500 MPa après un pallier de 4 heures à 510°C conduisant à la formation des précipités intermétalliques Ni3(Ti, Mo) et Fe2Mo. Dans certaines applications, l'acier maraging 350 est sujet à la fragilisation par l'hydrogène. Ce phénomène d'endommagement bien connu résulte de l'interaction de l'hydrogène avec les défauts microstructuraux, notamment ici avec les différents types d'interfaces présents dans la martensite (joints de grains, joints de paquets, joints de lattes…).

Dans un premier temps, nous avons caractérisé le comportement du matériau en traction, d'une part sous air, et d'autre part sous chargement cathodique à -1200mV/ECS, à différentes vitesses de déformation (10-4, 10-5, 10-7 s 1). Les chemins de fissuration ont été étudiés par observation fractographique au MEB et par cartographie EBSD.

On s'est intéressé dans un second temps à la possibilité de localiser l'hydrogène dans le matériau par la technique SKPFM (Scanning Kelvin Probe Force Microscopy) qui combine l'AFM avec le SKP. Nous présentons ici la méthodologie et quelques résultats préliminaires, obtenus sur un acier duplex.

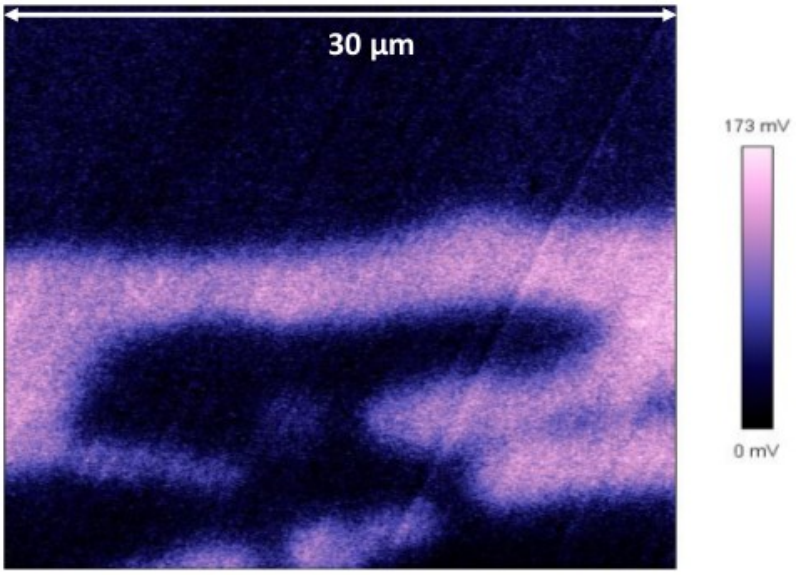

Figure 1: SKPFM map of hydrogen in a duplex steel. The accumulation of hydrogen in the austenite phase is evidenced.

INFLUENCE DE LA MICROSTRUCTURE DU VIRGOTM38 (ACIER INOXYDABLE SUPERMARTENSITIQUE BAS CARBONE 16CR-4NI) SUR SA RÉSISTANCE À LA CORROSION SOUS CONTRAINTE

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Contexte et objectif

Le VirgoTM38, un acier inoxydable supermartensitique bas carbone (16Cr-4Ni) est largement utilisé dans le milieu Oil&Gas. Ses propriétés lui permettent d'être utilisé, entre autres, pour la construction de roues de compresseurs centrifuges travaillant dans des milieux agressifs contenant du CO2 et de l'H2S. Sa microstructure se compose d'une matrice de martensite revenue α' et d'environ 20% d'austénite résiduelle. L'objectif de ce travail est de comprendre l'influence de l'austénite résiduelle sur la résistance à la CSC.

Résultats

Le traitement thermique dans une gamme de températures allant de 580°C à 660°C (ΔT=10°C) couplé avec des mesures DRX in situ et après refroidissement nous a permis d'optimiser la microstructure en maximisant le taux d'austénite au retour à température ambiante et en limitant la formation de martensite au refroidissement.

Des essais de compression nous ont permis de déterminer la stabilité de l'austénite en déformation. Le taux d'austénite diminue rapidement (-50% après 5% de déformation).

