

DGM

PROGRAMME AND ABSTRACTS

Intermetallics 2013



30 September–04 October 2013



Educational Center Kloster Banz
Germany

DGM

Deutsche Gesellschaft
für Materialkunde eV



www.dgm-intermetallics.de

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Organising Society

German Society for Materials Science (DGM)

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Dear Colleagues and Friends,

Facing an increasing demand for new materials with improved properties, intermetallic based materials offer a promising perspective. A possible combination of high strength, low density and good corrosion resistance qualifies them for structural applications, specifically at high temperatures and in severe environments. As some intermetallic phases show unique properties like shape memory and thermo electric effects or do have appealing magnetic properties they are also of interest for various functional applications. While intermetallics today are well accepted as shape memory alloys they are only now – after three decades of exhaustive research and development – used for structural applications, i.e. in flying gas turbines.

In spite of the current industrial breakthrough of intermetallic based materials, a regular meeting covering all aspects and types of intermetallic based materials is missing, in particular in Europe. It is therefore that DGM with Intermetallics 2013 initiated a new international conference. It encompasses fundamental aspects, characterisation, testing, modelling and application and is devoted to bringing together experts from all fields of science and industry involved in development and application of intermetallic based materials. Though it is a newly launched conference, it builds up on previous successful, specialised meetings such as γ -TiAl organised by GfE Metalle und Materialien GmbH, Nuremberg, and the Discussion Meeting on the Development of Innovative Iron Aluminium Alloys.

With about 150 attendees from 27 countries, contributing 79 oral and 58 poster presentations Intermetallics 2013 is one of the largest conferences ever devoted to intermetallic alloys and compounds. The choice of Kloster Banz should guarantee a stimulating conference. Situated in a scenic landscape above the valley of the river Main it is a remote place but also a well-known educational centre. With its good infrastructure and relaxing atmosphere it will foster the exchange of ideas and fruitful discussion as well as it will provide an opportunity to make new scientific connections. We believe that Intermetallics 2013 will further advance development and application of intermetallic based materials and wish all of us an exciting and productive meeting.

Your Intermetallics 2013 Organising Team

General information

Registration fees

Students	250 EUR
University/Institute	450 EUR
Industry	650 EUR
Accompanying person*	200 EUR

Social programme**

Get together • 30 September 2013	incl.
Excursion – hiking tour Bad Staffelstein • 2 October 2013	incl.
Excursion – bus tour Bamberg • 2 October 2013	10 EUR
Conference dinner • 3 October 2013	incl.
Farewell lunch • 4 October 2013	incl.

* Social programme is included

** Registration for the Social programme is required.

General terms and conditions

The general terms can be found on the conference website www.dgm-intermetallics.de.

Opening hours	Monday	Tuesday	Wednesday	Thursday	Friday
Industrial Exhibition	18 ⁰⁰ –20 ⁰⁰	08 ⁴⁵ –16 ³⁰	08 ⁴⁵ –14 ⁰⁰	08 ⁴⁵ –16 ³⁰	08 ⁴⁵ –14 ⁰⁰
Check-In	17 ⁰⁰ –20 ⁰⁰	08 ³⁰ –16 ³⁰	08 ³⁰ –14 ⁰⁰	08 ³⁰ –16 ³⁰	08 ³⁰ –14 ⁰⁰

Internet

Internet access is available at the Check-in. Wireless-Lan can be booked via Telecom Hotspot with your credit card. You receive details at the Check-In.

Certificate of attendance

Certificates of Attendance will be handed out to you on your last day of the congress at the Check-In.

Awards and poster prizes

The twenty best presentations will be published in a special edition of the journal “Intermetallics”.

Poster prizes

The three best posters will be awarded with 500 EUR each donated by BÖHLER Schmiedetechnik GmbH & Co KG.

Winners will be published in the special edition of the journal “Intermetallics”.



Evaluation

Please return the evaluation form to the Check-In on the last day of your stay. We are looking forward to your active participation and constructive criticism.

Catering

Wednesday dinner is not included in the conference fee. You can purchase voucher for the dinner at the Check-In (13 EUR).

The restaurant „Klosterschänke Kloster Banz“ is directly located on the premises of Kloster Banz and is open daily from 10⁰⁰–22⁰⁰ hrs.

For your information: the closest city with alternative restaurants is Bad Staffelstein, which is 5.5 km away (approximately 1h by foot, 7 minutes by car).

Smoking

Smoking is prohibited inside the entire conference centre.

Taxi • Taxi Dütsch

Phone +49 957 352 06

Price from Bad Staffelstein to Kloster Banz about 10 EUR*

Price from Lichtenfels to Kloster Banz about 13 EUR*

* Prices are subject to change.

Sponsors and media cooperations

We would like to thank the following partners for their great support for the Intermetallics 2013 conference.

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„Materials“

Submitting your presentation/technical information

Please prepare your presentation in 4:3 aspect ratio.

A presentation notebook with a PDF reader and MS Office PowerPoint 2007 will be provided. Notebook, presenter and laser pointer will be available at the speaker's podium in the lecture hall. You will be assisted by a technical supervisor. Please provide an adapter for VGA if necessary.

Please note: certain encodings for video and audio files could lead to technical problems.

Please submit your presentation at the lecture hall on the day before your presentation, but no later than two hours before the beginning of the presentation.

Speaking time

Please respect the timing allowed to the session and to each presentation. Chairs and moderators are encouraged to interrupt your presentation if you should overrun your time limit. Speaking time is assigned as follows:

Invited talk	30 minutes (incl. 5 minutes discussion)
Hot topic talk	25 minutes (incl. 5 minutes discussion)
Session lecture	20 minutes (incl. 5 minutes discussion)

Poster session

Posters will be rated on Tuesday, 1 October 2013, 19⁰⁰ hrs. Authors are requested to be present at their posters during the poster session.

Drinks and fingerfood will be served during the poster session.

Pinboards will be numbered. The pinboards are only to be used with the designated pins.

You will find your poster number in the programme book on page 23ff.

Posters are to be mounted until 16⁰⁰ hrs on Tuesday, 1 October 2013 and have to be removed by 10⁴⁰ hrs on Friday, 4 October 2013.

Welcome reception • Monday, 30 September 2013

Come together for drinks and snacks and enjoy this evening and allow yourself interesting conversations with colleagues, old friends, exhibitors and new acquaintances.

18 ³⁰ hrs	Welcome (in front of main hall)
20 ⁰⁰ hrs	Opening
20 ¹⁵ hrs	Opening talk Helmut Clemens

Excursion • Wednesday, 2 October 2013

You can choose between the hiking tour to Bad Staffelstein or a bus tour to Bamberg.

Hiking tour Bad Staffelstein

Start	14 ⁰⁰ hrs at Kloster Banz (car park)
Duration	4h
Fee	included

Please make sure to wear appropriate footwear. The tour ends at the beer garden of the Hotel Koster Banz.

Bus tour Bamberg

Start	14 ³⁰ hrs at Kloster Banz (car park)
Duration	4h
Fee	10 EUR (incl. a visit of the Cathedral)
Return	18 ⁰⁰ hrs at Promenadestraße/opposite to the Hotel Central

We invite you to discover the beautiful city of Bamberg which, with its significant historic monuments, its characteristic works of art and its many charming details, justifiably belongs to the UNESCO World Heritage.

The tour will start at 15⁰⁰ hrs with a guided tour and will allow you at the end some time for individual discoveries (17⁰⁰–18⁰⁰ hrs).

Conference dinner • Thursday, 3 October 2013

Take the chance to get in touch with friends and colleagues and have an enjoyable evening.

Start	19 ⁰⁰ hrs in the “Kaiser Saal”
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Monday, 30.09.2013	Tuesday, 1.10.2013		Wednesday, 2.10.2013		Thursday, 3.10.2013		Friday, 4.10.2013
Main hall	Main hall	Seminar room 9	Main hall	Seminar room 9	Main hall	Seminar room 9	Main hall
	09:00–10:20		09:00–10:20		09:00–10:20		09:00–10:20
	Invited Talk and Hot topics 1		Invited Talk and Hot topics 3		Invited Talk and Hot topics 4		Invited Talk and Hot topics 6
	S. 12		S. 16		S. 18		S. 22
	<i>Coffee break</i>		<i>Coffee break</i>		<i>Coffee break</i>		<i>Coffee break</i>
	11:00–12:20		11:00–12:20	11:00–12:20	11:00–12:20		10:40–12:25
	Invited Talk and Hot topics 2		Session TiAl 2	Session Miscellaneous	Invited Talk and Hot topics 5		Invited Talk and Hot topics 7
	S. 13		S. 16	S. 17	S. 18		S. 22
	<i>Lunch</i>		<i>Lunch</i>		<i>Lunch</i>		<i>Lunch</i>
	14:00–16:00	14:00–16:00	14:00–19:00		14:00–16:00	14:00–16:00	
	Session TiAl 1	Session Fe-Al 1	Excursion/Hike		Session Powder metallurgy 2	Session Functional intermetallics	
	S. 13	S. 14			S. 19	S. 19	
	<i>Coffee break</i>				<i>Coffee break</i>		
	16:30–18:30	16:30–18:30			16:30–18:30	16:30–18:30	
	Session Powder metallurgy 1	Session Fe-Al 2			Session TiAl 3	Session Superalloys, Ni-aluminides, silicides	
	S. 14	S. 15			S. 20	S. 21	
18:30			S. 10				
Opening Opening talk	19:00–21:00				19:00–23:00		
	Postersession with snacks and drinks				Conference Dinner, Awards and Award talk		
S. 12	S. 23				S. 10		

Scientific programme • Monday, 30 September 2013

- 18³⁰ Welcome (in front of main hall)
- 20⁰⁰–20¹⁵ Opening
Main hall Martin Heilmaier (Karlsruhe/DE)
- 20¹⁵–21⁰⁰ Opening talk
Main hall Advanced intermetallic titanium aluminides – development status and
L-IT 01 perspectives
Helmut Clemens (Leoben/AT)

Scientific programme • Tuesday, 1 October 2013

- 09⁰⁰–10²⁰ **Invited talk and Hot topics 1**
Main hall Chair: Helmut Mehrer (Obersteinenberg/DE)
- 09⁰⁰ **Invited talk**
L-IT 02 Characterization of intermetallics with the HIPPO & SMARTS neutron
beam-lines at LANSCE
Sven C. Vogel (Los Alamos/US)
- 09³⁰ **Hot topic**
L-IT 03 Structure solution of aluminides from precession electron diffraction
zonal data
Louisa Meshi (Beer Sheva/IL)
- 09⁵⁵ **Hot topic**
L-IT 04 Development of intermetallic materials using high-throughput thin film
experimentation and up-scaling
Alfred Ludwig (Bochum/DE)
- 10²⁰–11⁰⁰ Coffee break and Industrial exhibition

11 ⁰⁰ –12 ²⁰	Invited talk and Hot topics 2
Main hall	Chair: Helmut Clemens (Leoben/AT)
11 ⁰⁰	Invited talk
L-IT 05	Diffusion in binary intermetallics Helmut Mehrer (Obersteinenberg/DE)
11 ³⁰	Hot topic
L-IT 06	Progress in the industrialisation of titanium aluminides Volker Güther (Nuremberg/DE)
11 ⁵⁵	Hot topic
L-IT 07	Mechanical properties of an oxidation resistant TBC-coated titanium aluminide alloy Ariane Straubel (Dresden/DE)
12 ²⁰ –14 ⁰⁰	Lunch and Industrial exhibition
	Parallel sessions
14 ⁰⁰ –16 ⁰⁰	Session 1 • TiAl 1
Main hall	Chairs: Volker Güther (Nuremberg/DE), Ali El-Chaikh (Siegen/DE)
14 ⁰⁰	Microstructure and deformation mechanisms of a γ -TiAl intermetallic alloy – an <i>in situ</i> experimental study
L-TA 01	Jose M. Torralba (Madrid/ES)
14 ²⁰	Defect arrangements in a two-phase γ -TiAl alloy investigated by scanning and transmission electron microscopy
L-TA 02	Anja Weidner (Freiberg/DE)
14 ⁴⁰	Phase equilibria among β , $\alpha(\alpha_2)$ and γ -phases in Ti-Al-M ₁ -M ₂ quaternary systems at elevated temperature
L-TA 03	Hirotoyo Nakashima (Tokyo/JP)
15 ⁰⁰	Effect of molybdenum on the microstructure evolution and grain refinement in an intermetallic TiAl alloy system
L-TA 04	Svea Mayer (Leoben/AT)
15 ²⁰	Interplay between chemical disorder and Mo content on mechanical stability of cubic body centred TiAl
L-TA 05	David Holec (Leoben/AT)
15 ⁴⁰	Microstructural evolution in γ titanium aluminides during severe hot-working
L-TA 06	Ulrich Fröbel (Geesthacht/DE)

14⁰⁰–16⁰⁰ Session 2 • Fe-Al 1

Seminar room 9 Chairs: Ian Baker (Hanover/US), Claudio G. Schön (São Paulo/BR)

14⁰⁰
L-FA 01 Comparison of the microstructure and high temperature strength of ODS Fe-Al-Cr intermetallics with ODS binary Fe-Al and Fe-Cr
David Morris (Madrid/ES)

14²⁰
L-FA 02 Strengthening mechanism of Fe-Al-based alloys containing B2-type precipitates
Hiroyuki Yasuda (Osaka/JP)

14⁴⁰
L-FA 03 Strengthening of Fe₃Al alloys at high temperatures with grain boundary κ -Fe₃AlC precipitate films
Satoru Kobayashi (Tsukuba/JP)

15⁰⁰
L-FA 04 Deformation and dynamic recrystallization behaviour of two Fe-Al-Nb alloys reinforced with the Laves phase fibres
Srdjan Milenkovic (Madrid/ES)

15²⁰
L-FA 05 Twinning and shear localization during high strain rate deformation of Fe₃Al
Daniel Janda (Karlsruhe/DE)

15⁴⁰
L-FA 06 The EBSD study of recrystallization in rolled and hot deformed Fe- 40 at.% Al-0.1 at.% Zr-0.13 at.% B alloy
Jaromír Kopeček (Prague/CZ)

16⁰⁰–16³⁰ Coffee break and Industrial exhibition

Parallel sessions**16³⁰–18³⁰ Session 3 • Powder metallurgy 1**

Main hall Chairs: Alain Couret (Toulouse/FR), Sara Biamino (Torino/IT)

16³⁰
L-PM 01 Selective electron beam melting of γ -TiAl
Carolin Körner (Erlangen/DE)

16⁵⁰
L-PM 02 Microstructure of γ -TiAl (48-2-2) produced by selective electron beam melting
Vera Jüchter (Erlangen/DE)

17¹⁰
L-PM 03 *In situ* SEM analysis of the deformation and fracture mechanisms of a powder metallurgy γ -TiAl alloy
Jose M. Torralba (Madrid/ES)

- 17³⁰
L-PM 04 The effect of zirconium addition on sintering, microstructure and primary creep resistance of TNB-V5 (Ti-45Al-5Nb-0.2B-0.2C)
Juliano Soyama (Geesthacht/DE)
- 17⁵⁰
L-PM 05 Crystallographic modulation of B19 and α_2 phases in γ -TiAlX alloys (X = Nb, V, Mo, B, C)
Heike Gabrisch (Geesthacht/DE)
- 18¹⁰
L-PM 06 Aluminid-based PM intermetallic alloys
Alla Logacheva (Korolev/RU)
- 16³⁰–18³⁰
Seminar room 9 **Session 4 • Fe-Al 2**
Chairs: Hiroyuki Yasuda (Osaka/JP), Srdjan Milenkovic (Madrid/ES)
- 16³⁰
L-FA 07 Effect of thermodynamics on physical properties of ternary iron aluminides
Claudio G. Schön (São Paulo/BR)
- 16⁵⁰
L-FA 08 Atomic relaxation processes at very high temperature in a fine grain B2 Fe-Al intermetallic
Gabriel A. López (Bilbao/ES)
- 17¹⁰
L-FA 09 High-temperature measurements of thermal expansion, lattice parameter and elastic moduli in B2-type FeAl
Mi Zhao (Sendai/JP)
- 17³⁰
L-FA 10 Structure and stability of the γ brass-type high-temperature phases in Al-rich Fe-Al(-Mo) alloys
Frank Stein (Düsseldorf/DE)
- 17⁵⁰
L-FA 11 Bending test of micro pillars in solution – a new approach to studying the hydrogen embrittlement of iron-aluminum intermetallics
Mohammad Zamanzade (Saarbrücken/DE)
- 18¹⁰
L-FA 12 Oxidation behavior of binary Fe-Al alloys in air and saturated steam at 700°C
Lena Rempel (Aachen/DE)
- 19⁰⁰ Postersession at Foyer with snacks and drinks (page 23ff)