Ce phénomène pourrait être à l'origine d'une sensibilité accrue de cette nuance à la FPH due à la libération d'hydrogène en pointe de fissure. En effet une série de tests sous chargement cathodique montre une diminution de l'allongement à rupture de 25% à l'air à 5% dans la solution NACE3b (30g/L NaCl désaérée sous CO2 et acidifiée à pH=1.5 avec HCl). Les tests en présence de Na2S, qui permettent de simuler H2S conduisent à une forte fragilité, sans striction. Le caractère intergranulaire de ces ruptures indique un couplage possible entre ségrégations intergranulaires et effet fragilisant de l'hydrogène.

IN-SITU TEM NANO-COMPRESSION AND MECHANICAL ANALYSIS OF NANOPARTICLES

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Keywords: *In situ TEM, plastic deformation, dislocations, nanoparticles, MgO nanocubes.*

Nanometer-sized objects (micropillars, thin films and nanowires) are attracting large attention nowadays due to their breaking through mechanical properties such as high hardness, crack propagation resistance and high elastic limit in comparison to their bulk counterparts. Moreover, these nano-objects exhibit large plastic deformation under high load. The origin of this plastic deformation is still not very well defined. The mechanisms proposed are size-dependent and report this behavior to dislocation nucleation at surfaces and slipping on certain planes depending on the crystal orientation with respect to the solicitation direction.

In this study, we propose an innovative mechanical observation protocol of nanoparticles in the 100nm size range. It consists of *in-situ* TEM nano-compression tests of isolated nanoparticles (figure 1). Load–real displacements curves, obtained by Digital Image Correlation, are analyzed and these analyses are correlated with Molecular Dynamics simulations. A constitutive law with the mechanical parameters (Young modulus, Yield stress...) of the studied material at the nano-scale can be obtained [1]. *In-situ* TEM nano-compression tests were performed on ceramic MgO nanocubes. Magnesium oxide is a model material and its plasticity is very well known at bulk. The MgO nanocubes show large plastic deformation, more than 50% of plastic strain without any fracture [2].

This protocol reveals to be very useful to study the behaviour of ceramic nanoparticles and to determine their deformation mechanism. It can be used for metals and has already been used, a large number of studies being nowadays dedicated on plastic deformation of metals at the nano-scale while few being reported on ceramics.

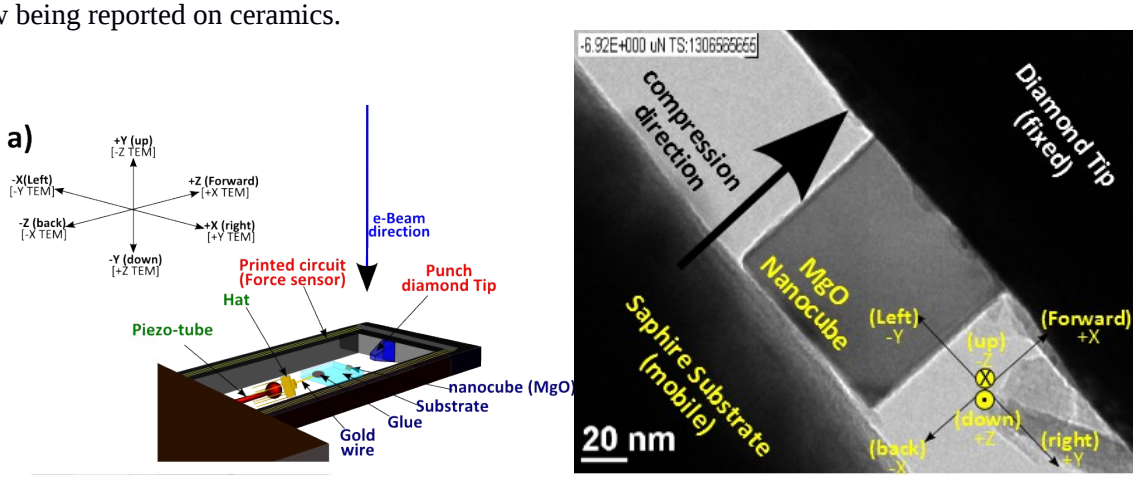

Figure 1: a) Schematic representation of the front piece of the sample holder used for *in-situ* testing, b) MgO nanocube just before *in-situ* nanocompression test. Arrows show possible movements of the sample

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LASER CLADDING: INFLUENCE OF PROCESS PARAMETERS ON THE MICROSTRUCTURE AND INTERNAL STRESSES DURING THE CLADDING OF GLASS MOLDS

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The manufacturing process of bottles consists in injecting the viscous glass (700-1200°C) within copper-aluminum or cast iron molds knowing that they also serve as heat exchangers during the production cycles. The glass problematic appears during molding since abrasion and corrosion occur on sensitive parts of the mold as well as thermal fatigue associated to the molding cycle.