- 09⁰⁰–10²⁰ **Invited talk and Hot topics 3**
Main hall Chair: David Morris (Madrid/ES)
- 09⁰⁰ **Invited talk**
L-IT 08 Iron aluminides produced by Laser Engineered Net Shaping (LENS)
Jerzy Bystrzycki (Warsaw/PL)
- 09³⁰ **Hot topic**
L-IT 09 Strength and hardness of Fe₃Al iron aluminides as a function of heat
treatment
Georg Hasemann (Magdeburg/DE)
- 09⁵⁵ **Hot topic**
L-IT 10 Dry sliding wear of B2 aluminides and related two-phase alloys
Ian Baker (Hanover/US)
- 10²⁰–11⁰⁰ Coffee break and Industrial exhibition
- Parallel sessions**
11⁰⁰–12²⁰ **Session 5 • TiAl 2**
Main hall Chair: Masao Takeyama (Tokyo/JP)
- 11⁰⁰ The effect of initial microstructure on the forgeability in β contained
L-TA 07 TiAl alloy
Keiji Kubushiro (Yokohama/JP)
- 11²⁰ Cost efficient thermo-mechanical processing of a β -stabilized
L-TA 08 γ -TiAl alloy for aircraft engine application
Daniel Huber (Kapfenberg/AT)
- 11⁴⁰ Microstructure and tensile mechanical properties of boron-containing
P-TA 10 β -solidifying γ titanium aluminide alloys
Valery Imayev (Ufa/RU)
- 12⁰⁰ Phases evolving in Ti-Al-Cr-Zr oxidation protective coatings on γ -TiAl alloys
P-TA 04 during thermal cycling at 1000°C
Reinhold Braun (Cologne/DE)

- 11⁰⁰–12²⁰ **Session 6 • Miscellaneous**
Seminar room 9 Chair: Ridwan Sakidja (Kansas City/US)
- 11⁰⁰ Electrodeposition and structural characteristics of as-plated and heat
L-MI 01 treated intermetallic nickel-tin based coatings
Emmanuel Georgiou (Leuven/BE)
- 11²⁰ Electron microscopy study of magnetic field-induced variant selection
L-MI 02 during disorder-order transformation in CoPt alloy
Hiroshi Akamine (Osaka/JP)
- 11⁴⁰ Microstructure of tetragonal Ni₂B compound solidified from the
L-MI 03 undercooled melt
Matthias Kolbe (Cologne/DE)
- 12⁰⁰ Thermodynamic of new intermetallics for lithium-ion battery within the
P-MI 01 Cu-Li-Sn system
David Henriques (Jülich/DE)
- 12²⁰–14⁰⁰ Lunch and Industrial exhibition
- 14⁰⁰ Excursion/Hike (page 10)

- 09⁰⁰–10²⁰ **Invited talk and Hot topics 4**
Main hall Chair: Florian Pyczak (Geesthacht)
- 09⁰⁰ **Invited talk**
L-IT 11 Spark plasma sintering – A route for manufacturing TiAl blades?
Alain Couret (Toulouse/FR)
- 09³⁰ **Hot topic**
L-IT 12 TiAl alloys produced by electron beam melting
Sara Biamino (Torino/IT)
- 09⁵⁵ **Hot topic**
L-IT 13 Mo-Si-B alloys – Can they be made lighter and stronger than superalloys?
Martin Heilmaier (Karlsruhe/DE)
- 10²⁰–11⁰⁰ Coffee break and Industrial exhibition
- 11⁰⁰–12²⁰ **Invited talk and Hot topics 5**
Main hall Chair: Jose San Juan (Bilbao/ES)
- 11⁰⁰ **Invited talk**
L-IT 14 Slip in shape memory alloys – theory and experiments
Huseyin Sehitoglu (Urbana/US)
- 11³⁰ **Hot topic**
L-IT 15 Atomistic modelling of martensitic transformations in shape memory
alloys
Oliver Kastner (Bochum, Potsdam/DE)
- 11⁵⁵ **Hot topic**
L-IT 16 Using advanced ingot metallurgy to contribute to a better understanding
of NiTi and NiTiCu shape memory alloys
Jan Frenzel (Bochum/DE)
- 12²⁰–14⁰⁰ Lunch and Industrial exhibition

Parallel sessions14⁰⁰–16⁰⁰

Main hall

Session 7 • Powder metallurgy 2

Chairs: Jerzy Bystrzycki (Warsaw/PL), Carolin Körner (Erlangen/DE)

14⁰⁰

L-PM 07

Powder metallurgical production of NiTi parts with fully expressed shape memory properties

Martin Bram (Jülich/DE)

14²⁰

L-PM 08

Microstructure and phase constitution of Ti-SiC coatings fabricated by selective laser melting

Pavel Krakhmalev (Saint-Etienne/FR)

14⁴⁰

L-PM 09

Microstructure and mechanical properties of Co-base γ - γ' composites by mechanical alloying and field assisted hot pressing

Jose M. Torralba (Madrid/ES)

15⁰⁰

L-PM 10

High temperature creep behaviour of nanocrystalline aluminides (NiAl and FeAl) and their *in-situ* Al₂O₃ reinforced composites

Niraj Chawake (Chennai/IN)

15²⁰

L-PM 11

Additive manufacturing of a binary iron aluminide by laser metal deposition and selective laser melting

Gesa Rolink (Aachen/DE)

15⁴⁰

L-PM 12

In situ laser cladding of single-phase iron aluminide on plain steel and pure nickel

Benjamin Bax (Saarbrücken/DE)

14⁰⁰–16⁰⁰

Seminar room 9

Session 8 • Functional intermetallics

Chairs: Huseyin Sehitoglu (Illinois/US), Jan Frenzel (Bochum/DE)

14⁰⁰

L-FU 01

Influence of fabrication conditions on structure, magnetic property and magnetocaloric effect in Mn-based Heusler alloys

Dan Nguyen (Ha Noi/VN)

14²⁰

L-FU 02

Production, microstructure, and properties of single crystalline NiMnGa magnetic shape memory alloys

Emmanouel Pagounis (Stockach/DE)

14⁴⁰

L-FU 03

Anisotropic stress-strain-behaviour of NiTi

Peter M. Kadletz (Munich/DE)

- 15⁰⁰
L-FU 04 Shape recovery and long term behavior of the Ni₁₄Ti₅₁Pd₃₅ high temperature shape memory alloy
Anne Denquin (Chatillon/FR)
- 15²⁰
L-FU 05 Superelastic and shape memory behaviour in copper-based functional intermetallics
Jose San Juan (Bilbao/ES)
- 15⁴⁰
L-FU 06 HAADF-STEM studies of L1₀-type Fe-Pd alloy ordered under magnetic field
Sahar Farjami (Fukuoka/JP)
- 16⁰⁰–16³⁰ Coffee break and Industrial exhibition
- 16³⁰–18³⁰
Main hall **Parallel sessions**
Session 9 • TiAl 3
Chairs: Svea Mayer (Leoben/AT), Ulrich Fröbel (Geesthacht/DE))
- 16³⁰
L-TA 11 Microstructural deformation and fatigue damage in γ -TiAl produced by additive manufacturing
Mauro Filippini (Milano/IT)
- 16⁵⁰
L-TA 12 Low cycle fatigue-creep behaviour of cast TiAl-7Nb alloy at high temperature
Miroslav Smid (Brno/CZ)
- 17¹⁰
L-TA 13 Thermomechanical fatigue of a high-strength multiphase titanium aluminide alloy (TNB-V2)
Ali El-Chaikh (Siegen/DE)
- 17³⁰
L-TA 14 Mechanism study on rare earth microalloying to improve the thermoplastic behavior powder of metallurgical beta γ -TiAl alloy
Wei Zhang (Changsha/CN)
- 17⁵⁰
L-TA 15 Thermo-mechanical fatigue behaviour of HVOF coated third generation γ -TiAl alloy
Markus Hoffmann (Freiberg/DE)
- 18¹⁰
L-TA 16 Influence of the composition of the liquid activation agent for the halogen effect on technical γ -TiAl-alloys
Raluca Pflumm (Frankfurt a. M./DE)

- 16³⁰–18³⁰ **Session 10 • Superalloys, Ni-aluminides, silicides**
Seminar room 9 Chairs: Manja Krüger (Magdeburg/DE), Werner Skrotzki (Dresden/DE)
- 16³⁰
L-SN 01 Development of sintering process based on eutectoid decomposition
and redox reaction for thermoelectric β -FeSi₂ composite alloy fabrication
Yoshisato Kimura (Yokohama/JP)
- 16⁵⁰
L-SN 02 Heat treated microstructure and mechanical properties of high Cr
content Nb-Si based alloy
Lina Jia (Beijing/CN)
- 17¹⁰
L-SN 03 Determination of the ternary eutectic in the Nb-Si-Cr system and its
influence on the microstructure formation during directional
solidification
Florian Gang (Karlsruhe/DE)
- 17³⁰
L-SN 04 Mechanical properties and oxidation resistance of directionally
solidified NiAl-Mo composites at room and high temperatures
Lei Hu (Aachen/DE)
- 17⁵⁰
L-SN 05 Discrete dislocation dynamics modeling of loading orientation effect on
the low stress creep of single crystal Ni base superalloys
Seyed Masood Hafez Haghigat (Düsseldorf/DE)
- 18¹⁰
P-SN 08 High temperature elastic properties of the nickel-base superalloy CMSX-4
Kathrin Demtröder (Bochum/DE)
- 19⁰⁰
Kaiser Saal Awards, poster prizes and conference dinner (page 10)

- 09⁰⁰–10²⁰ **Invited talk and Hot topics 6**
Main hall Chair: Yoshisato Kimura (Yokohama/JP)
- 09⁰⁰ **Invited talk**
L-IT 17 Designs in refractory metal-based silicides and aluminides for
high-temperature coating applications
Ridwan Sakidja (Kansas City/US)
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L-IT 01

Advanced intermetallic titanium aluminides – development status and perspectivesH. Clemens¹, S. Mayer¹, W. Smarlsy²¹Montanuniversität Leoben, Department of Physical Metallurgy and Materials Testing, Leoben, Austria²MTU Aero Engines GmbH, Munich, Germany

After almost three decades of intensive fundamental research and development activities intermetallic titanium aluminides based on the γ -TiAl phase have found applications in automotive and aircraft engine industry. The advantages of this class of innovative high-temperature materials are their low density and their good strength and creep properties up to 750°C. A drawback, however, is their limited ductility at room temperature, which is expressed in a low plastic fracture strain. This behaviour can be attributed to both hindered dislocation movement and microstructural inhomogeneity. Advanced TiAl alloys are complex multi-phase alloys which can be processed by ingot or powder metallurgy as well as precision casting methods. Each process leads to specific microstructures which can be altered and optimized by thermo-mechanical processing and/or subsequent heat-treatments. The background of all these heat-treatments is at least twofold, i.e. concurrent increase of ductility at room temperature and creep strength at elevated temperature. In order to achieve this goal the knowledge of the occurring solidification processes and the following phase transformation sequences are essential. Therefore, thermodynamic calculations were conducted to predict the phase diagram of engineering TiAl alloys. After verification with experimental methods, e.g. short and long-term heat-treatments, differential scanning calorimetry and X-ray diffraction analysis, these phase diagrams provided the base for the development of smart heat-treatments. To account the influence of deformation and kinetic aspects sophisticated ex- and in-situ methods have been employed to investigate the evolution of the microstructure during thermo-mechanical processing and subsequent multiple heat-treatments. For example, in-situ high-energy X-ray diffraction was conducted to study dynamic recovery and recrystallization processes during hot-deformation tests. Due to the peculiar scattering behaviour of TiAl alloys neutron diffraction was also used for the investigation of order/disorder reactions. Summarizing all results a consistent picture regarding microstructure formation and its impact on mechanical properties in advanced TiAl alloys can be given. Finally, the development status and perspectives of these alloys as innovative automotive and aircraft engine materials are presented.

L-IT 02

Characterization of intermetallics with the HIPPO & SMARTS neutron beam-lines at LANSCES. C. Vogel¹, D. W. Brown¹, M. Okuniewski², A. Stebner³, F. Stein⁴¹Los Alamos National Laboratory, Los Alamos Neutron Science Center, Los Alamos, United States²Idaho National Laboratory, Idaho Falls, United States³California Institute of Technology, Pasadena, United States⁴Max-Planck-Institut für Eisenforschung, Düsseldorf, Germany

The HIPPO and SMARTS neutron time-of-flight beam-lines at LANSCE are general purpose powder diffraction and engineering diffraction instruments, respectively. In this presentation, we will give an overview of neutron diffraction applied to studies of intermetallics. We present highlights of studies of deformation mechanisms in NiTi shape memory alloys, for which crystal structure determination, quantitative phase analysis, strain and texture information characterized by neutron diffraction contributed to elucidating deformation mechanism and informing deformation models.

We show results from in situ high temperature powder diffraction studies to complete the phase diagrams of ternary Fe-Al-X system. Finally, we will present a re-assessment of the time-temperature-transformation diagram of the U-10Mo system using neutron diffraction. In this system, body-centered cubic γ U-10Mo decomposes into orthorhombic α -uranium while in parallel redistribution of Mo atom leads to the formation of meta-stable intermetallic phases. Phase fractions, lattice parameters and textures measured as a function of time using neutron diffraction allowed to gain new insight into this complex problem.

L-IT 03

Structure solution of aluminides from precession electron diffraction zonal dataL. Meshi^{1,2}, S. Samuha^{1,2}¹Ben Gurion University of the Negev, Materials Engineering, Beer Sheva, Israel²Ben Gurion University of the Negev, Ilse Katz Institute for nanosized science and technology, Beer Sheva, Israel

Due to the direct correlation among the physical properties and crystal structure of materials, study of the crystal structure of nanosized materials is crucial for fundamental understanding of their unique properties. Traditional single-crystal and powder X-ray diffraction methods cannot be applied for this purpose due to the unavailability of single crystals and/or small quantity and size of these crystals in the multiphase specimens. Thus electron crystallography is sometimes the only viable tool for their structure analysis.

In the previous century, electron diffraction (ED) was considered to be unsuitable for structure determination due to the problems of data quality arising from dynamical effects. At the last decades, researchers have shown that influence of dynamical effects can be substantially reduced if beam precession (PED) is used. This benefit has led to application of PED in the field of structure solution of various materials. Several approaches were proposed – such as, for example, usage of electron diffraction tomography for successful structure solution.

Our group focuses on application of PED zonal data for structure solution of intermetallics in general and aluminides in particular. This study is technologically essential since aluminides usually form as nanosized precipitates in metallic matrix and their structure cannot be solved by any other method. It must be noted that, structures of intermetallics were not solved previously using solely ED methods. Reason for that is in the nature of intermetallic compound's structures. Contrarily to other complex materials, the atomic distances and angles of intermetallics are not fixed and coordination polyhedra are usually unknown. Thus, structure solution of these compounds is harder to validate. Current work suggests an appropriate structure solution path for aluminides through analysis of influence of various parameters on structure solution of known Al-Mg intermetallide and subsequent successful full structure solution of several unknown new ternary aluminides.

L-IT 04

Development of intermetallic materials using high-throughput thin film experimentation and up-scalingA. Ludwig¹¹Ruhr-Universität Bochum, Institut für Werkstoffe, Bochum, Germany

New or optimized multifunctional and structural intermetallic materials are needed, e.g. for miniaturization of technological products with improved functionality even in extreme conditions or for efficient production/storage/conversion of energy carriers. For the discovery and optimization of new materials combinatorial and high-throughput experimentation methods are very effective. The materials to be investigated are deposited in the form of materials libraries by special magnetron sputter deposition methods (co-deposition, wedge-type multilayer deposition, shadow masking). These materials libraries are subsequently processed and characterized by high-throughput experimentation methods (automated EDX, XRD, temperature-dependent resistance and stress screening) in order to relate compositional information with structural and functional properties. The talk will cover examples of the combinatorial development of Ni- and Fe-based intermetallic materials for shape memory (Ni-Ti-X-Y, Fe-Pd-X) and other applications. The obtained results are visualized in the form of composition-function diagrams. Examples of up-scaling from thin film findings to bulk applications are discussed.

L-IT 05

Diffusion in binary intermetallicsH. Mehrer¹¹Institut für Materialphysik, Fachbereich Physik, Universität Münster, Obersteinenberg, Berghof 3, Germany

Diffusion in solids is fundamental in the art and science of materials. A deeper knowledge about diffusion requires information on the position of atoms and how they move in solids. In crystalline solids like intermetallics the atomistic mechanisms of diffusion are closely connected with defects. Vacancy-type defects often mediate self-diffusion and diffusion of substitutional solutes. Dislocations and grain-boundaries are other types of defects and can act as high-diffusivity paths, because the mobility of atoms along such defects is usually much higher than in the lattice.

Binary intermetallics are alloys or compounds of two metals or of a metal and a semimetal. Their structures are different from those of the constituents. Some intermetallics are interesting functional materials others have attracted attention as high-temperature structural materials. An overview of recent advances in the area of diffusion in binary intermetallics is presented with emphasis on the relationship between structure properties and thermal vacancies. Examples of binary intermetallics from systems such as Fe-Al, Fe-Si, Co-Nb, and Mo-Si are discussed.

Literature:

H. Mehrer, Diffusion in Solids – Fundamentals, Methods, Materials, and Diffusion-controlled Processes, Textbook, Springer 2007; Japanese edition 2012.

H. Mehrer, S. Divinski, Diffusion in metallic Elements and Intermetallics, Defect and Diffusion Forum 289- 292, 15 (2009).