The molds must therefore undergo a specific treatment (recharging using a high property suitable material), from the beginning of their manufacturing, on their sensitive parts (edges), in particular.

To do this, nickel powder depositions were carried out by laser cladding [1] especially at the neck of the bottle mold; that also constitutes a curvilinear area of the mold.

The microstructural behavior analyzed by EBSD (Electron BackScatter Diffraction), the chemical composition checked by EDS (Energy Dispersive X-ray Spectroscopy) and the mechanical properties, in terms of residual stresses generated by thermal gradients, measured by X-ray diffraction, have been investigated in accordance with the process parameters.

It was thus possible, with the help of all these techniques, to better understand the development of defects (cracks, lack of bonding ...) linked to the process parameters and in fact, to control and optimize the conditions of additive manufacturing and thus avoid these defects.

(a)

(b)

Figure 1: (a) Neck Ring mold and (b) a bottle

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STOCHASTIC VIBRATIONS INDUCED BY ROUGHNESS UNDER STEADY SLIDING CONDITIONS

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The dry friction between two rough surfaces is characterized by a succession of shocks between surface micro-asperities. It results in a dynamic vibro-impact regime that leads to the so-called roughness noise. The noise level depends mainly on the topography of surfaces, the sliding velocity and the vibrational eigenmodes [1]. Previous studies have led to better characterize these macroscopic relationships. However, their microscopic origin in terms of individual micro-impacts remains poorly understood [2].

To contribute filling this lack, we developed a model experiment in which we measure simultaneously the vertical acceleration and the electric contact voltage of a rough stainless steel slider. It moves at high velocity on a rough stainless steel track and is submitted to its own weight. We will show how the high frequency of the data allows us to provide a time resolved statistical description of the jumping dynamics of the slider as a function of the driving velocity, normal load and surface roughness. These measurements are compared with the results of a simplified numerical model of the system [3].

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EFFECT OF CRYSTALLOGRAPHIC ORIENTATION ON DEFECT GENERATION AND PERIODIC NANOSTRUCTURE FORMATION DURING FEMTOSECOND LASER IRRADIATION

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Keywords: *LIPSS, dislocations, EBSD, crystal orientation.*

The formation of laser-induced periodic surface structures (LIPSS) has been widely observed following solid target irradiation with ultrashort laser pulses. The interference between incident laser and a surface scattered wave leads to inhomogeneous energy deposition at the illuminated surface. The material response after absorption of the photon energy may also contribute to LIPSS formation through relaxation and self-organization. Surface planes with different crystal orientations possess different densities of surface atoms, surface energies and potential bonding sites, therefore these planes respond differently to the energy absorption, which may consequently play a role during LIPSS formation. This study shows the influence of crystal orientation on LIPSS formation. Electron Backscatter Diffraction (EBSD) characterization has been exploited to provide structural information within the laser spot on irradiated samples to determine the dependence of LIPSS formation upon the crystal orientation [1]. The results provide experimental evidence that laser-induced lattice damage and the formation of LIPSS are crystal orientation dependent [2]. Significant differences are observed at low-to-medium number of laser pulses at low fluence regime, outstandingly for (111)-oriented surface which favors dislocation storage rather than LIPSS formation.

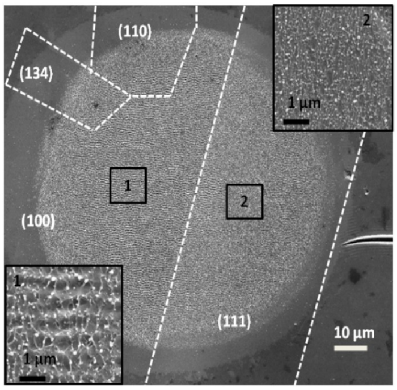

Figure 1: Crystal orientation dependence of LIPSS formation seen on poly-Ni.

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COATED FOAM DEEP CHARACTERIZATION USING X-RAY TOMOGRAPHY

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Keywords: *X-ray tomography, coating, foam.*

Heat and mass transfer improvement is an important part of research in catalytic process engineering. In this context, the use of materials such as metal or ceramic foams which develop a large surface area seems promising. Indeed, recent studies show that their use improves the mixing and heat exchange properties.