L-IT 06

Progress in the industrialisation of titanium aluminidesV. Güther¹, M. Achtermann¹, J. Klose²¹GfE Metalle und Materialien GmbH, Advanced Materials, Nuremberg, Germany²GfE Fremat GmbH, Product Development, Freiburg, Germany

Intermetallic titanium aluminide alloys have been considered as promising candidates to replace heavy Ni based superalloy in the last stage(s) of the low pressure turbine in aircraft engines for more than 20 years. Whereas the alloy development resulted in alloys exhibiting well balanced materials properties, the lack of industrialized materials manufacturing and processing technologies was a major draw-back for any commercial applications except high class race sports applications. The increasing pressure on the improvement of aircraft engine efficiency forced the engine manufacturer to re-consider the use of titanium aluminides in the low pressure turbine. At least General Electric has successfully pioneered γ -TiAl LPT blades in the GENx engine series which are powering the B787 Dreamliner and the B747-8 for now more than 2 years. Other aircraft engine manufacturers are going to follow.

Unfortunately, the conventional Ti-alloy technologies to produce feedstocks for investment casting or forging operations via wrought processing of large ingots are basically not applicable to titanium aluminides. Furthermore, it has been demonstrated in the past that quasi-isothermal extrusion pressing of canned small sized γ -TiAl ingots is technically feasible but commercially limited to specialty niche market products. Thus, the development and permanent improvement of the corresponding industrial materials production technologies with regard to both quality/homogeneity and costs was a pre-condition on the way to bring titanium aluminides into real commercial applications. In order to meet the requirements of γ -TiAl component manufacturers, adjusted metallurgical ingot conversion technologies have been developed and successfully industrialized at AMG-GfE. In parallel, the level of materials homogeneity has been remarkably improved which offers the opportunity to produce components exhibiting reliable thermo-mechanical properties on a very high level. Effective recycling technologies of non-contaminated process materials improve the overall materials yield. AMG-GfE offers ingots and adjusted feedstock materials for subsequent investment casting or forging of any γ -TiAl alloy composition with a wide variety of sizes and dimensions.

L-IT 07

Mechanical properties of an oxidation resistant TBC-coated titanium aluminide alloyA. Straubel¹, C. Leyens¹, S. Friedle², M. Schütze², N. Laska³, R. Braun³¹Technische Universität Dresden, Institute of Materials Science, Chair of Materials Engineering, Dresden, Germany²DECHEMA-Forschungsinstitut, High Temperature Materials, Frankfurt am Main, Germany³German Aerospace Center, Institute of Materials Research, Cologne, Germany

Titanium aluminides strongly contribute to the emission reduction and high specific thrust targets envisioned by Flight-path 2050 – “Europe’s Vision for Aviation”. However, titanium aluminides have a resistance to oxidation only up to approximately 750°C. The typical turbine operating temperatures are from 700°C up to 1100°C, which places significant challenges on the materials used.

The present paper addresses a materials system comprised of a TNM-B1 titanium alloy that was fluorine-treated for oxidation protection and then overcoated with a thermal barrier coating (TBC) for heat insulation. Halogen treatment results in the formation of a protective alumina scale upon heat treatment at 900°C for 24h rather than a mixed alumina-titania oxide layer built on untreated titanium aluminides. Moreover, the alumina scale served as a bonding layer for the ~150 µm TBC that was deposited by electron-beam physical vapor deposition at two different deposition temperatures. In order to investigate the effect of fluorine treatment and TBC on the mechanical properties of TNM-B1, tensile tests at room temperature and elevated temperatures were performed. Careful microstructure characterization along with crack surface analysis enabled fundamental understanding of the mechanisms governing failure. Oxidation and TBC deposition led to changes in microstructure, grain refinement in the subsurface zone and reduction of β_0 -phase. Experimental results indicated that heat treatment during TBC deposition caused a loss of strength and strain.

L-IT 08

Iron aluminides produced by Laser Engineered Net Shaping (LENS)J. Bystrzycki¹¹Military University of Technology, Department of Advanced Materials and Technologies, Warsaw, Poland

This work is an overview of our efforts in the production and modification of microstructure and mechanical properties of iron aluminides by using the LENS technology. The work presents original results on the porosity, structure, microstructure, microtexture and mechanical properties of different aluminides fabricated by using LENS. The study was conducted in the Military University of Technology in Warsaw in the framework of a national R&D project funded by the National Centre for Research and Development. The LENS MR-7 and 850-R systems utilizing a high-power laser together with aluminide powders were used to build fully dense structures directly from 3-D CAD solid models. Different parts were produced layer by layer under the control of the software that monitors a variety of parameters to ensure geometric and mechanical integrity. The block and thin wall builds were deposited. The presentation will show examples of various microstructures with respect to grain size and shape and texture created in the materials by suitable selection of the process parameters. There are numerous possibilities for the design and control of microstructures in the investigated iron aluminides. The process parameters strongly influence the thermal behavior of the material during laser forming especially the cooling rate. The possibility of laser deposition of functionally graded materials with aluminides as well as the direct synthesis of ternary Fe-Al-Ti alloys will be shown. The strength and ductility as well as fatigue behavior of the LENS processed components were determined through tensile tests at room temperature. Based upon the obtained microstructures and microtextures, their effect on the deformation behavior of the Fe-Al components will be discussed as compared to the corresponding conventionally processed iron aluminides. Selective applications of iron aluminide components manufactured by LENS will be shown in the sector of fossil and renewable energy.

L-IT 09

Strength and hardness of Fe₃Al iron aluminides as a function of heat treatmentG. Hasemann¹, J. H. Schneibel², M. Krüger¹, M. Zamanzade³, E. P. George^{4,5}¹Institute of Materials and Joining Technology, Otto-von-Guericke University, Magdeburg, Germany²8809 Carriage House Way, Knoxville, TN, United States³Saarland University, Department of Materials Science, Saarbrücken, Germany⁴Oak Ridge National Laboratory, Materials Science and Technology Division, Oak Ridge, TN, United States⁵University of Tennessee, Materials Science and Engineering Department, Knoxville, TN, United States

The materials properties of Fe-Al alloys strongly depend on their heat treatment. There are considerable investigations in the literature on the mechanical behavior of quenched FeAl and its strong dependence on the temperature from which the quench occurs. While most studies were performed on FeAl alloys with Al concentrations ranging from 35 at.% to 50 at.%, little is known about the mechanical properties of quenched iron-rich Fe₃Al alloys near 25 at.% Al. A strong dependence of the yield strength of Fe₃Al on heat treatment was found which gives evidence that D0₃-structured iron aluminides show a similar behavior as FeAl alloys, albeit not as pronounced. The present work reports on the yield strength and hardness of Fe₃Al alloys as a function of the prior heat treatment. Various quenching experiments were performed using extruded alloys in the composition range between 23.5 at.% Al and 32 at.% Al. The relation between the yield strength and the quench temperature was investigated by using room temperature compression tests. Furthermore, experiments were performed to assess the kinetics of the yield strength reduction after annealing for various times at temperatures below the quench temperature. Possible reasons for the observed mechanical behavior are discussed and evaluated in light of information available in the literature. Additional nanoindentation experiments were carried out to determine whether the observed macroscopic effects are reproducible in small scales. This idea was substantiated by the increase of hardness after similar quenching experiments using nanoindentation method. It is concluded that room temperature strengthening in Fe₃Al is consistent with the behavior expected for quenched-in thermal vacancies.

L-IT 10

Dry sliding wear of B2 aluminides and related two-phase alloysI. Baker¹¹Dartmouth College, Thayer School of Engineering, Hanover, United States

Over the last 15 years, we have performed pin-on-disk wear tests under similar conditions on several alloys that are either single-phase B2 compounds or two-phase alloys that contain B2 compounds. For single-phase materials, we have examined the effects of stoichiometry on the wear behavior of both NiAl and FeAl and studied NiAl containing either 10 or 20 at.% Fe. For two-phase materials, we have examined Fe-Ni-Al alloys that consist of a B2 phase containing b.c.c. precipitates, while for Fe-Ni-Al-Mn alloys we have examined alloys that consist of ultrafine B2 + b.c.c. or B2 + L₂₁ (a further ordering of the B2 structure) phases, and lamellar-structured B2/f.c.c. alloys. This paper, presents an overview of the results of work on these alloys, which is still ongoing, and compares the wear behavior of these different materials. For most of these alloys, we also examined the effects of four different testing environments: air, oxygen, 4% hydrogen in nitrogen, and argon: in all cases argon was found to be a much more benign testing environment than the other environments. Most tests were performed against a partially-stabilized zirconia counterface at room temperature although a few tests were performed against a stainless steel counterface at both room temperature and 673 K. The mass losses and friction coefficients were determined from several wear tests for each alloy. The tips of the worn pins were examined using both scanning electron microscopy and transmission electron microscopy, the latter using specimens produced by focused ion beam milling, and the wear tracks on the zirconia disks were measured using profilometry. In nearly all cases, zirconia particles were found to be embedded in a tribolayer on the worn tips of the pins. Two-body and three-body abrasive wear, as well as, in most cases, plastic deformation and delamination, were the main wear mechanisms. The abrasive particles largely consisted of the counterface material.

L-IT 11

Spark plasma sintering – A route for manufacturing TiAl blades?A. Couret¹, T. Voisin¹, L. Durand¹, M. Thomas², J.- P. Monchoux¹¹CEMES-CNRS, Toulouse, France²ONERA/DMSM, Châtillon, France

Spark Plasma Sintering is a powder metallurgy technique, for which the heating of the sample occurs by the application of a pulsed direct electric current. The aim of the present work is to investigate this technique as a route for the manufacture of TiAl turbine blades of aircraft engines.

The first goal is to develop alloys with improved properties, satisfying the industrial requirements. The microstructures are mastered through the optimization of the chemical composition and the control of the parameters of the SPS cycle. Alloys with refined microstructures are found to exhibit the most interesting properties: i) a good reproducibility of these properties due to the use of the powder metallurgy route, ii) a plastic elongation at room temperature higher than 1.5% due to the presence of an amount of γ phase and to the limited size of lamellar grains and iii) an exceptional creep resistance at 700°C due to the predominance of the lamellar structure and the strength of the γ phase.

In the second part, the method followed to sinter directly near net shape blades by SPS will be described. The basic idea is to use the graphite assembly containing the powder to give the final shape to the piece. Finite Element Modelling is used as a predictive tool to determine the processing parameters and to control the temperature. The density and the microstructure of the products will be examined. The aim is to achieve materials with the optimized properties without applying subsequent thermal treatments.

As a conclusion, this work demonstrates that SPS may allow the production of blades, made of performing TiAl alloys.

L-IT 12

TiAl alloys produced by electron beam meltingS. Biamino¹, F. Pelissero², S. Sabbadini², P. Fino¹, M. Pavese¹, C. Badini¹¹Politecnico di Torino, Department of Applied Science and Technology, Torino, Italy²Avio SpA, Torino, Italy

Electron Beam Melting (EBM) has become an outstanding process for near net-shape metal parts manufacturing. The process allows great freedom of design and low impurities pick-up, the parts being built in vacuum layer by layer from a metal powder. It already proved its suitability to produce Ti-48Al-2Cr-2Nb specimens for aerospace application [1]. The present work focuses on the evaluation of powder recyclability. A certain amount of powder remains un-sintered among the built components at the end of the process. This powder is recovered and inserted into the machine to be used in the following jobs. The powder recyclability is assessed in terms of number of times that the powder can be recycled without changing its main characteristics of powder grain size distribution and presence of contaminants, in particular oxygen and nitrogen. This will affect very much part cost and environmental aspects.

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L-IT 13

Mo-Si-B alloys – Can they be made lighter and stronger than superalloys?M. Heilmaier¹, D. Schliephake¹¹KIT Karlsruhe, Institute for Applied Materials, Karlsruhe, Germany

Recently, it was found that macro-alloying of ternary Mo-Si-B with Ti stabilizes the tetragonal $D8_m$ $(Mo,Ti)_5Si_3$ phase on the expense of the cubic $A15$ Mo_5Si_3 phase. This is beneficial not only to achieve lower densities, but also to improve oxidation and creep resistance. The former aspect was already demonstrated in recent investigations on single-phase $Mo(Ti)_5Si_3$. At all relevant temperatures in the range 750-1300°C, this intermetallic compound was found to be superior to its single-phase binary counterparts, Mo_5Si_3 and Ti_5Si_3 , respectively. This can be rationalized by the formation of a protective SiO_2 - TiO_2 duplex scale upon oxidation, which dramatically reduces outward diffusion of Ti. The latter behaviour is focused on with two alloy compositions, Mo-9Si-8B-29Ti and Mo-12,5Si-8,5B-27,5Ti (all compositions in at.%), respectively, which are predicted by thermodynamic calculations to lie in the desired 3-phase field region of $Mo(Ti)_{ss}$, $Mo(Ti)_5Si_3$ and Mo_5SiB_2 (the T2 phase). Alloys were prepared by both, cast and powder metallurgy. Subsequent 1600°C high temperature annealing does result in the thermodynamically expected above phases. Rather, $Mo(Ti)_3Si$ and $Ti(Mo)_3Si_3$ prevail. Thorough annealing treatments and detailed thermodynamic calculations readily rationalize above findings and a methodology is presented to establish above desired phases.

Creep behaviour was investigated in compression under constant strain rate and stress conditions, respectively, at temperatures between 1100 and 1300°C to identify the operating creep mechanisms. While yielding a density reduction of more than 10% as compared to pure nickel, our Mo-Si-B-Ti alloys show superior creep resistance in terms of an at least one order of magnitude lower minimum creep rate was observed at all conditions compared to a state-of-the-art nickel-base single crystalline alloy CMSX-4.

L-IT 14

Slip in shape memory alloys – theory and experimentsH. Sehitoglu¹, J. Wang¹, H. J. Maier²¹University of Illinois, Mechanical Science Eng., Urbana, United States²Leibniz Universität Hannover, Institut für Werkstoffkunde, Garbsen, Germany

We provide an extended Peierls-Nabarro (P-N) formulation of generalized stacking fault energy (GSFE) to establish flow stress in a Ni_2FeGa shape memory alloy. The resultant martensite structure in Ni_2FeGa is $L1_0$ tetragonal. The atomistic simulations allowed determination of the GSFE landscapes for the (111) slip plane and,

$\frac{1}{2}[\bar{1}01]$ $\frac{1}{2}[\bar{1}10]$ $\frac{1}{6}[\bar{2}11]$ and $\frac{1}{6}[11\bar{2}]$ slip vectors.

The energy barriers in the (111) plane were associated with superlattice intrinsic stacking faults, complex stacking faults and anti-phase boundaries. The smallest energy barrier was determined as 168 mJ/m² corresponding

to a Peierls stress of 1.1 GPa for the $\frac{1}{6}[11\bar{2}](111)$ slip system. Experiments on single crystals of Ni_2FeGa were conducted under tension where the specimen underwent austenite to martensite transformation followed by elasto-plastic martensite deformation. The evidence of dislocation slip in Ni_2FeGa martensite was also identified with transformation electron microscopy observations. We also investigated dislocation slip in several important shape memory alloys and predicted Peierls stresses in Ni_2FeGa , $NiTi$, Co_2NiGa , Co_2NiAl , $CuZn$ and Ni_2TiHf austenite in excellent agreement with experiments.

L-IT 15

Atomistic modelling of martensitic transformations in shape memory alloysO. Kastner^{1,2}

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Shape memory alloys exhibit characteristic thermo-mechanical coupling related to revertible phase transformations between austenite and martensite. Load, strain and temperature are coupled through the thermodynamic phase equilibrium criterion. The process diagrams exhibit characteristic hystereses and additionally are affected by functional fatigue properties during cyclic loading. In the thermodynamic theory, hysteresis and functional fatigue both are explained by the energetic impact of microstructural elements like phase interfaces, lattice defects and dislocations which are inevitably produced during martensitic transformations and thus dynamically interfere the phase equilibrium.

In this contribution we present a qualitative molecular dynamics simulation study of this situation. Our model material exhibits a non-diffusive martensitic transformation comprising square to hexagonal lattice transformations in 2D and bcc to fcc transformations in 3D, respectively. We discuss the nucleation process of martensite and show the propagation of motile transformation fronts, martensitic plate growth, the twinning process and the formation/accommodation of martensitic domain structures. During transformation processes, lattice defects are generated which affect subsequent transformations. Such defects may provide a memory of previous structures, and thereby may be the basis of a revertible shape memory effect. Employing thermodynamic arguments we explain how the energetic implications of such defect structures may contribute to pronounced hysteresis and functional fatigue.

L-IT 16

Using advanced ingot metallurgy to contribute to a better understanding of NiTi and NiTiCu shape memory alloysJ. Frenzel¹, G. Eggeler¹

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In the present work, we demonstrate how advanced ingot metallurgy can contribute to a better understanding of functional and structural properties of NiTi shape memory alloys (SMAs). In the first part of our work, we investigate the effect of Ni-concentration on the martensitic transformation. NiTi ingots with precisely controlled compositions were produced through an optimized arc melting procedure. It was found that increasing Ni-concentrations are associated with decreasing transformation temperatures. As a striking new result, the Ni-content also affects the hysteresis width of NiTi SMAs. This observation can be rationalized on the Ball and James theory; a higher Ni-concentration is associated with a better crystallographic compatibility between the high and the low temperature phase, and thus with a lower nucleation barrier for the martensitic transformation. In the second part of the present work, we investigate how the functional stability of NiTi and NiTiCu spring actuators can be optimized through both microstructural and chemical modifications. Grain refinement allows for a minimization of irreversible (plastic) deformation processes. However, it is also associated with a decrease in phase transformation temperatures since the high density of internal interfaces impedes the nucleation of martensite during cooling. As an alternative, alloying Cu to binary NiTi is a very effective way to improve functional stability. With increasing Cu-concentration (replacing Ni), the crystallographic compatibility between the high and the low temperature phase improves. Thus, less defects form during the martensitic transformation.

L-IT 17

Designs in refractory metal-based silicides and aluminides for high-temperature coating applications
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Refractory metal-based aluminides and silicides offer a range of attractive materials properties suitable for high-temperature structural applications including their mechanical strength and oxidation resistance. One of the potential applications of these compounds has been their use as protective coatings, primarily to augment the materials performance of the refractory-metal based alloys as the base material at high temperatures. In this talk, the advantages and challenges in implementing selected number of leading refractory-based silicides and aluminides as the main phase constituent of the coating structures will be discussed. While the general outlook of their applications is very promising, the existence of different, and sometimes conflicting, sets of material criteria demanded for such coating applications necessitates the immediate needs for a more comprehensive high-temperature materials database of these compounds leading to a more judicious materials selection process. Such database can be acquired expeditiously through a synergy of experimental means and theoretical calculations. We gratefully acknowledge the support of National Energy Technology Laboratory (NETL) – U.S. DOE.

L-IT 18

Microstructure size scale effects on the deformation and fracture behavior of Mo-Mo₃Si-Mo₅SiB₂ alloys
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Mo-Si-B alloys containing the phases Mo solid solution, Mo₃Si and Mo₅SiB₂ are of specific interest for structural applications at high temperatures due to their outstanding properties. Since the Mo solid solution phase provides a good fracture toughness and ductility, a homogeneous and continuous formation of this phase is required. The two intermetallic phases should be formed as homogeneously distributed islands for improved creep and oxidation resistance. In this study we show how mechanical alloying (MA) influences the distribution of the three phases and how this step affects the microstructure size scale of samples processed by powder metallurgy (PM). Additionally, materials having the same nominal compositions were produced by a zone melting (ZM) technique. While these two processes lead to similar volume fractions of the three phases the phase distribution and grain sizes were found to vary significantly. MA for 20 hours lead to fine grained microstructures with a continuous Mo matrix, shorter milling caused inhomogeneous distribution of the Mo phase and a coarse grain structure. Microstructural characterization of ZM alloys showed larger Mo particles embedded in a Mo₅SiB₂-Mo₃Si matrix.

Fracture toughness tests at room temperature indicated that the fine grained PM alloys with a continuous Mo matrix possess the potential of crack absorption in the tough Mo matrix. There again, the PM alloys with an intermetallic matrix showed similar fracture toughness values as the ZM materials. The brittle-to-ductile-transition-temperature (BDTT) of different PM alloys decreased by around 150 K due to switching over from an intermetallic matrix into a Mo solid solution matrix. Surprisingly, the BDTT of the ZM materials of about 900°C was found to be in the same range as for the fine grained PM alloys with Mo matrix. Furthermore, the creep resistance at temperatures between 1093°C and 1400°C was shown to be improved with increasing grain size.

L-IT 19

A thought on sigma phase as potential strengthener in austenitic heat resistant steelsM. Takeyama¹¹Tokyo Institute of Technology, Metallurgy and Ceramics Science, Tokyo, Japan

The σ phase with crystal structure of tP30, one of the topologically close-packed (TCP) phases, is often observed in conventional austenitic heat resistant steels after long-term aging. This phase is considered as detrimental phase to deteriorate the creep rupture strength. However, the σ phase is an equilibrium phase formed through precipitation of transition metal carbides, so that it could be a potential strengthener for long-term creep strength if the precipitation kinetics and morphology are appropriately controlled, just like Fe₂Nb Laves phase.

The nose temperature of σ phase in TTP diagram is important to control the kinetics and morphology. In this presentation, the formation process of σ phase in fcc γ phase in the carbon-free Fe-Cr-Ni alloys at 1073 K is presented first. It is found that the σ phase preferentially forms at grain boundaries through transformation from the prior formation of non-equilibrium bcc phase with higher Cr content (α -Cr) precipitated at the grain boundaries. The kinetics of the σ phase formation becomes further sluggish when formation of the α -Cr phase is suppressed. The precipitation kinetics of the σ phase in the γ matrix is much more sluggish than that at the grain boundaries. Interestingly, the morphology of the grain boundary σ phase can be refined by the addition of boron, similar to the Laves phase. The stability of σ phase by alloying will also be touched, and the possibility of the σ phase as a strengthener for the austenitic steels will be discussed. This research is partly supported by the Grants-in-Aid for Science Research Program (14205102), Japan Society for the Promotion of Science (JSPS).

L-IT 20

Local ordering of the atoms in intermetallic compoundsF. Haarmann¹¹RWTH Aachen, Institute of Inorganic Chemistry, Aachen, Germany

Intermetallic compounds are a fascinating class of materials with respect to structural chemistry and technological applications. The interest of basic research focuses on the chemical bonding and the formation of disorder. Thus, NMR spectroscopy seems to be an ideal tool for the investigation of intermetallic compounds since it is a local probe related to the chemical bonding. Combined application of NMR spectroscopy, quantum mechanical calculations of NMR coupling parameters and X-ray diffraction was performed to study the peculiarities of the bonding situation of Ga atoms in alkaline earth metal gallides [1,2]. The compounds were chosen as a model system to determine reliable NMR spectroscopic parameter for the investigation of intermetallic compounds possessing.

Very good agreement of calculated and experimental coupling parameters was achieved for the electric field gradient (EFG). This can be split into its contributions according to the calculation scheme used by the program WIEN2k [3]. An analysis of the EFG contributions reveals the local character of the EFG.

Due to the reliability of the EFG for the investigation of intermetallic compounds it was chosen to study the influence of disorder on the local bonding situation. The anisotropic conductivity of the samples was used to align the crystallites in the magnetic field resulting in an increased experimental resolution. Line shape analysis of orientation dependent NMR experiments of the aligned powders reveals a reduced symmetry and a change of the coupling parameter. Super lattice structures were derived to model the varying local arrangements of the atoms. DFT calculations based on these super lattice models were done to calculate the quadrupole coupling parameters. Reasonable agreement of calculated values and the experimental findings was obtained. Thus, the EFG can also be used to study structure and chemical bonding of disordered compounds possessing metallic conductivity.

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L-IT 21

Texture and microstructure evolution in NiAl deformed by high pressure torsionW. Skrotzki¹, C. Tränkle¹, R. Chulist¹, B. Beausir², T. Lippmann³, J. Horky⁴, M. Zehetbauer⁴¹Technische Universität Dresden, Institut für Strukturphysik, Dresden, Germany²University of Lorraine, LEM3, Metz, France³Helmholtz-Zentrum Geesthacht, Geesthacht, Germany⁴University of Vienna, Faculty of Physics, Vienna, Austria

NiAl is an intermetallic compound with B2 structure which shows a brittle-to-ductile transition temperature at about 300°C. Characteristics like a low density, a high melting point and very good oxidation resistance are advantageous for high temperature applications, but processing at low temperatures is difficult because of brittleness [1]. As shown in [2], fracture stress and fraction strain are increased under high pressure. On account of this, deformation at low temperatures is only possible at high pressures, as for instance used in high pressure torsion (HPT).

For this work, small discs of polycrystalline NiAl were deformed by HPT at temperatures from room temperature up to 500°C and pressures ranging from 2 to 8 GPa. In this way, very high local shear strains at the edge of the samples of about 70 could be achieved. Local textures were measured by high-energy synchrotron radiation at different positions from the center to the edge of the sample. Due to increasing shear strain along this line a texture gradient is observed. The texture also changes with processing temperature and pressure. Type and intensity of texture will be discussed with respect to slip system activity and recrystallization. Besides, the microstructure was analyzed by electron back scatter diffraction in a scanning electron microscope. Grain size, grain shape and local misorientation are found to be associated with the texture evolution.

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L-IT 22

Refractory high-entropy alloys as high-temperature structural materialS. Maiti¹, W. Steurer¹¹ETH Zurich, Department of Materials, Zurich, Switzerland

High-entropy alloys (HEA) are made of four to ten elements with equimolar or near-equimolar ratios. Multi-component alloys with one principal element have been studied extensively for aluminum-based, iron-based and nickel-based alloys. These one-principal element alloys mostly have complex microstructures resulting in brittleness, a drop in hardness at elevated temperature and processing difficulties. On the contrary, HEAs are characterized by their high configurational entropy, which supports the formation of highly disordered simple structures of solid-solutions and suppresses the formation of complex intermetallics. Many interesting properties such as high hardness, high strength, wear resistance and oxidation resistance are generally observed due to the distorted lattice and slow diffusion rate in HEAs. In this project, HEAs based on refractory metals like Hf, Mo, Nb, Ta, Ti, W and Zr are studied with the goal to develop high-temperature structural materials and understand their structure-property relationship.

HEA compositions like $\text{Mo}_{0.25}\text{Nb}_{0.25}\text{Ta}_{0.25}\text{W}_{0.25}$, $\text{Mo}_{0.20}\text{Nb}_{0.20}\text{Ta}_{0.20}\text{W}_{0.20}\text{V}_{0.20}$ and $\text{Ta}_{0.20}\text{Nb}_{0.20}\text{Hf}_{0.20}\text{Zr}_{0.20}\text{Ti}_{0.20}$ have been studied for their phase-stability, disorder, hardness and strength with respect to the long-term aging. All the synthesized refractory HEAs form a body-centered cubic (BCC) disordered phase in the as-cast and also in the long-term annealed homogenized (900-1200°C) samples. X-ray diffractions on single crystals reveal some weak streak-like diffuse intensities adjacent to the (110) reflections. The asymmetric nature of the diffuse scattering might be due to a short-range order of one or more elements combined with atomic size differences. The hardness (450-600 Vickers hardness at ambient temperatures) of the synthesized refractory HEAs can be three to four times higher with respect to that of the hardness of pure elements. The hardness did not decrease with long-term aging/annealing (1-8 weeks), unlike most of the conventional metallic materials. This provides a potential way to achieve high-temperature (beyond 1200°C) sustaining materials by simple melting, casting and homogenization.

L-IT 23

Complex intermetallic alloy phases in the Al-Mg-Zn systemG. Kreiner¹, R. Berthold¹, U. Burkhardt¹, Y. Prots¹, A. Amarsanaa¹, W. Carrillo-Cabrera¹, M. Mihalkovic²¹Max-Planck-Institut für Chemische Physik fester Stoffe, Inorganic Chemistry, Dresden, Germany²Slovak Academy of Science, Institute of Physics, Bratislava, Slovakia

The Al-Mg-Zn system is important for the development of light and high strength alloys. They are used, for instance in automobile and aerospace construction and have been investigated therefore frequently in the past. In addition, the Al-Mg-Zn system has attracted much interest because four complex metallic alloy phases, called τ_1 [1], τ_2 , q [2], and Φ [3] form as ternary intermetallic compounds. We started a reinvestigation of the Al-Mg-Zn system in order to clarify the structure and the homogeneity ranges of the Φ phase and the τ_2 phase. The Φ phase has a wedge-like shape of the homogeneity range of about 13 at.% for Al and Zn and 2-3 at.% for Mg [3]. The homogeneity range of the τ_2 phase is about 3 at.% for Al and Zn. During the investigation a number of new phases in the vicinity of the τ_2 phase and the Mg-Zn subsystem have been observed. Three new compounds, a Zn-rich isotypic structure of the β phase Al_3Mg_2 with 1168 atoms per unit cell, a decagonal approximant with 308 atoms per unit cell and a Laves block phase with 1488 atoms per unit cell will be discussed.

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L-TA 01

Microstructure and deformation mechanisms of a γ -TiAl intermetallic alloy – an *in situ* experimental study
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γ -TiAl alloys are important intermetallics targeted for high temperature aerospace applications in low pressure turbines (LPT) because they can provide increased thrust-to-weight ratios and improved efficiency. LPT materials must operate in aggressive environments at temperatures up to 750°C, where γ titanium aluminides present reasonably good creep and oxidation resistance. Thus, such alloys have the potential to replace the heavier Ni-base superalloys currently used. However, their high-temperature deformation behavior must be better understood in order to optimize their microstructure.

The γ -TiAl intermetallic alloy, Ti-45Al-2Nb-2Mn(at.%) - 0.8v.%TiB₂, was processed by centrifugal casting. Finer and coarser lamellar microstructures were obtained after two different heat treatments. The relationship between the microstructure and the deformation mechanisms of the intermetallic alloy was studied by means of *in situ* tensile experiments at 580°C and 700°C and tensile-creep tests at 700°C inside a scanning electron microscope (SEM). In addition, electron backscatter diffraction (EBSD) was performed before and after straining. From the EBSD orientation maps possible crystal rotations could be suggested at defined conditions. Dislocation slip was proved under the lowest temperature and the highest strain rates at the coarser microstructures, while colony boundary and interface sliding was more dominant at the refined microstructures under high temperatures and low strain rates conditions.

L-TA 02

Defect arrangements in a two-phase γ -TiAl alloy investigated by scanning and transmission electron microscopy
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Different methods of scanning and transmission electron microscopy (SEM, TEM) were applied on a γ -TiAl alloy TNB-V5 after a thermo-mechanical fatigue test. Electron channelling contrast imaging (ECCI) and electron backscattered diffraction were carried out on bulk specimen. In addition, ECCI and scanning transmission electron microscopy in the SEM were carried out on a TEM-foil in the electron opaque and the electron transparent region, respectively. The investigations were completed by transmission electron microscopy in form of standard bright field imaging as well as by taking corresponding diffraction patterns.

The aim of the presented studies is to demonstrate that the application of the ECCI technique on bulk specimens as well as on TEM-foils is a very powerful method to study the arrangements of deformation induced defects like micro twins, stacking faults and/or regular dislocations. The results obtained from combined EBSD and ECCI investigations on the bulk specimen demonstrate vividly that dislocation structures can be visualized both in the globular γ -TiAl grains as well as in the γ -TiAl lamellae of the lamellar regions (alternating γ -TiAl and α_2 -Ti₃Al lamellae). The visualized defect types and/or arrangements by application of ECCI technique are in resolution comparable to those obtained from STEM or TEM investigations.

It becomes evident from the presented ECCI investigations that on a bulk specimen the investigation of dislocation and/or defect arrangements can be performed with a reasonable good statistics. In combination with EBSD it is even possible to investigate the influence of crystallographic orientation with a reasonable time exposure.

L-TA 03

Phase equilibria among β , $\alpha(\alpha_2)$ and γ Phases in Ti-Al-M₁-M₂ quaternary systems at elevated temperature

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The stability of β -Ti phase at elevated temperature is very important for the development of wrought TiAl alloy from the viewpoints of hot free-forging in process and toughening in service. We proposed the design concept for wrought TiAl alloy by using a unique transformation pathway of $\beta + \alpha \rightarrow \alpha \rightarrow \beta + \gamma$ based on our phase diagram study on Ti-Al-M (M: V, Nb, Cr, Mo) ternary systems. We already proved that the alloys can be hot free-forged with no damage in the $\beta + \alpha$ two-phase region and become tougher by controlling the microstructure to fully lamellar microstructure with α particles precipitated at the lamellar interfaces. In this study, phase equilibria among β , α and γ phases in Ti-Al-M₁-M₂ quaternary systems at elevated temperatures were examined in order to identify the phase stability of β phase by combined addition of the b stabilizing elements. The alloys with compositions of Ti-42Al-4Nb-xM (at.%) (M: V, Cr, Mo; $0.5 \leq x \leq 4.7$) were selected. They correspond to Ti-42Al-(6.4-10)Nb^{eq} quasi-ternary alloys under the Nb equivalency of M, $k_{Nb/M} = 1.2, 2.1$ and 4.7 , respectively, estimated from the Ti-Al-M ternary phase diagrams. The alloys homogenized at 1673 K (β single phase region), followed by water quench, were equilibrated at 1473 K for 2 weeks. Microstructure analysis was done by FE-SEM and EPMA. The critical composition of Nb to have thermodynamically stable β phase, $[X_{Nb}^c]$ is 9 at.% in Ti-Al-Nb ternary system at 1473 K. However, some of the quaternary alloys exhibit the β phase even below the total Nb equivalency less than 9 at.% ($[X_{Nb}^c]^{eq} < [X_{Nb}^c] = 9$). This apparently indicates the existence of negative interaction between Nb and M atoms to stabilize β phase. The degree of interaction among the M elements and the composition dependence will be presented.

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L-TA 04

Effect of molybdenum on the microstructure evolution and grain refinement in an intermetallic TiAl alloy system

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Molybdenum, being a strong β -stabilizer, is an important alloying element in TiAl alloys, since the occurrence of the α_2 -phase can more or less be suppressed. As a result, the so-called β/γ -alloys are formed with microstructures in which homogeneously distributed β - and γ -phases are the main constituents.