However, their use in catalytic reactor needs solid catalyst immobilization on/in their macroporous structure [1] [2] . The catalyst washcoating process is the key point to preserve the properties of the material. These methods, already described, depend on both substrate and foam nature.

This work presents the use of X-ray tomography associated to the common characterization methods (adhesion test, SEM, ...) in order to verify the homogeneity and thickness of the catalytic layer on these porous solids (Figure 1).

Scanning Electron Microscopy requires sample preparation (inclusion in resin, cutting, polishing, ...) and informations obtained are only adapted to small sample thickness. Although a spectral mapping technique could be used to scan the catalytic layer on the entire sample, this technique remains quite local.

X-ray tomography is a non-destructive method and does not require special preparation. Associated to image analysis, it allows a volume visualization of the catalyst layer, an important attractive feature for this type of solids. However, this technique can not be used in all cases, the X-ray adsorption properties of the materials involved (solid and deposit) must be of the same order of magnitude.

The spatial distribution of the coating has been compared for different washcoating procedures. The first results show, depending on the substrate used, the complementarity of the characterization methods. The information obtained has allowed to find the key steps in the catalyst deposition techniques to improve the homogeneity of the final catalyst layer.

Figure 1: Example of catalyst-coated foam (cylinder diameter: 20mm, length: 24mm).

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DENSIFICATION OF CERIUM GADOLINIUM OXIDE ELECTROLYTE BY LASER TREATMENT: APPLICATION TO SINGLE-CHAMBER SOLID OXIDE FUEL CELLS

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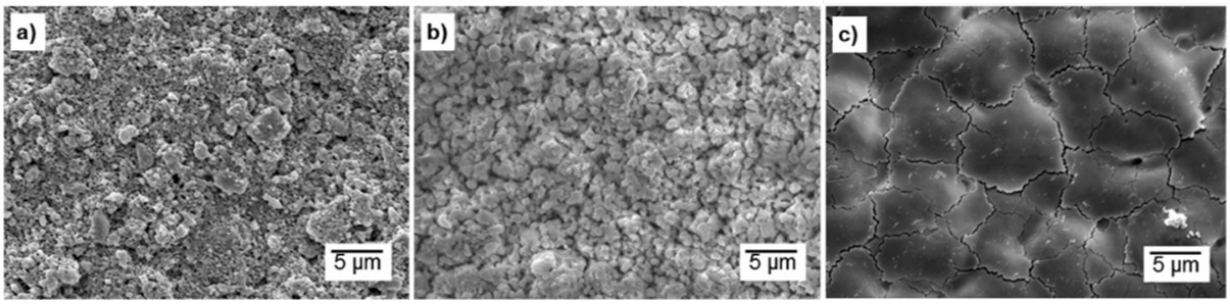
Keywords: *Single chamber SOFC, surface densification, laser processing.*

In single-chamber solid oxide fuel cells (SC-SOFC), anode and cathode are placed in a gas chamber where they are both exposed to a fuel/air mixture. Similarly to conventional dual-chamber SOFC, the anode and the cathode are separated by an electrolyte, but in the SC-SOFC configuration it does not play tightness role between compartments. For this reason, the electrolyte can be processed by screen printing, a technique particularly appropriated for preparing thick porous layers. However, it is necessary to have a diffusion barrier to prevent the transportation of hydrogen produced locally at the anode to the cathode through the electrolyte that reduces fuel cell performances.

This study aims to obtain directly a diffusion barrier through the surface densification of the electrolyte by a laser treatment. The material chosen for the electrolyte was cerium gadolinium oxide $Ce_{0.9}Gd_{0.1}O_{1.95}$ (CGO) which is deposited by screen printing on a composite NiO-CGO anode. UV laser and IR laser irradiations were used at different fluences and number of pulses to modify the density of the electrolyte coating. Microstructural characterizations confirmed the modifications on the surface of the electrolyte for appropriate experimental conditions showing either grain growth or densified but cracked surfaces (figure 1). Electrical conductivity of CGO was evaluated by electrochemical impedance spectroscopy. Subsequently, SC-SOFC performances were improved for the cells presenting grain growth at the electrolyte surface. In order to understand interaction between the laser and the material, simulations of the surface temperature during laser annealing of CGO will also be discussed.