At higher Mo contents the high-temperature β -phase can indeed be retained upon quenching; only an ordering reaction is observed. This behavior is related to changes in microstructure and hardness when compared to the initial asHIPed condition. A subsequent long-term heat treatment leads to a pronounced grain refining effect and to a significant increase in hardness. Unfortunately, the effect of Mo on the individual phases and their transition temperatures is not completely known. For this reason, the fine-grained microstructure of a β -solidifying intermetallic Ti-44Al-7Mo-0.1B (in at.%) alloy is analyzed in this study. After the subsequent heat-treatments, the volume fraction, the specific surface and the morphology of the corresponding ordered phases β_o -TiAl and γ -TiAl are studied by means of scanning and transmission electron microscopy, electron backscatter diffraction, focused ion beam/SEM tomography, 2D and 3D image analysis as well as high energy X-ray diffraction. Local texture analysis reveals nano-sized γ precipitates formed in the β_o matrix with $\{111\}$ γ -TiAl parallel to $\{110\}$ β_o -TiAl. The 3D morphology shows an interconnected network for both phases even across former grain boundaries, a fact which is not deduced from 2D image analysis. Further on, in-situ investigations using neutron radiation have been introduced to determine the ordering temperatures. All the results of these investigations, combined with the results of metallographical characterization, are compared and discussed.

L-TA 05

Interplay between chemical disorder and Mo content on mechanical stability of cubic body centred TiAl

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Titanium aluminides are a prominent class of lightweight, creep and oxidation resistant materials which are used in automotive and aircraft industry. In order to overcome some of its inferior properties, such as limited hot-workability, various concepts including alloying and sophisticated processing routes have been explored and developed. One of the most promising novel alloys is the TNM alloy containing Nb and Mo which stabilise the disordered body centred cubic (bcc) TiAl phase (β), thus aiding its hot-workability. Depending on the Mo content, the disordered β -phase orders to B2 structure (β_0) at certain temperature.

We report on first principle calculations employing Density Functional Theory to study the interplay between effects of Mo alloying and chemical disorder on the stability of bcc ordered and disordered phases. Enthalpy of formation of the bcc phases is higher than that of the γ -phase which is thermodynamically preferred at low temperatures. However, an evaluation of elastic constants reveals that the β_0 -phase is also mechanically unstable at low temperatures in the binary Ti-Al system. This is further confirmed by calculating the phonon dispersion curves exhibiting imaginary frequencies.

Replacing Al with Mo while keeping constant Ti content of 50 at.% results in stabilising the cubic phase, i.e. in satisfying the Born stability criteria. Surprisingly, our calculations of elastic properties indicate that a lower Mo content is needed to stabilise the disordered β -phase than to stabilise its ordered variant. Similar trend is also confirmed by phonon calculations. Based on a careful analysis of local deformations and the electronic structure we propose that the disorder breaks the strong tendency for a tetragonal deformation towards the structure of the ordered Ti-Al interactions, thus represents an additional stabilising mechanism.

L-TA 06

Microstructural evolution in γ titanium aluminides during severe hot-workingU. Fröbel¹, A. Stark¹¹Helmholtz-Zentrum Geesthacht, Metal Physics, Institute of Materials Research, Geesthacht, Germany

Hot-working of γ titanium aluminide alloys often lead to incompletely transformed and inhomogeneous microstructures that show crystallographical and morphological textures. The textures reflect the plastic anisotropy of the alloys resulting from the lack of independent slip systems. The microstructural inhomogeneities, on the other hand, result from the low dislocation mobility impeding deformation and the low mobility of the lattice atoms and grain boundaries hampering recrystallization. The deformation strain must therefore be increased significantly to intensify the recrystallization processes and obtain more homogeneous microstructures.

During severe deformation, however, the phenomena of shear localization and subsequent crack formation can be particularly pronounced. They are caused by the plastic anisotropy of the lamellar structure. Shear localization processes are difficult to avoid because the lamellar structure is the major microstructural constituent of the alloys in the as-cast state. But they can largely be suppressed by using hot-working techniques which produce small shear stresses. Extrusion is favourable in this respect, because hydrostatic stresses appear in addition to relatively small shear stresses effectively preventing the formation of shear zones and cracks. In an attempt to achieve high strains and simultaneously counteract texture formation the material was extruded twice and the two extrusion directions were oriented perpendicular to each other. The resulting microstructures were characterized with regard to homogeneity and texture using electron microscopy, synchrotron radiation as well as mechanical testing techniques. The findings and also the shear localization mechanisms will be discussed.

L-TA 07

The effect of initial microstructure on the forgeability in β contained TiAl alloyS. Takahashi¹, K. Kubushiro¹, M. Takeyama¹¹IHI corporation, Research Laboratory, Yokohama, Japan

The γ alloy of Ti-43Al-5V-4Nb can be hot-free forged with no difficulty along with our design principle using β -Ti phase, even though the alloy is in as-cast condition which consists of mainly lamellar grains. In this study, microstructure control of bar stock to optimize forgeability of the alloy was examined based on the equivalent Ti-Al-V ternary phase diagram. The bar stock for the compression tests were heat treated prior to compression tests.

The alloy heat treated in the $\beta + \alpha$ two-phase region, exhibits an equiaxed α grains with the grain boundaries decorated by the β phase, where as the alloy heat treated in the ($\beta + \alpha + \gamma$) three-phase region, exhibits a finer-grained triplex microstructure consisting of β , α and γ grains. High temperature compression tests of the specimens which have the above microstructures revealed that the lowest peak stress in the triplex microstructure at 1200°C, strain rate 10s⁻¹. The effects of TiB on forgeability in Ti-43Al-5V-4Nb alloys will be also presented.

L-TA 08

Cost Efficient thermo-mechanical processing of a β -stabilized γ -TiAl alloy for aircraft engine applicationD. Huber¹, M. Stockinger¹, H. Clemens²¹Böhler Schmiedetechnik GmbH & Co KG, Kapfenberg, Austria²Montanuniversität Leoben, Department Physical Metallurgy and Materials Testing, Leoben, Austria

Due to the strong demand for higher efficiencies, reduced CO₂ and NO_x emissions and weight reduction in aircraft engines, the substitution of presently used nickel based alloys by novel light-weight, high-temperature alloys like γ -TiAl based alloys has started already. The requirement for balanced high mechanical properties of low pressure turbine blades in new generation aircraft engines favors a hot working strategy. Thermo-mechanical processing of γ -TiAl based alloys is a complex and challenging task due to a small processing window. Isothermal forging, as state of the art process for this material group, results in high productions costs and lower productivity. Due to these facts Bohler Schmiedetechnik GmbH & Co KG has developed a higher efficient near conventional thermo-mechanical processing technology. Lower die temperature and processing at standard atmosphere as well as the use of standard hydraulic presses with higher ram speed results in a highly economical process. Subsequent heat treatment strategies can be used to tailor microstructure and therefore mechanical properties according to customer needs.

The paper summarizes our effort to establish a "near conventional" forging route for the fabrication of TiAl components for aerospace industry. The path from lab scale compression tests for material data generation via finite element modeling to industrial scale forging trials and mechanical properties evaluation is reported.

L-TA 09 – withdrawn

L-TA 10 – withdrawn

L-TA 11

Microstructural deformation and fatigue damage in γ -TiAl produced by additive manufacturing

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In order to design efficient and light components for the aircraft industry preserving the safety of the design, more sophisticated design criteria are required for the application of new materials. In particular, the usage of novel manufacturing processes to produce advanced materials as the γ titanium aluminide alloys (γ -TiAl) requires the investigation of the influence of the microstructure in the local damage accumulation processes.

In this work, a Ti-48Al-2Cr-2Nb alloy obtained with a additive manufacturing technique by electron beam melting (EBM) has been examined by conducting monotonic and fatigue experiments both on tension and compression samples.

The full-field residual strain maps corresponding to different applied stress levels and number of cycles are obtained through the use of high-resolution Digital Image Correlation (DIC). The local strain heterogeneities were measured out of the load frame by means of an optical microscope at high magnifications. The strain maps were then overlaid with the images of the microstructure and detailed analyses were performed to investigate the features of the microstructure where high local strain heterogeneities arise. High local residual plastic strains were measured inside lamellae colonies, which are detected as the precursor to crack initiation. The measure of the residual strains also provides further information on the role of the intermetallic phases on the deformation behavior of γ -TiAl alloys.

L-TA 12

Low cycle fatigue-creep behaviour of cast TiAl-7Nb alloy at high temperatureM. Smid¹, K. Obrtlík¹, M. Petre nec², T. Kruml¹¹Institute of Physics of Materials, Academy of Sciences of the Czech Republic, Brno, Czech Republic²Tescan a.s., Brno, Czech Republic

Low cycle fatigue-creep behaviour of the cast TiAl-7Nb alloy was studied at 750°C in air. Continuous cyclic tests and cyclic tests with 10 minutes dwells were conducted under strain control with constant total strain amplitude and total strain rate. The dwell was introduced in the tensile tip of each hysteresis loop. Plastic strain amplitude, stress amplitude and stress relaxation data were evaluated from recorded hysteresis loops.

Cyclic hardening/softening curves, cyclic stress strain curves and fatigue life curves were obtained for both fatigue and fatigue-creep tests. The introduction of tensile dwells leads to significant changes in the stress response of the material. Stable cyclic behaviour during continuous cycling changed to cyclic softening followed by cyclic hardening. However, no detrimental effect of tensile dwells on the fatigue life was observed.

Selected specimen gauge lengths were thoroughly observed by scanning electron microscopy with the aim to describe surface relief developed after both types of tests. Frequent slip markings found after continuous cycling didn't exhibit any changes with the introduction of tensile dwells. Transmission electron microscope observations revealed substantial phase transformations in the structure of the material due to synergistic influence of prolonged thermal exposure during cyclic tests with tensile dwells. The phase transformations can be responsible for variations observed in cyclic stress response of the material.

L-TA 13

Thermomechanical fatigue of a high-strength multiphase titanium aluminide alloy (TNB-V2)A. El-Chaikh¹, F. Appel², H.- J. Christ¹¹Institut für Werkstofftechnik/Universität Siegen, Siegen, Germany²Institute for Materials Research/Helmholtz-Zentrum Geesthacht, Geestacht, Germany

In this study the thermomechanical fatigue (TMF) behaviour of a high-strength γ -base TiAl intermetallic alloy TNB-V2, for high-temperature applications as turbine blade in aero engines, was studied. Strain-controlled TMF tests under both in-phase (IP) and out-of-phase (OP) loading were performed at two strain amplitudes (0.4% and 0.6%) applying a constant minimum temperature of 350°C. The maximum temperature ranges from 650°C to 850°C. The tests were performed in air and under vacuum.

The TMF performance depends on the testing parameters and test environment. Large strain amplitudes, high maximum test temperatures and testing in air reduce the TMF life for both IP and OP loading. IP TMF produces significant continuously developing compressive mean stresses with the result that the TMF life is significantly longer than that for OP loading, and for the lower strain amplitude applied even longer than the fatigue life under isothermal low cycle fatigue. The degradation of the fatigue performance under OP loading in air is partially associated with environmental embrittlement due to the formation of an embrittled surface layer. The effect is enhanced at small strain amplitudes.

The investigations were supplemented by transmission electron microscope (TEM) examination using both conventional and high resolution techniques. The alloy investigated exhibits a very complex microstructure, which, upon OP TMF, degrades at a very fine scale. The mechanisms involved will be demonstrated. Particular emphasis is placed on stress induced phase transformation and recrystallization.

L-TA 14

Mechanism study on rare earth microalloying to improve the thermoplastic behavior of powder metallurgical beta γ -TiAl alloy

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The development of novel beta γ -TiAl alloys is an important direction to promote the practical use of titanium aluminum based intermetallics. Based on self-developed Ti-45Al-3Fe-2Mo (at.%) alloy, this application is to study the composition of powder metallurgy technology selected on the basis, the microstructure evolution, the thermoplastic mechanism and some other critical infrastructure problems such as tracing the rule of rare earth elements on powder metallurgy green body forming and microstructure evolution, the impact of rare earth elements on high temperature resistance to oxidation and high temperature strength and the thermoplastic deformation behavior, as well as hot rolling and superplastic behavior. This subject will further clarify a series of scientific laws in powder metallurgy method in the design and preparation of low-cost, high-performance TiAl alloy process. It also will form a powder metallurgy beta γ -TiAl alloy composition with independent intellectual property and the prototype of containerless sheet rolling and superplastic forming technology.

L-TA 15

Thermo-mechanical fatigue behaviour of HVOF coated third generation γ -TiAl alloy

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The thermal spray process HVOF (high velocity oxy-fuel) ensures coatings with low porosity, low defect density and good applicability on different surfaces. In the field of coatings for turbine blades the HVOF process is well established and even provides repair coatings. In order to demonstrate the potential to extend the fields of application of γ -titanium-aluminide alloys, this study characterises the influence of a HVOF thermal sprayed coating on the thermo-mechanical fatigue behaviour of the 3rd generation γ -TiAl-alloy TNB-V5.

The group of titanium aluminide alloys offers excellent specific strength at high temperatures and appropriate high temperature oxidation properties. Due to the operation conditions in motor engines and turbines the used components undergo a cyclic mechanical and a cyclic thermal load. Hence, isothermal low cycle fatigue (LCF) or thermo-mechanical fatigue (TMF) can occur. Therefore, strain controlled LCF and TMF tests have been carried out at a temperature range from 400°C to 800°C and mechanical strain amplitudes from 0.0045 to 0.0065. These tests were carried out at the substrate material, the coating-substrate compound and the stand-alone coating material. At small strain amplitudes in LCF tests, the fatigue lives of the coated specimens were comparable to those of the uncoated samples. The phase-angles of the TMF tests were 0° (in-phase, IP) and 180° (out-of-phase, OP). The post-mortem SEM investigations, with special emphasis to the coating-substrate interface, exhibit a Ni-rich diffusion zone and only slight delaminating secondary fatigue cracks in the interface.

L-TA 16

Influence of the composition of the liquid activation agent for the halogen effect on technical γ -TiAl-alloys
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TiAl-intermetallics show great potential for application in high temperature-components due to their low density and excellent high-temperature strength. Their major drawback is the insufficient oxidation resistance at temperatures above 750°C. It has been shown that one way to improve this resistance by several orders of magnitude consists of the fluorination of the surface zone of the material. This fluorine treatment is performed at low temperatures and influences only the surface region of the components, so that the bulk properties are not affected. The fluorine presence in the subsurface zone of a γ -TiAl-alloy activates a change in the oxidation mechanism during subsequent exposure at temperatures higher than 750°C in such a way that the formation of a thick non-protective mixed oxide scale is replaced by a thin protective alumina scale. This change in the oxidation behaviour of γ -TiAl defines the so-called "positive fluorine or halogen effect". No additional coatings are necessary for the full protection of the substrate against further oxidation. One efficient and simple technique for bringing the fluorine onto the TiAl surface is by using different liquid phases. This work focuses on the influence of the liquid phase composition on the lifetime of the protective alumina layer. Possible beneficial synergistic effects between F and Si on the oxide nucleation behaviour are discussed in comparison to other fluorination methods such as ion implantation. Furthermore the influence of the high temperature exposure technique used for the activation of the fluorine effect (i.e. conventional furnace exposure versus short-time laser treatment) on the protective effect of the alumina scale is examined.

L-FA 01

Comparison of the microstructure and high temperature strength of ODS Fe-Al-Cr Intermetallics with ODS binary Fe-Al and Fe-Cr

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The introduction of stable oxide particles by mechanical milling is well established as a way to obtain excellent high temperature strength and creep resistance in a wide range of materials. The present study compares the microstructures and high temperature strength of oxide-dispersion-strengthened (ODS) ternary Fe-Al-Cr intermetallic alloys and their ODS binary Fe-Al and Fe-Cr parent alloys. The ternary alloys examined have Al contents of 30-40% (atomic) and Cr contents of 5-10%, and contain 0.5-1% (volume) of yttria. The grain sizes and oxide dispersions obtained are examined, and relationships between oxide particle size and distribution and grain size considered. The fineness of the oxide particles depends on the ease of particle nucleation during processing, with oxide particles being finer in the ternary alloys because of the formation of an easily nucleated metastable coherent oxide. Material strength has been determined from room temperature up to 800°C and creep behaviour studied above 700°C. The ternary alloys generally show higher strength than the binary alloys, related to differences of grain size and particle sizes and numbers, with the matrix strength also playing a significant role in defining the material strength. Creep resistance of the ternary alloys is also considerably better than for the binary ODS alloys, again explained by the presence of finer oxide particles, which act as strong obstacles to the propagation of the individual superdislocations.

L-FA 02

Strengthening mechanism of Fe-Al-based alloys containing B2-type precipitates

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Deformation behavior of Fe-Al-Ni and Fe-Al-Co single crystals containing the B2-type precipitates was reviewed. In the Fe-23Al-Ni and Fe-15Al-15Co (at.%) crystals, the NiAl and CoAl phases were precipitated in the bcc matrix satisfying the cube-on-cube orientation relationship, respectively. These crystals showed a high strength even at 823-923 K. The activated slip system of the Fe-Al-Ni and Fe-Al-Co single crystals at room temperature depended strongly on loading axis. At orientation, both the crystals deformed by {110} which is favorable for the bcc matrix. The NiAl and CoAl precipitates were cut by a pair of 1/2 dislocations in the bcc matrix. However, the primary slip systems of the NiAl and CoAl single phases are known to {110} and {010}, respectively. Slip was difficult to occur in the B2 precipitates resulting in high strength. In contrast, {110} and {010} slip took place in the Fe-Al-Ni and Fe-Al-Co crystals at appropriate loading axes. Slip was generally impossible to occur in the bcc matrix, which also led to strong hardening. The stress increment by the B2-type precipitates was evaluated based on the Fleischer model. It is also noted that temperature dependence of yield stress in these crystals was consistent with the activated slip system and the dissolution of the B2 precipitates.

L-FA 03

Strengthening of Fe₃Al alloys at high temperatures with grain boundary κ-Fe₃AlC precipitate films

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We have been investigating the effects of grain boundary (GB) precipitate films on strengthening Fe₃Al based alloys at high temperatures. In the presentation, firstly, we will present that GB precipitates can be more effective than fine matrix precipitates in the strengthening, based on the strength levels experimentally obtained by tensile tests at 600°C in samples with different types of microstructures: (I) GBs covered with film-like κ-Fe₃AlC type carbide precipitates by more than 80% in area fraction and grain interiors (GIs) strengthened by fine M₂C type carbide precipitates, (II) No GB precipitate films but GIs strengthened by a high density of fine M₂C particles, and (III) GBs and GIs without appreciable second-phase particles.

Secondly, we will show that a Hall-Petch coefficient changes from a negative value to a positive value by introducing GB κ precipitate films, based on the 0.2% stresses at 600°C experimentally obtained from samples with different matrix grain sizes each in the presence/absence of GB κ precipitate films. Thirdly, we will explain the roles of GB precipitate films on the high temperature strengthening of Fe₃Al alloys, based on the comparison of dislocation structures at GBs in specimens with and without GB precipitate films.

L-FA 04

Deformation and dynamic recrystallization behaviour of two Fe-Al-Nb alloys reinforced with the Laves phase fibres
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Fe-Al based alloys have been studied extensively to exploit their high-temperature properties, in particular their good resistance to oxidizing environments. Melting and fabrication techniques have been developed which make it possible to produce wrought products from commercial-size heats. However, the hot workability of Fe-Al alloys can be a problem. The occurrence of surface and internal cracks has been reported during hot forging of alloys.

In the present work high-temperature deformation behaviour and workability of two Fe-Al-Nb alloys reinforced with Laves phase fibres have been investigated. Alloys with compositions Fe-15Al-10Nb and Fe-26Al-9.5Nb were induction melted and drop cast into copper moulds. The hot deformation behaviour of as-cast Fe-Al-Nb alloys has been characterized on the basis of their flow stress variation obtained by isothermal constant true strain rate compression testing in the temperature range 700-1100°C and strain rate range 0.001-100 s⁻¹. The mechanisms of hot working have been evaluated using different techniques, which included shape of stress-strain curves, kinetic analysis, processing maps and dynamical systems approach.

Several types of deformation behaviour were observed and the data were fitted to general constitutive equations to determine the hot working constants of these alloys. The effect of Zener-Hollomon parameter on the characteristic points of flow curves was studied using the power law relation, and the dependence of critical stress on peak stress was determined. In addition, based on the obtained results, processing maps were calculated to evaluate the efficiency of the hot working and to identify the instability regions of the flow behaviour. General outline of the maps does not change with the strain level and at both strain levels, there is only one domain with maximum efficiency, which increases with the temperature. Moreover, dynamic recrystallization behaviour was evaluated by using a mechanical approach based on the analysis of the work hardening rate. Many samples exhibited typical DRX stress-strain curves with a single peak stress followed by a gradual fall toward the steady-state stress. Finally, the deformation activation energy and the dynamic recrystallization kinetics model of the investigated alloys were established.

L-FA 05

Twinning and shear localization during high strain rate deformation of Fe₃Al

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To identify the limits of so far known deformation mechanisms, increasing the strain rate is a common tool to deepen the understanding of those mechanisms. The influence of strain rate at elevated temperatures on the mechanical response of iron-aluminides has been studied predominantly on B2-ordered FeAl-alloys to verify the model of vacancy hardening by George and Baker. In contrast, information about the mechanical response of D0₃-ordered Fe₃Al at high strain rates is very limited, both at ambient and elevated temperatures.

Hence, it is the purpose of this talk to extend the knowledge on deformation characteristics of D0₃-ordered Fe₃Al over a wide range of deformation rates. The mechanical response of centrifugally cast Fe₃Al with composition Fe-27Al (at.%) containing micro-alloying additions of Nb, Zr, C, and B is investigated at room temperature and strain rates between 10⁻⁴ and 2400 s⁻¹.

Tests were carried out in compression using a screw-driven load frame at low, a drop impact tester at intermediate, and split Hopkinson pressure bar at high strain rates.

The formation of twins and shear localization was observed when exceeding a strain rate of 10^3 s^{-1} . The twins and the locally sheared areas were characterized by EBSD, TEM and mechanical testing of micro-pillars machined by a focused ion beam (FIB) instrument. The strength of the micro-pillars inside and outside the locally sheared areas of the high strain rate deformed sample is on the same level. However, the microstructures in those areas are quite different. Inside the locally sheared areas subgrains with sizes of about 100 nm are formed, whereas a homogenous distribution of dislocations was observed in uniformly deformed areas. The twinning system was characterized by TEM and the critical strain rate for plasticity by twinning was determined. In general the mechanical response is in good agreement with the microstructural observations.

L-FA 06

The EBSD study of recrystallization in rolled and hot deformed Fe-40 at.% Al- 0.1 at.% Zr-0.13 at.% B alloy
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The rolling of B2-based iron aluminides is problematic and usually not successful process. Even the hot rolling leads to significant intergranular cracking [1]. That's why the applied alloys developed in past decades, as Pyroferal [2], were just cast. The successful rolling come through the application of two technological steps [1,3]: the vacuum casting into the ultrasound forced mold and the using of the sheet bandage as a special hot-rolling protection preventing thermal shocks.

Those procedures improve structure of the rolled material significantly. The ultrasound forcing of the melt during solidification improves the mold filling, prevents formation of shrinkage cavities in the cast piece and refine grain size, which is related mainly to enhanced nucleation. The rolling of the bandaged material prevents thermal shock cracking caused by the direct contact between hot material and cold rolls. The cooling effect of the surrounding atmosphere is suppressed as well.

Here we present recrystallization study of Fe-40 at.% Al- 0.1 at.% Zr-0.13 at.% B alloy. The EBSD method is used in present study to investigate recrystallization in rolled materials and in materials being model for them. The rolling process is optimized by the defined deformation of the model samples in Gleeble 3800 plastometer. In such samples, processes of dynamic and static recrystallization can be distinguished using various thermomechanical treatments following the hot deformation. The first set of samples was quenched immediately after the deformation, whereas the second set was annealed at defined conditions before quenching. The features in the microstructure of samples give the parameters of thermomechanical behavior, which can be used to optimize rolling process. The examples of rolled samples exploiting previous study document the asset of used methodology [4-6].

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L-FA 07

Effect of thermodynamics on physical properties of ternary iron aluminidesC. G. Schön¹, L. T. F. Eleno^{1,2}, H. M. Petrilli²¹Escola Politécnica da Universidade de São Paulo, Metallurgical and Materials Engineering, São Paulo, Brazil²Instituto de Física da Universidade de São Paulo, Materials Physics and Mechanics, São Paulo, Brazil

Physical properties of materials are always defined in situations where mass, momentum or energy transport takes place in the system, i.e. necessarily outside thermodynamical equilibrium. Nevertheless, equilibrium thermodynamics has an impact over these properties, by setting the boundary conditions which constrain the solution of the non-equilibrium problem. In the case of aluminides, for instance, this is particularly complex. Long-range (LRO) and short-range order (SRO) effects introduce non-linear contributions to the composition dependency of the chemical potentials and internal energy, which impose non-trivial composition dependencies for the properties. This will be illustrated by considering two technologically important non-equilibrium processes: ternary diffusion and antiphase boundary formation. Ab-initio calculations using the Full Potential- Linear Augmented Plane Wave (FP-LAPW) method are performed in systems Fe-Al-M (M=Ti,Mo,Nb) and are employed to obtain the basic parameters for the complete thermodynamic model of the body centered cubic (bcc) phase and of its superlattices, in the framework of the irregular tetrahedron cluster approximation of the cluster variation method (CVM). By employing specific algorithms, isopotential lines and antiphase boundary isoenergy lines are calculated and plotted against the (eventually metastable) aluminide stability field in the phase diagram. It is shown that complex non-linear effects, like slope discontinuities, complex curvatures and isoline concentrations take place in special regions of the phase diagram. These results are discussed with regard to the shape of diffusion paths in diffusion couples and to superdislocation geometry and its impact over plastic deformation.

L-FA 08

Atomic relaxation processes at very high temperature in a fine grain B2 Fe-Al intermetallicG. A. López¹, P. Simas², L. Dirand¹, M. Nó¹, J. San Juan²¹Universidad del Pais Vasco, Física Aplicada II, Bilbao, Spain²Universidad del Pais Vasco, Física Materia Condensada, Bilbao, Spain

Intermetallic materials are being developed for many high temperature applications and consequently its behaviour and stability must be carefully characterized and understood. Mechanical spectroscopy gives a unique insight into the mechanisms of defect's mobility, and internal friction measurements have been performed on many intermetallic alloys and in particular on FeAl. In this work, a high-temperature mechanical spectrometer, working up to 1800 K, is used to perform internal friction and dynamic modulus variation measurements during recrystallization of an advanced oxide dispersion strengthened Fe-38%Al intermetallic, which takes place at 1505 K. In parallel, the microstructural evolution has been characterized by scanning (with EBSD) and transmission electron microscopy.

In the recrystallized state, a relaxation peak is observed at about 750 K, which is attributed to the stress-induced reorientation of Al atom-pairs. In order to better understand the effect of the microstructural aspects on the mechanisms controlling plasticity at high temperatures, the atomic mobility processes at very high temperature, but avoiding recrystallization, have been studied. A new relaxation peak at about 1350 K has been observed, superimposed to the high temperature background, which has been studied as a function of temperature for different frequencies. A deconvolution of the internal friction spectra at different frequencies has been performed, allowing the determination of the activation enthalpy, $H_{act}=5.4\pm 0.3$ eV. The broadening of this peak corresponds to the typical one of point defects relaxation, and two different potential interpretations have been suggested and discussed.

L-FA 09

High-temperature measurements of thermal expansion, lattice parameter and elastic moduli in B2-type FeAl
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B2-type FeAl contains a high concentration of thermal vacancies at high temperature because of its low vacancy formation energy. A number of early works had investigated quenched-in vacancy behavior through room temperature measurements since vacancies formed at high temperature were easily frozen into the material upon cooling due to a low vacancy migration speed. However, in order to make better understanding of thermal vacancy behavior at a corresponding temperature, high-temperature measurements of thermal vacancies were necessary. In this study, samples with the composition of Fe-40 and -43Al (at.%) were prepared and the temperature dependence of the thermal expansion, lattice parameter and elastic moduli were investigated to study thermal vacancy behavior at elevated temperature. It was found that the thermal expansion of the samples increased in a quadratic way while the lattice parameter linearly increased with increasing temperature. From the divergence between the sample length and the lattice parameter changes, thermal vacancy concentration was evaluated and it was indicated that the vacancy concentration exponentially increased with increasing temperature. On the other hand, the elastic moduli linearly decreased with increasing temperature. Therefore, neither the size nor modulus effect of thermal vacancies was observed at elevated temperature, suggesting that the solid-solution hardening by thermal vacancies does not work at elevated temperature in B2-type FeAl.

L-FA 10

Structure and stability of the γ brass-type high-temperature phases in Al-rich Fe-Al(-Mo) alloys
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The effect of Mo additions on the stability and crystal structure of the high-temperature phase Fe₃Al₈ (frequently called ϵ phase) has been investigated. From previous studies it is known that the binary phase has a cubic Cu₃Zn₈-type structure (so-called γ_1 brass) [1,2]. On the other hand, in some quenched samples containing Mo, the hexagonal Cr₂Al₆-type γ_2 brass structure was detected at room temperature [3]. A number of ternary alloys containing up to 10 at.% Mo were produced by crucible-free levitation melting and characterized by electron probe microanalysis, differential thermal analysis (DTA), and metallographic investigations. As it is well-known that the binary high-temperature Fe₃Al₈ phase decomposes at 1095°C by a spontaneous eutectoid reaction which can not be suppressed even by rapid quenching, *in-situ* high-temperature time-of-flight neutron diffraction experiments have been performed at the Los Alamos Neutron Science Center (LANSCE) at Los Alamos National Laboratories (LANL). All investigated ternary alloys show phase transformations in the temperature range 1050 to 1150°C in agreement with the DTA results. The high-temperature crystal structures were analysed by a Rietveld method and compared to the binary case.

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L-FA 11

Bending test of micro pillars in solution – a new approach to studying the hydrogen embrittlement of iron-aluminum intermetallics

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This is a study of the effect of hydrogen on the plasticity of a Fe₃Al intermetallic with 26 at.% of Al. The macro scale studies cannot explain precisely the effect of hydrogen on the deformation mechanism of alloys because they are too global to resolve the interaction between adsorbed hydrogen atoms and dislocations. With our novel method, bending of electrochemically charged micro pillars, we tried to study the effect of hydrogen on the dislocations nucleation and their mobility. With the EBSD technique, we identified the orientation of different grains and cut some pillars in the defined orientations, namely (001) and (112), with the aid of the focused ion beam technique. Using cylindrical micro-pillars guarantees the homogeneous distribution of hydrogen atoms after a very short time of cathodic charging. The bending test was performed using a nanoindenter with a two dimensional transducer in order to measure both normal and lateral applied forces. With analyzing the load-displacement curves, we evaluate the effect of hydrogen on the Young's modulus and flow stress. Additionally, with ex-situ SEM measurement, the effect of hydrogen on the form of slip lines was studied.

L-FA 12

Oxidation behavior of binary Fe-Al alloys in air and saturated steam at 700°C

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Currently, there is a focus on the development of power plants with steam temperatures of about 700/720°C. This requires components which are able to withstand very high temperatures and pressure loads caused by flowing steam in the interior of the boiler tubes. Therefore, materials should have good creep strength, high oxidation resistance in the presence of pressurized steam and a good fireside corrosion resistance. It is well known that alumina forming alloys exhibit a superior oxidation/corrosion resistance at high temperatures due to the formation of a protective alumina scale. The aim of this work is to study the effect of water vapor content on the oxidation behavior of binary Fe-Al alloys in order to judge its effect on alumina scale formation and growth, especially with respect to the stability of iron-oxides. Therefore binary Fe-Al-systems with Al-concentrations between 10 and 45 at.% were investigated by isothermal oxidation tests for 1000 h at 700°C in air and steam. The oxide layers which were formed during the tests were analyzed by optical microscopy, SEM and XPS. In general, parabolic rate constant was higher in steam and the oxide scales formed show significant differences in air in steam.

L-PM 01

Selective electron beam melting of γ -TiAlC. Körner¹, J. Schwerdtfeger²¹WTM, Uni-Erlangen, Erlangen, Germany²ZMP, Uni-Erlangen, Fürth, Germany

Recently, additive manufacturing (AM) has emerged as a new attractive processing route of high temperature alloys like super alloys or intermetallics. AM seems to be especially interesting for materials where traditional casting techniques, e.g. investment casting, are difficult due to material deterioration in consequence of reactions with the mold material.

Selective electron beam melting (SEBM) belongs to the powder bed based additive manufacturing technologies. Besides the characteristic advantages of AM technologies there are different aspects which make especially SEBM attractive for the processing of γ -TiAl. The high vacuum prevents the materials from being contaminated by unintended absorption of elements from the atmosphere like oxygen, hydrogen, carbon or nitrogen. In addition, the electron beam can be moved with extremely high velocities and is used for global and local heating during layer by layer building. The high building temperatures prevent crack formation and distortion.

In this contribution, we present the SEBM process for Ti-48Al-2Cr-2Nb. The focus is on the influence of the processing parameters on the consolidation of the powder. Processing maps are presented and discussed with respect to the element distribution within the layers. Special emphasis is on the possible aluminum loss during melting.

L-PM 02

Microstructure of γ -TiAl (48-2-2) produced by selective electron beam meltingJ. Schwerdtfeger¹, V. Jüchter², C. Körner²¹ZMP, Uni-Erlangen, Fürth, Germany²WTM, Uni-Erlangen, Erlangen, Germany

Their physical properties make titanium aluminides very interesting for high temperature applications involving dynamic components. However, processing of TiAl alloys remains to be complex and presents a major obstacle for its more wide spread application. A relatively new approach to this problematic is the use of additive manufacturing in the shape of selective electron beam melting (SEBM). It presents a very attractive route for the production of near net or even net shape parts from TiAl.

In SEBM parts are build layer by layer using an electron beam to locally melt thin layers of metal powder. The local nature of the process leads to a decoupling of external geometry from cooling conditions. These are instead largely governed by the process conditions among which the beam parameters are the most prominent. This removes possible restrictions in terms of aspect ratios of parts and the ability to change beam parameters very quickly also gives the possibility for local microstructure adjustment. In the presented work we discuss the influence of the beam parameters and different scan strategies on the microstructure and properties of TiAl48-2Cr-2Nb for a broad processing window covering scan speed from a few hundred to several thousand mm/s.