INTERFACIAL PROPERTIES OF HYDRIDES IN A-ZR: A THEORETICAL STUDY

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Keywords: *hydrides, interfaces, ab-initio calculations, elastic properties, segregation.*

In order to better understand hydride formation in Zr alloys, heterophase interfacial energies are of prime importance but are theoretically difficult to obtain due to (i) the constrained state of the interface related to the epitaxial correspondence [1] and (ii) the large number of possible atomic interfacial configurations. To tackle these issues, basal heterophase interfaces between α zirconium and γ hydrides are investigated by means of ab initio atomic-scale simulations [2] coupled with continuous elasticity [3]. Using multilayered α|γ systems with various periods and phase proportions, our thorough atomic-scale study carried out for a selected interface variant allows to separate out the coherent interface energy from the elastic contribution [4] rising from α|γ epitaxy. The former part, with value 200 mJ.m^{-2}, is noticeably higher than previously found for other types of coherent hydrides (ζ) in Zr [5]. Furthermore, we show that the elastic energy can be consistently obtained either from atomic-scale simulations with long-period systems or from elasticity calculations. This agreement suggests that the α|γ coherent interface energy is easily accessible from a limited set of ab initio calculations, and this efficient [atomic-scale+elasticity] approach is employed for other interface variants. Finally, by considering interfacial changes of H contents, the usually overlooked possibility of competing elasticity and chemistry effects for interface stability is analyzed.

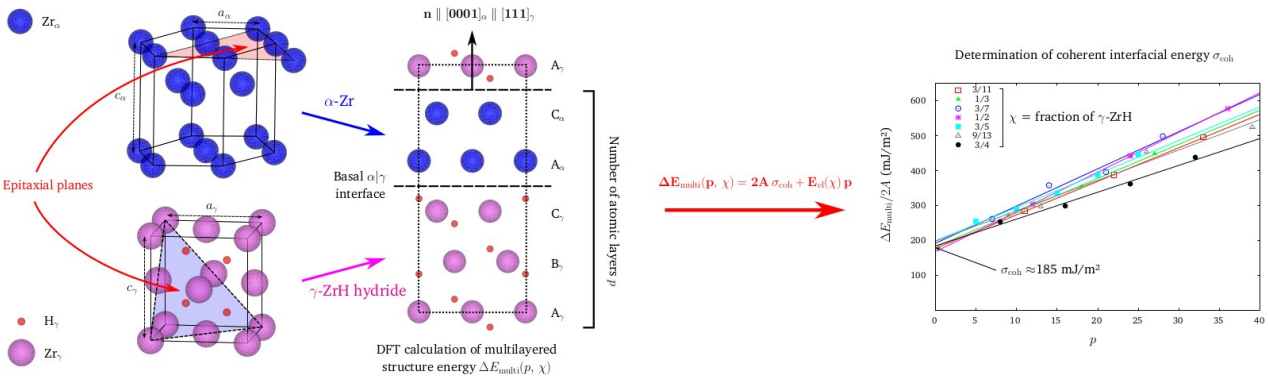

Figure 1: α-Zr and γ-ZrH phases used for the construction of α|γ multilayered structures (left) and energies of basal multilayers as a function of period for different hydride ratios (right).

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RELIABILITY OF T91 STEEL DEFORMED IN LEAD BISMUTH EUTECTIC

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Keywords: *liquid metal embrittlement, ADS, nuclear materials.*

The aim of this communication is to summarize the results obtained after more than 10 years of research performed at UMET on the mechanical behaviour of T91 steel (Fe9Cr1MoNbV) in presence of Lead-Bismuth Eutectic (LBE). This research has been performed in the frame of the national project GDR GEDEPEON and of European projects linked with accelerator driven systems or Gen IV nuclear reactors. One of these research objectives was to determine if LBE could embrittle or accelerate damage, and if so to determine the most critical parameters and the related mechanisms.
The following parameters were analysed:

- Heat treatment of the steel
- Loading mode (monotonic or cyclic)
- Mechanical Loading rate
- Test temperature
- Dissolved oxygen content in LBE

Taking air as the reference environment and a monotonic loading , T91 steel remains ductile in general in presence of LBE but a detrimental effect, a ductile□brittle transition, may occur all the more so as:

- T91 is tempered increasing its hardness
- The loading rate is low
- The oxygen content in LBE is low
- The test temperature approaches a critical temperature

However, the recommended heat treatment and an oxygen- saturated LBE bath do not guarantee immunity against embrittlement.
Under cyclic loading, the resistance to both fatigue crack initiation and propagation is decreased by the presence of LBE.

A common interpretation of embrittlement and accelerated damage by LBE of T91 steel is proposed.