L-PM 03

***In situ* SEM analysis of the deformation and fracture mechanisms of a powder metallurgy γ -TiAl alloy**R. Muñoz-Moreno^{1,2}, E. M. Ruiz-Navas², C. J. Boehlert³, M. T. Perez-Prado¹, J. M. Torralba^{1,2}¹IMDEA Materials Institute, Getafe (Madrid), Spain²Universidad Carlos III Madrid, Leganes (Madrid), Spain³Michigan State University, Department of Chemical Engineering and Materials Science, Michigan, United States

γ -TiAl alloys are important intermetallics targeted for high temperature aerospace applications in Low Pressure Turbines (LPT) because they can provide increased thrust-to-weight ratios and improved efficiency. LPT materials must operate in aggressive environments at temperatures up to 700°C, and γ titanium aluminides are projected to replace the heavier Ni-base superalloys currently being used. An analysis on their deformation and fracture mechanisms would allow for better development of methods for microstructural optimization which lead to a delay in crack initiation and propagation, thereby enhancing the intermetallic's life. In addition, since the costs of this material are considerably high, powder metallurgy techniques are well positioned in order to reduce costs of processing.

The γ -TiAl intermetallic alloy, Ti-45Al-2Nb-2Mn(at.%) - 0.8v.%TiB₂, was processed from prealloyed powder and consolidated by hot isostatic pressing (HIP) with posterior heat treatments. The relationship between the microstructure and the deformation mechanisms of the intermetallic alloy was studied by means of *in situ* tensile experiments performed at 580°C and 700°C and tensile-creep experiments at 700°C inside a scanning electron microscope (SEM). In addition, electron backscatter diffraction (EBSD) was performed before and after straining. The incidence of the different deformation mechanisms was analyzed based on the observation of the evolution of the microstructure and the texture with straining. Diffusion based mechanism were suggested from the observations at the higher temperature tests, where intergranular cracking was dominant. However, at lower temperatures and constant strain rates (10⁻³ s⁻¹) interlamellar ledges were observed as well as slip traces along the equiaxed grains areas.

L-PM 04

The effect of zirconium addition on sintering, microstructure and primary creep resistance of TNB-V5 (Ti-45Al-5Nb-0.2B-0.2C)J. Soyama¹, M. Oehring¹, W. Limberg¹, T. Ebel¹, K. U. Kainer¹, F. Pyczak¹¹Helmholtz-Zentrum Geesthacht, Geesthacht, Germany

Since the presence of β phase might be detrimental for the creep properties of titanium aluminides, elements that stabilize the β phase, e.g. niobium, molybdenum, etc. can only be used within a limited range. Neutral elements such as zirconium are promising candidates for heavy alloying without having the side effect of stabilizing the β phase. In this work, the addition of zirconium to TNB-V5 (Ti-45Al-5Nb-0.2B-0.2C, in atomic percent) through elemental powder metallurgy was investigated. Different alloys with zirconium content varying from 1 to 5 at.% were prepared by uniaxial pressing of a mix of elemental and pre-alloyed powders together with a binder system. The mechanical properties were evaluated by hardness and compression creep tests.

The results indicate that zirconium significantly decreases the melting point of the base alloy, which directly influences the sintering behaviour. As the zirconium content increases, the optimum sintering temperature decreases. For the extreme case of 5 at.% zirconium addition, the optimum sintering temperature was almost 100°C lower than for TNB-V5. The primary creep resistance, however, showed a general improvement in comparison to the base alloy.

L-PM 05

Crystallographic modulation of B19 and α_2 phases in γ -TiAlX alloys (X = Nb, V, Mo, B, C)H. Gabrisch¹, U. Lorenz¹, F. Pyczak¹, M. Rackel¹, N. Schell¹, F.-P. Schimanky¹, A. Schreyer¹, A. Stark¹¹Helmholtz-Zentrum Geesthacht, Geesthacht, Germany

Alloys with the base line composition Ti-(40-44)Al-8.5Nb exhibit a good combination of high temperature strength and room temperature ductility. These alloys are characterized by a microstructure that contains the orthorhombic B19 phase in close vicinity to the α_2 phase within lamellar ($\alpha_2 + \gamma$) colonies. In-situ high-energy-x-ray diffraction revealed that the B19 phase forms from α_2 phase by small atom displacements in the α_2 (0001) plane. In transmission electron microscopy the α_2 and B19 phases are recognized by typical contrast variations caused by strain between the two unit cells or by high resolution imaging.

The first Ti-(40-44)Al-8.5Nb alloys were produced by hot extrusion and subsequent heat treatments. In the current work a powder metallurgical processing route is employed which is expected to result in improved chemical homogeneity and a refined grain size. We use in-situ HEXRD to determine the condition where a maximum amount of B19 phase is formed and explore the effect of alloying with V, Mo, B or C on the stability of the B19 phase relative to niobium. The spatial arrangement of the α_2 and B19 phases on the nanometer scale is characterized by transmission electron microscopy.

L-PM 06

Aluminid-based PM intermetallic alloysA. Logacheva¹, I. Logachev^{1,2}, I. Razumovskiy¹, A. Beresnev¹¹JSC "Kompozit", New metallurgical technologies Institute, Korolev, Moscow Region, Russia²University, metallurgy, Moscow, Russia

In report are discussed the advantages and disadvantages of cast intermetallic alloys based on aluminides (system Ni-Al, Nb-Al). In order to minimize or eliminate the major drawbacks of intermetallic alloys proposed transition to a new process for the production of these products – powder metallurgy. The features of doping systems of new generation of powder alloys are examined. The results of experimental studies of products obtained from the intermetallic alloy powder are presented.

L-PM 07

Powder metallurgical production of NiTi parts with fully expressed shape memory propertiesM. Bram¹, M. Bitzer¹, D. Stöver¹, H. P. Buchkremer¹¹Forschungszentrum Jülich GmbH, Institut IEK-1, Jülich, Germany

Within the last years, shape memory alloys based on NiTi gained increasing interest especially for biomedical applications and actuators. Powder metallurgy is an attractive production method which enables manufacturing of near-net-shaped NiTi components with complex geometries, while keeping material losses during processing at a minimum. As starting materials, pre-alloyed NiTi powders are favoured over elemental Ni and Ti powder mixtures if aiming on single-phased microstructures after sintering. In this work, P/M manufacturing of complex-shaped NiTi components with fully expressed shape memory properties is described in detail. Metal injection moulding (MIM) was preferentially used for net-shaping. Prototypes of biomedical foot staple and orthodontic brackets were produced to demonstrate potential of this technology for NiTi alloys. Optionally, functional macro porosity up to 50 vol. % was achieved by introduction of temporary space holder particles, which is quite promising e.g. for bone implants or damping applications. As reference, NiTi components free of pores were produced by hot isostatic pressing (HIP). The paper provides a detailed description of relationship between P/M processing conditions and resulting mechanical and shape memory properties.

L-PM 08

Microstructure and phase constitution of Ti-SiC coatings fabricated by selective laser meltingP. Krakhmalev¹, H. Schwab¹, I. Yadroitsev²¹Karlstad University, Karlstad, Germany²Ecole Nationale d'Ingénieurs de Saint-Etienne (ENISE), DIPI Laboratory, Saint-Etienne, France

The composite coatings were fabricated by selective laser melting (SLM) of Ti-20,30,40 wt.% SiC powder mixtures with PM 100 SLM made by Phenix Systems. The continuous, 1.075 μm wavelength Ytterbium fibre laser of 50W power was used. The rolled on Ti substrate layer of powder of 50 μm thickness, was sintered with scanning speed of 120 mm/s, using the two-zones strategy when a powder layer was processed first with a hatch distance 120 μm then the laser beam passed in between two neighbouring tracks. The total thickness of coatings was 175-200 μm . Electron microscopy of the sintered coatings showed an ultrafine intermetallic/carbide composite structure formed in-situ in the central regions of tracks. The phase analysis revealed TiC_x , $\text{Ti}_2\text{Si}_3\text{C}_x$, TiSi_2 and SiC phases, an actual composition of the coatings depended on the initial SiC content. An equilibrium Ti_3SiC_2 phase was not observed in any of the SLM coatings. The SLM Ti-20,30,40 wt.% SiC composite intermetallic coatings demonstrated high hardness values of 11.0, 15.5 and 17.2 GPa respectively, average hardness of the coatings increased with SiC content. Two types of the structural inhomogeneity were observed in the SLM coatings. Firstly, incompletely remolten particles of SiC phase situated on interlayer interfaces, and secondly, chemical inhomogeneity, enrichment with Ti, in the central regions of sintered tracks. These defects discussed in relation to the SLM process and it was assumed that there were two main reasons for the observed inhomogeneities. Firstly, the laser penetration was not deep enough and some SiC particles left unmolten at the interlayer interface. Secondly, insufficient mass transport in the melt, caused the chemical inhomogeneities in central regions of the SLM tracks. An approach to increase the laser energy input was suggested to optimize a microstructure by influencing of both, laser penetration depth and mass transport in the liquid phase processes to fabricate homogeneous intermetallic coatings by SLM technique.

L-PM 09

Microstructure and mechanical properties of Co-base γ - γ' composites by mechanical alloying and field assisted hot pressingM. Carton-Cordero¹, B. Srinivasa Rao², M. Campos¹, J. M. Torralba^{2,1}¹Universidad Carlos III Madrid, Leganes (Madrid), Spain²IMDEA Materials Institute, Getafe (Madrid), Spain

Co-base alloys strengthened by $\text{L}_{1-2}\text{-}\gamma'$ ($\text{Co}_3(\text{Al,W})$) particles are envisioned to be very promising materials for high temperature applications. In the current study the microstructure evolution of $\text{Co}_{79}\text{Al}_{12.5}\text{W}_{12.5}$ and $\text{Co}_{79.5}\text{Al}_6\text{W}_{12.5}$ alloys during mechanical alloying, after compaction by field assisted hot pressing and its relation to the mechanical properties were investigated. During initial periods of milling Co-hcp phase transformed to the supersaturated fcc phase containing aluminium and partial amount of tungsten and later transformed in to a metastable disordered B2-bcc solid solution. The formation of metastable phase is found to be due to the enhanced defect density, formation of nano grain size, heavy dissolution of tungsten and contamination from the milling media. Sintering resulted in the decomposition of disordered B2 solid solution to the stable phases containing Co-Fcc, Co_3W , Co_7W_6 , CoAlW and L_{1-2} phases depending on the sintering conditions. By choosing proper sintering temperature and thereby controlling the amount of phases and grain size, a high compressive strength of 1.5 GPa was achieved.

L-PM 10

High temperature creep behaviour of nanocrystalline aluminides (NiAl and FeAl) and their *in-situ* Al₂O₃ reinforced composites

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Present study deals with high temperature creep studies on dense nanocrystalline B2 aluminides (NiAl and FeAl) and their composites with *in-situ* Al₂O₃ reinforcement. The monolithic aluminide powders were formed using elemental powders, while *in-situ* composites by reducing respective metal oxides (NiO and Fe₂O₃) by Al in high energy ball mill. During mechanical alloying, the progressive aluminothermic reduction of oxides resulted in forming Al₂O₃ and subsequently the aluminide composites. To ensure the complete reduction of metal oxides (NiO, Fe₂O₃) by Al, milled powders were heat treated at high temperatures. Subsequently, heat treated powders were spark plasma sintered at 1000°C at 50 MPa for attaining maximum density. Compression creep studies were performed on these alloys in the temperature range of 600-800°C and in the stress range of 100-300 MPa. Creep results were analysed together with detailed microstructural characterization studies while comparing with that of monolithic intermetallics.

L-PM 11

Additive manufacturing of a binary iron aluminide by laser metal deposition and selective laser melting

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In recent years, industry showed increasing interest in iron aluminides, especially as structural material for lightweight applications. Beside advantages such as hot corrosion resistance, good wear resistance and low density, one of its disadvantages is the low ductility, due to environmental embrittlement and grain coarsening during casting. Improving the ductility requires refining of the grain size. Grain growth in casting is due to the low temperature gradients during solidification. In contrast, laser additive manufacturing stands for high temperature gradients and fast cooling and therefore can induce a fine microstructure for a better ductility. This presentation shows first results of Laser Metal Deposition (LMD) and Selective Laser Melting (SLM) of the binary Fe-16 wt.% Al alloy. LMD involves feeding of Fe-Al-powder coaxial to the laser beam through a powder feed nozzle. The powder and a thin layer of a substrate material is fused together in a local inert gas atmosphere forming a metallurgical bonding. A bulk volume is build-up by several layers. SLM is performed in a glove box under argon atmosphere. Powder is deposited as thin layer (30-50 μm) on a substrate and selectively melted by a laser beam according to a CAD model. Layer by layer a bulk volume is built-up. Effects of process parameters on the microstructure (phases, porosity, cracks, grain size) are described and discussed. A main focus lies on the correlation between process parameters and grain growth. Using a coarse grained substrate of iron aluminide leads to epitaxial solidification despite the fast cooling during LMD and SLM. Grains of several mm lengths are growing across the single layers oriented in build-up direction. Porosity is low and cracks were not detected. Grain growth can be reduced when using a dissimilar substrate, e.g. stainless steel.

L-PM 12

In situ laser cladding of single-phase iron aluminide on plain steel and pure nickel

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In this work, we produce and study iron aluminide coatings on steel and pure nickel by laser cladding with elemental iron and aluminium powders. The coatings are investigated by optical microscopy, scanning electron microscopy, energy dispersive X-ray spectroscopy and X-ray diffraction. By optimising the cladding parameters, completely single-phase and defect-free coatings can be achieved. The dilution of the substrate material and its influence on the resulting phases is also discussed. In the case of the steel substrate, the composition shifts to higher Fe-contents and in the case of nickel, the system becomes ternary. This work shows not only the possibility of creating protective coatings but is also a fundamental step to a rapid prototyping process which involves the stacking of many iron aluminide layer. It could also be possible to transfer the findings to other intermetallic compounds which are difficult to create by other processing routes.

L-MI 01

Electrodeposition and structural characteristics of as-plated and heat treated intermetallic nickel-tin based coatings
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Materials consisting of intermetallic compounds have been the subject of significant research and development efforts over the past decades due to their peculiar physical, chemical, and/or mechanical properties. In that respect, it is not surprising that there is an increasing interest for intermetallics as innovative coatings for demanding technological applications. For example, the hardness, the tarnish resistance, and the resistance to chemical reagents of nickel-tin alloys exceed by far the ones of either nickel or tin. Nowadays Ni-Sn alloy coatings are widely applied in the electronic, food and watch industry. However, despite the extensive use of nickel-tin alloy coatings, very little is actually known about their synthesis as intermetallic compounds and their intrinsic and extrinsic properties.

Therefore, in this research work intermetallic Ni-Sn based coatings with a Sn content ranging from 10 up to 65 wt.% were electrodeposited on Vanadis 23 steel substrates. Structural and chemical characterization of the coatings was performed by X-ray Diffraction (XRD), Transition Electron Microscopy (TEM), Scanning Electron Microscopy (SEM) and Electron Dispersive X-ray Analyses (EDX). The as-plated Ni-Sn coatings were found to consist of a crystalline Ni solid solution and Ni-Sn intermetallic phases, whereas in certain cases bulk intermetallic coatings were obtained. The addition of Sn as an alloying element into electrolytic Ni coatings led to a significant increase of hardness and Young's Modulus of these coatings, due to the formation of metastable NiSn, Ni₃Sn and Ni₃Sn₂ intermetallic phases. Post-deposition heat treatment at 400°C for 1 h resulted in the formation of stable intermetallic phases corresponding to the ones mentioned in the equilibrium Ni-Sn binary phase diagram and, to a further increase of the hardness and Young's Modulus of these intermetallic coatings.

L-MI 02

Electron microscopy study of magnetic field-induced variant selection during disorder-order transformation in CoPt alloy
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CoPt alloy undergoes disorder-order transformation from a cubic disordered phase (A1) to a tetragonal ordered phase (L1₀) at 1045 K. Because of decrease in crystal symmetry, three ordered variants are formed. A two-step ordering heat-treatment under a magnetic field was performed in order to control formation and selection of preferred variant with its tetragonal c-axis parallel to the applied magnetic field. However, the ordering process and mechanism of variant selection have not been clarified yet. In the present work, transmission electron microscope (TEM) and scanning transmission electron microscope (STEM) observations were carried out to clarify the process of microstructure formation and variant selection in CoPt alloy ordered by the two-step heat-treatment under a magnetic field of 10 T.

After the first step of ordering, ordered domains of about 5 nm in size were observed and fraction of the preferred variant was slightly higher than that of the other two variants. Formation of tweed microstructure along {011} was confirmed at the initial stage of ordering. This structure was probably derived from the periodic alignment of interface between two ordered variants. At the early stage of the second step, numerous micro-twins were formed through tweed microstructure and volume fraction of the preferred variant was increased accompanying with twin rearrangement, while that of other two variants was decreased. Anti-phase boundaries introduced with high density were also rearranged and relaxed together with twin arrangement. After the second step of ordering, the twins were vanished and single variant structure was obtained.