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ATOMIC SCALE MODELISATION FOR THE Fe-Al-Mn-C ALLOY USING PAIRS MODELS AND MONTE CARLO CALCULATIONS

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Keywords: ab initio, Monte Carlo, cluster expansion, phase diagram

The Fe-Al-Mn-C system is widely studied for an automotive application due to its interesting mechanical properties and its relatively low density [1]. The aim of this study is to study the thermodynamic properties of Fe-Al-Mn-C using atomic-scale models. To start with, we focused on the Fe-Al binary system and we tested our capability to reproduce its phase diagram from *ab initio* calculations using the cluster expansion method. Four models have been adjusted on different input sets (IS) corresponding to atomic configurations that contain: pure iron, one aluminium atom on a substitutional site diluted in iron, pairs of substitutional aluminium atoms located at different neighbour shells (first, second, third,…) and eventually complementary structures (B2, B32 and $D0_3$). The FeAl-IS2 model is adjusted on an *ab initio* database containing pairs until the second neighbourhood and we added the complementary structures in the database to build the FeAl-IS2c model. The same notation has been used for models "IS3(c)".

Long range order parameters have been defined to analyse the equilibrium configurations generated by Monte Carlo runs in the semi-grand canonical ensemble. They correspond to the occupation of four sublattices of the bcc lattice.

The phase diagrams have been plotted for each model (Fig. 1), and they can be compared to an experimental one (Fig. 2). Models fitted on IS without complementary structures do not predict B2 for FeAl but the models including the ordered structures in give a very good qualitative agreement with the experiment. Some differences are observed for the second and third neighbour models, the FeAl-IS2c model predicts a dual phase domain between $D0_3$ and B2 whereas FeAl-IS3c does not. The methodology used in this work allow us to explore the ternary (Fe-Al-Mn) and quaternary (Fe-Al-Mn-C) system.

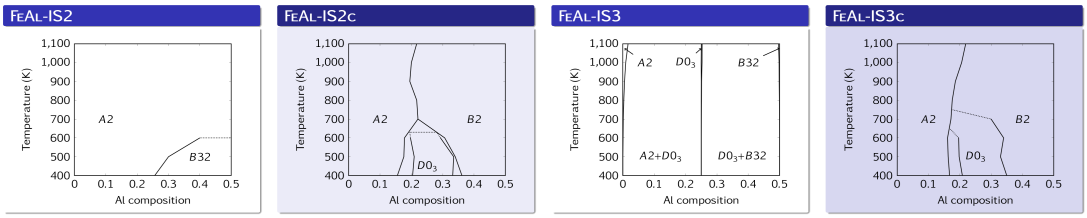

Figure 1: Phases diagrams obtain with our energetic models.

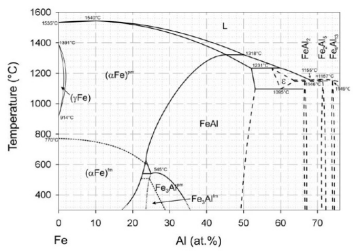

Figure 2: Experimental phase diagram [2]

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FROM CALPHAD TO EXPERIMENTAL: HOW FAR WILL THE SIGMA PHASE OF THE HEA SYSTEM COCRFEMNNI COULD GO?

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Keywords: *High entropy alloy, Calphad, XRD, SEM.*

The conventional alloy design strategy usually has one element as the principal constituent and other minor elements added for further optimization of properties and performances. In 2004, *Yeh et al.* [1] pointed out the critical role of the configurational entropy to form a multi-element solid solution and proposed the name of “high entropy alloys” (HEA). HEA were defined as alloys composed of five or more principal elements with concentrations between 5 and 35 at. %. By definition, this new concept material makes it possible to explore an almost infinite field of chemical compositions. But in the meantime, the thermodynamic stability of these systems is very poorly known and severely limits the choice of alloy compositions.

It is for this reason that we have decided to tackle this subject by studding thermodynamic stability of the Co-Cr-Fe-Mn-Ni system. To define the stability’s zone of the solid solution, we use the Calphad approach to predict the thermodynamically stable phases. The microstructure of the alloy could be calculated with the new HEA base depending on the composition and temperature. However, the quality of the results provided by Thermo -Calc need to be checked. This is undertaken with the elaboration and characterization (XRD, SEM) of three alloys.

The observation of multiphase alloys confirms predictions of Calphad method. In contrast, the phase characterization is not the same. The sigma phase is present and more stable than what suggests the prediction calculations. This challenges the description of the sigma phase by the base HEA, but in no way the description of the FCC phase. According to the calculations of 10 627 compositions, the FCC phase is present over a wide area.

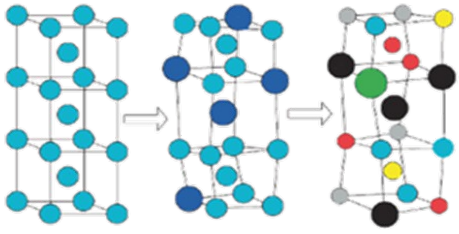

Figure 1: Schematic illustration of BCC crystal structure: (a) perfect lattice; (b) distorted lattice caused by additional one component; (c) serious distorted lattice caused by many atoms in multi-component solid solutions [2].

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EVALUATION OF AN ORIGINAL GRADE OF HEA ALLOY FROM AlCrFeMnNi FAMILY

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Keywords: *High Entropy Alloys, HEA, AlCrFeMnNi*

High entropy alloys (HEAs) represent a new class of metallic materials, based on an innovative approach to materials development. A general definition of HEAs says that these are solid solutions that contain five or more principal elements in equal or near equal atomic percentage, usually between 5 at% and 35 at% [1]. First work to analyse multi component alloys was done by Cantor et al. in 1981, where compositions containing from 5 to 20 elements in equiatomic ratios were analysed. As a result they obtained one alloy whose structure was single phased: the $Co_{20}Cr_{20}Fe_{20}Mn_{20}Ni_{20}$. Independent work was made by Yeh et al. whose publications were the first to explain the concept of HEAs by providing experimental results and related theory [2]. Many compositions have been analysed in particular based on Cantors equiatomic CoCrFeMnNi. Often substitutions of one of the elements have been performed to alternate the microstructure and mechanical properties [3,4].

An equiatomic AlCrFeMnNi alloy has been chosen as a promising composition based on Thermo-Calc calculations. Mechanical properties have been described by hardness measurements and compression tests. Microstructure has been observed at different levels, from the scale of light microscopy through SEM to TEM in combination with EDX, XRD and DSC. The alloy was analysed in as cast state since hot forging was impossible to perform. Microstructural observations proved complex hierarchical structure with different morphologies in the dendrites and interdendritic spaces, *Fig. 1a*. It was shown that the alloy is composed of two BCC phases, one rich in FeCr (dark) and one rich in NiAl with B2 ordering (bright). Presence of a third MnNi based phase has been detected by TEM. AlCrFeMnNi alloy shows promising mechanical properties with YS over 1000 MPa up to 500°C and predicted YS in room temperature (RT) of the order of 1300 MPa, *Fig. 1b*. Even though the material is brittle at RT no fractures were observed during compression above 350°C. The alloy shows sensibility to deformation rate which might be at the origin of brittleness during forging. No influence of annealing on hardness or behaviour in compression was observed.

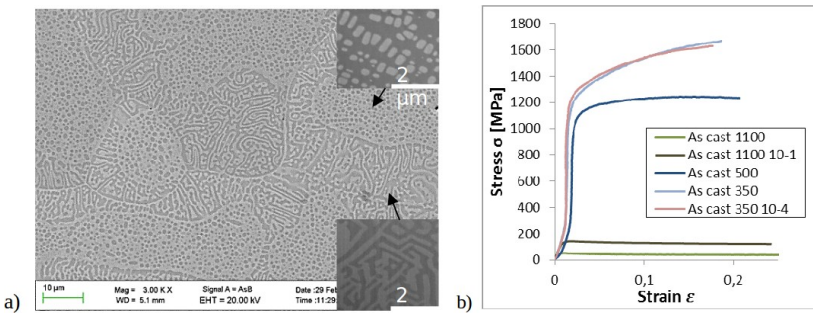

Figure 1: a) . SEM BSE image of the as cast sample. Two close ups of the structure of the dendrite (top) and the interdendritic space (bottom) in the perpendicular direction after FIB polishing; b) Engineering stress – strain curves of as cast state, tests at 350°C, 500°C and 1100°C with strain rate 10^{-2} s^{-1}, 10^{-1} s^{-1} or 10^{-4} s^{-1}

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MULTI-SCALE CHARACTERISATION OF A MICRO-ALLOYED "MEDIUM MANGANESE" STEEL

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Keywords: *micro-alloying, 3rd generation steel, characterisation.*

Among the 3rd generation advanced high strength steels (AHSS), medium Mn steels provide exceptional combinations of strength and ductility by an optimised distribution of phases (ferrite, martensite, retained austenite) and a careful control of the strain-induced martensitic transformation. Micro-alloying is expected to provide additional strength; however the interactions between micro-alloying elements precipitation and phase transformations leading to the multi-phase microstructures are complex and still ill understood. A micro-alloyed "medium manganese" steel grade has here been characterised at several steps throughout the elaboration process. Characterisation techniques provide complementary information at different scales.

Samples were selected at three stages of the metallurgical path that consists in: austenitisation followed by hot rolling and coiling steps, intermediate batch annealing, and cold rolling step then a final annealing.

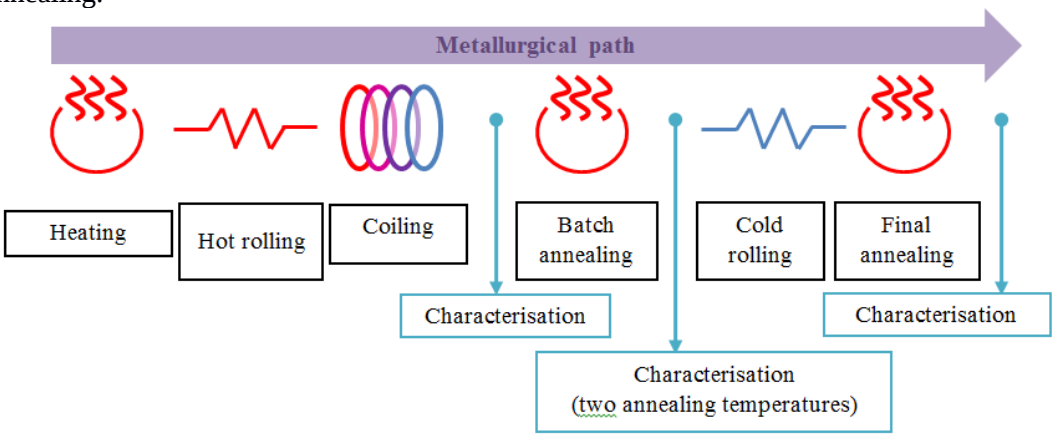

Figure 1: Metallurgical path and sample picking

Optical microscopy metallography revealed different phases, by means of a selective chemical attack.

A scanning electron microscope (SEM) in the back-scattered electrons (BSE) detector of, coupled to analysis by energy dispersive X-ray spectrometry (EDS) revealed that the distribution of the microalloying element can be heterogeneous. Electron back-scattered diffraction (EBSD) mapping allowed the assessment of grain size, shape and orientation.

Small angles scattering techniques (SANS, SAXS) indicated the presence of fine precipitation (~10nm), but also of bigger particles (>30nm).

The presence of these precipitates has been confirmed by transmission electron microscopy (TEM), notably through diffraction patterns. The structure and chemistry of these particles have then been identified thanks to the phase indexation software A-STAR, coupled to an EDS mapping.

USING THE REAXFF FORMALISM TO MODEL SEGREGATION-INDUCED EMBRITTLEMENT?

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Many non-metallic species segregating at the grain boundaries (GBs) in a polycrystalline metal can dramatically change its physical (mechanical, thermal, electrical, optical) properties and its lifetime. While some may improve its mechanical properties, sulfur (S) segregating in Nickel (Ni) GBs leads to its embrittlement, when the S-content reaches a critical concentration [1].

In [2] it has been shown that GBs with different orientations observed via the NanoSIMS display different enrichments, so that the critical concentration may depend on the atomic-scale structure of the GB. But the expected small thickness of the segregated layers prevents a straightforward observation and requires complementary atomic-scale models.

Apart from ab initio computations, that only deal with very specific GBs for tractability reasons, few methods have been developed to describe the Ni-S system, whereas many very efficient interatomic potentials can deal with pure metals and metallic alloys [3]. The recently developed ReaxFF approach has shown that an amorphous S-layer within the GB cores of a polycrystalline Ni favors a intergranular crack. Here we discuss its use to reach an atomic-scale description accounting for a chemistry-induced structural evolution of a given GB. For this purpose, we studied first the equilibrium properties of pure and defected bulk Ni (including cohesive energy, lattice parameter, elastic constants, vacancy formation energy), surfaces and S-adsorption and compared the results with both experimental and theoretical values.

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