L-MI 03

Microstructure of tetragonal Ni₂B compound solidified from the undercooled meltM. Kolbe¹, S. Binder¹, S. Klein¹, D. M. Herlach¹¹DLR, Institut für Materialphysik im Weltraum, Cologne, Germany

Solidification of an undercooled melt is inherently correlated to dendrites, which are growing with favoured directions into the undercooled melt. The directions originate from the anisotropic nature of both the interfacial energy and atomic attachment kinetics. It arises from the crystal structure at the solid side of the interface. In the case of cubic crystals, dendrites are growing along the $\langle 100 \rangle$ directions. In the present work crystal growth is investigated in the non-cubic intermetallic compound Ni₂B of tetragonal symmetry.

Samples of diameter of 2-6 mm were containerlessly undercooled and solidified by electrostatic levitation and electromagnetic levitation, respectively. The advancement of the solidification front is observable in-situ, as it releases latent heat, which leads to a steep rise in temperature at the front and a light emission. This event is monitored by a high speed video camera. The interpretation of this recording, in order to derive the velocity of solidification is not unambiguous. (i) The fine dendritic structure cannot be resolved by the optics of the camera, (ii) the light emission shows only the surface-near part of the sample. We show that a reliable relation between undercooling of the melt and velocity of the solidification front can be established, if the geometrical features of the observed solidification front can be correlated to the as-solidified microstructure. In the case of Ni₂B the geometrical shape of the envelope of the dendritic network is given by a rectangular prism with an axis length ratio $a = b = 0.85c$. Surprisingly the ratio 0.85 is the inverse of the ratio of the respective crystal axes.

L-MI 04 – withdrawn

L-FU 01

Influence of fabrication conditions on structure, magnetic property and magnetocaloric effect in Mn-based Heusler alloys

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Mn-based Heusler alloys ($A\text{-Mn-}B$, $A = \text{Co, Ni}$; $B = \text{Si, Sn, Sb}$) in forms of ingot and ribbon were prepared by using arc-melting and melt-spinning methods, respectively. Structure-property relationships in these systems (Co-Mn-Si, Ni-Mn-Sn, Ni-Mn-Sb...) of Heusler alloys were systematically investigated and compared. The results showed that structure, magnetic property and giant magnetocaloric effect (GMCE) of the alloys are strongly influenced by fabrication conditions such as quenching rate, annealing temperature, annealing time. The coexistence of ferromagnetic (FM) and antiferromagnetic (AFM) orders can be observed in these systems with appropriate compositions and fabrication conditions. The magnetic phase transitions can be controlled by changing composition and annealing condition of the alloys. GMCEs with large magnitude and wide working temperature range have been obtained on these alloys showing their application potential for magnetic refrigeration technology.

L-FU 02

Production, microstructure, and properties of single crystalline NiMnGa magnetic shape memory alloys

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Magnetic shape memory (MSM) alloys are a relative new class of actuator and sensor materials that change their shape when subjected to a moderate magnetic field. They are typically Heusler type Ni_2MnGa single crystalline materials, which give a 6% elongation in a magnetic field of 0,5 T. These alloys were first studied as potential thermal shape memory materials before the MSM effect was discovered in 1996. The Ni-Mn-Ga alloys show a variety of different martensitic structures which are strongly composition dependant. Their unique magneto-mechanical properties have attracted considerable interest both by research and industry community for applications in actuators, sensors and energy harvesters. Single crystalline Ni_2MnGa alloys have been grown at ETO MAGNETIC GmbH using traditional Bridgman techniques. The crystals had a diameter ranging from 12 to 60 mm, and a length of 120 mm. Various process parameters and compositions have been used during production. The crystals were analysed in their as-cast and heat treated state using optical microscopy. For orientation and single crystallinity EBSD techniques were used. Rectangular sticks were cut and trained to receive single variant state. The sticks were tested as regard to their stroke-force as well as their stroke-field behavior. They demonstrate a reduced twinning stress, increased work output and increased energy efficiency compared to MSM materials produced so far. The homogeneity of the single crystal has been investigated using chemical composition analysis. In addition several MSM sticks cut along the crystal length were investigated as regards their magneto-mechanical properties. It has been proven that using the Bridgman process homogeneous NiMnGa single crystals can be produced.

L-FU 03

Anisotropic stress-strain-behaviour of NiTiP. M. Kadletz¹, W. W. Schmahl¹, M. Hölzel², T. Kassar¹¹LMU, Dept. of Geo- and Environmental Sciences, Munich, Germany²FRM II, Garching n. Munich, Germany

NiTi finds widespread medical and engineering applications of its superelastic and shape memory (SM) properties. The SM effect occurs in the monoclinic B19' NiTi martensite phase and it is based on ferroelastic twin switching in the twin domain microstructure under the action of stress.

Heating to the austenite phase (T(Af) ~ 70°C in our alloy) recovers the original shape and the original stress-free twin-structure is re-accommodated on cooling. Despite its technological importance, the elastic constants of the monoclinic B19' martensite are still unknown, as untwinned single crystals are not available. DFT calculations have provided elastic constants for zero K involving a monoclinic angle of $\gamma \sim 107^\circ$ instead of $\sim 97^\circ$ which is observed experimentally, so room temperature elasticity of monoclinic NiTi is still a matter of debate.

The deformation of a SMA under applied stress is a superposition of a ferroelastic twin-switching and an elastic distortion. To measure and separate both components diffraction techniques are needed. For investigation of anisotropic elastic properties of materials we developed a rotatable loading frame (RLF) for SPODI @ FRM II, where the load axis can be rotated in an Eulerian cradle. We applied uniaxial stress to obtain a sufficient amount of information to constrain the elastic constant tensor using samples of a commercial Ni_{50.29}Ti_{49.71} SM alloy (martensitic at RT). For data evaluation we used MAUD fitting the uniaxial stress necessary to produce the observed peak shifts due to elastic strain using a modified version of the DFT elastic constant tensor of Wagner & Windl (2008). The evaluation of the measurements indicated complex behaviour with changes from constant-strain (Voigt) to constant-stress (Reuss) microstructures in response to increasing applied strain. When macrostrain is initially applied from 0% to 0.4% there is a relaxation of residual stresses in the sample by ferroelastic domain switching. A similar anomaly occurs between 1% and 1.2% macroscopic strain, which corresponds to the onset of the stress-plateau in the macro stress-strain curve due to the twin-switching process.

L-FU 04

Shape recovery and long term behavior of the Ni₁₄Ti₅₁Pd₃₅ high temperature shape memory alloyA. Denguin¹, A. Bachelier-Logq¹, L. Peltier², E. Patoor²¹ONERA, Chatillon, France²ENSAM, Metz, France

Shape memory alloys (SMAs) have the ability to perform both sensing and actuating functions and have consequently been identified as potential materials for various functions within turboengines components. The introduction of this category of materials in aerospace gas turbines however requires SMAs with martensitic transformation (MT) temperature above 250°C, which is well over MT temperatures of commercially available materials. The partial substitution of Ni by Pd in the Ni-Ti system has been reported to increase phase transformation temperatures. In this study, a NiTiPd alloy has been developed for actuation temperatures of about 300°C.

Influence of thermomechanical treatment on shape memory properties (transformation temperatures, strain recovery) has been characterized through tensile or compression tests. Thermal cycling under load has been performed in order to determine the work output. Long term behavior was studied through residual properties determination after heat treatments performed in the temperature range which can be encountered in service. The influence of compositional variations and thermomechanical processing will also be discussed.

L-FU 05

Superelastic and shape memory behaviour in copper-based functional intermetallicsJ. San Juan¹, J. F. Gomez-Cortes¹, I. Lopez-Ferreño¹, G. A. Lopez², M. L. Nó²¹Universidad del Pais Vasco, Fisica Materia Condensada, Bilbao, Spain²Universidad del Pais Vasco, Fisica Aplicada II, Bilbao, Spain

Shape Memory Alloys (SMA) undergo reversible martensitic transformation in response to changes in temperature or applied stress, exhibiting specific properties of superelasticity and shape memory. SMA are the prototype of functional intermetallic materials and in particular the intermetallic Ti-Ni, worldwide known as Nitinol, is being commercially used. Nowadays copper-based (SMA), which are Heusler-type intermetallics, are becoming very attractive as alternative materials to the traditional Ti-Ni. At present there is a high scientific and technological interest to develop SMA properties at small scale, in order to apply SMA as sensors and actuators in MEMS technologies. Recent works [1, 2] show that Cu-Al-Ni SMA exhibit good thermo-mechanical properties of superelasticity and shape memory at micro and nano scale, with some advantage over the Ti-Ni. However the martensitic transformation seems to exhibit a different behaviour at small scale than in bulk materials and size effects on superelasticity were recently reported [2].

The present work is focused on the copper-based SMA with Heusler intermetallic structure, in order to study their properties at small scale. We have micromachined, by Focused Ion Beam technique, some basic devices, micro and nano pillars, that exhibit superelasticity and shape memory effect with displacements of some hundreds nanometers with complete reversibility.

First, we will overview the thermo-mechanical properties of copper-based functional intermetallics at the nano-scale, with special emphasis on size effects. In the second part, new results on superelastic cycling at nano-scale will be presented. We may advance that thousands of superelastic cycles have been obtained in micro and nano pillars, what allow us to anticipate a good reliability of small devices using these intermetallics.

Finally, the above commented size effects will be discussed on the light of the microscopic mechanisms controlling the martensitic transformation at nano scale.

[1] J. San Juan, M.L. Nó, C.A. Schuh. *Advanced Materials* 20 (2008) 272.[2] J. San Juan, M.L. Nó, C.A. Schuh. *Nature Nanotechnology* 4 (2009) 415.

L-FU 06

HAADF-STEM studies of L1₀-type Fe-Pd alloy ordered under magnetic fieldS. Farjami¹, Y. Tanaka¹, M. Mitsuahara¹, M. Itakura¹, M. Nishida¹, T. Fukuda², T. Kakeshita²¹Kyushu University, Department of Engineering Science for Electronics and Materials, Fukuoka, Japan²Osaka University, Department of Materials Science and Engineering, Osaka, Japan

L1₀ ordered intermetallic alloys such as Fe-Pd, Fe-Pt and Co-Pt have been attracting interest in recent times because of their large uniaxial magnetocrystalline anisotropy. The tetragonal ordered phase (L1₀) of these alloys is formed from a cubic disordered phase (A1) through a disorder-order transformation. Usually the ordered phase consists of three lattice corresponding variants with the tetragonal c-axis as the easy axis of magnetization. To clarify the effect of magnetic field on microstructure formation, a two-step ordering heat treatment has been carried out and consequently formation of single variant in ferromagnetic ordered phase of Fe-55 at.% Pd alloy has been revealed [1]. In this study, conventional transmission electron microscopy (CTEM) and high-angle annular dark field scanning transmission electron microscopy (HAADF-STEM) techniques have been applied to investigate the effect of magnetic field on microstructure formation during the two-step ordering process. A HAADF-STEM image after the first step under magnetic field of 10 T shows random distribution of three variants and size of each ordered variant is about 5 nm. Due to the application of magnetic field at this stage, fraction of the preferred variant is slightly higher than that of the other two variants. During the second step of ordering, growth of the preferred variant occurs selectively and single variant is obtained. Based on observation, we will discuss the effect of magnetic field on microstructure formation through the disorder-order transformation.

[1] S. Farjami, T. Fukuda and T. Kakeshita, *Mater. Trans.* 49 (2008) 1970-1974.

L-SN 01

Development of sintering process based on eutectoid decomposition and redox reaction for thermoelectric β -FeSi₂ composite alloy fabrication

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The β -FeSi₂ is one of eco-friendly thermoelectric materials which can be used to convert waste heat into clean electric power at relatively high temperatures around 1073 K. Since β -FeSi₂ is formed by the peritectoid reaction between α -FeSi₂ and ε -FeSi, the formation of β -FeSi₂ requires long term heat treatment depending on α and ε two-phase microstructure. Therefore the development of fabrication process has always been problems to be overcome. We have proposed a method of powder metallurgy based on phase equilibria and several reactions to fabricate a new composite type thermoelectric alloy consisting of β -FeSi₂ and SiO₂ (or Fe and Si oxides mixture). Starting materials are α -FeSi₂ single-phase powder and Fe oxide powder such as Fe₃O₄ and Fe₂O₃.

The idea is that α -FeSi₂ firstly decomposes into β -FeSi₂ and Si by the eutectoid decomposition, then SiO₂ and Fe are formed by the redox reaction between eutectoid Si and Fe oxide, and simultaneously β -FeSi₂ is formed by the solid reaction between Fe and residual eutectoid Si. The formation of eutectoid Si, SiO₂ and β -FeSi₂ during the present process was observed by X-ray diffractometry and scanning transmission electron microscopy (STEM). Chemical composition analyses using STEM were also conducted for the phase identification. Thermoelectric properties of β -FeSi₂ + SiO₂ composite alloys were measured in the temperature range from 300 to 1073 K. Note that Co and Mn were selected as n-type and p-type dopants respectively for intrinsic semiconductor β -FeSi₂. Compared with nearly single-phase β -FeSi₂ sintered alloys (fabricated by us), Electrical power factor, consisting of Seebeck coefficient and electrical resistivity, is not very good. However, values of dimensionless figure of merit ZT of the present composite alloys are comparable to those of β -FeSi₂ alloys since the lattice thermal conductivity can be effectively reduced by dispersed SiO₂ in composite alloys.

L-SN 02

Heat treated microstructure and mechanical properties of high Cr content Nb-Si based alloy

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Niobium-silicide-based alloys have attracted significant attention due to their high melting points, low densities and excellent high-temperature performance. These alloys show great potential for replacing superalloys in the hottest sections of advanced turbine engines. However, their poor oxidation resistance and low ductility severely limit their further development. Previous studies have indicated that the addition of Cr can improve oxidation resistance. Therefore, the development of high-Cr-content NbSi-based alloys is promising. Unfortunately, the room-temperature mechanical properties of high-Cr-content NbSi-based alloys degrade dramatically because Cr usually leads to the formation of large block-like Nb₅Si₃ particles. To overcome this limitation, heat treatment is employed to improve the microstructure.

A high-Cr-content NbSi-based alloy is prepared by rapid directional solidification at a withdrawal rate of 100 mm/min. Heat treatments are carried out at 1425°C for 2, 4, 8, 10, 12h, respectively. Microstructure and mechanical properties of the heat treated alloys are studied. The metastable phase Nb₃Si is decomposed completely to Nb₅Si₃ + Nb₅₅ during heat treatments. Vermicular-like Nb₅Si₃, approximately 20 μ m in length and 5 μ m in width, is uniformly distributed in the Nb₅₅ matrix after heat treatment at 1425°C for 10h. This process optimises the fracture toughness (18.7MPa·m^{1/2}) and tensile strength (890MPa).

L-SN 03

Determination of the ternary eutectic in the Nb-Si-Cr system and its influence on the microstructure formation during directional solidificationF. Gang¹, K. von Klinski-Wetzel¹, M. Heilmaier¹¹Karlsruhe Institute of Technology, Institute for Applied Materials, Karlsruhe, Germany

Niobium silicide based alloys are promising candidate materials to replace nickel-based superalloys in high temperature structural applications, as they offer great potential for appropriate creep and oxidation resistance. Unfortunately, both properties are difficult to be achieved simultaneously in a satisfying manner. Improving the creep properties of a given alloy with adequate oxidation behavior may be obtained by directional solidification (DS) as this processing route can minimize the amount of transversal grain boundaries within the material, hence, leading to enhanced creep lifetimes.

A literature survey reveals that directional solidification was applied to several multinary niobium based alloys [1, 2]. Although the achieved microstructures were aligned perpendicular to the solidification front, the continuity of the phases was limited. In contrast to these multinary, non-eutectic alloys, it is assumed that directional solidification of eutectic alloy compositions, due to their distinct melting point, can result in a more continuous and even finer lamellar microstructure. To verify this hypothesis, the model system Nb-Si-Cr is chosen here, as it possesses, besides reasonable oxidation properties, a (Nb-rich) ternary eutectic. However, literature reveals contradictory information on the exact composition of the ternary eutectic [3, 4].

Therefore, its exact location is critically reviewed with samples of different appropriate alloy compositions produced by levitation melting and examined regarding their formed phases and microstructure using SEM, EDX and XRD measurements. Furthermore, thermal stability of the microstructure is investigated by heat treatments, aiming to obtain an equilibrium-near microstructure formation. The identified composition of the ternary eutectic is subsequently directionally solidified using the vertical float zone process and the resulting microstructure is comparatively assessed with the results from levitation melting.

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L-SN 04

Mechanical properties and oxidation resistance of directionally solidified NiAl-Mo composites at room and high temperaturesL. Hu¹, W. Hu¹, G. Gottstein¹, S. Bogner², A. Bührig-Polaczek²¹Institute for Physical Metallurgy and Metal Physics, RWTH Aachen University, Aachen, Germany²Foundry Institute, RWTH Aachen University, Aachen, Germany

Mo fiber reinforced NiAl *in-situ* composites with a nominal composition Ni-43.8Al-9.5Mo (at.%) were produced by specially controlled directional solidification (DS) using a laboratory-scale Bridgman furnace equipped with a liquid metal cooling (LMC) device. In these composites, single crystalline Mo fibers were formed by eutectic transformation and were aligned parallel to the growth direction of the ingot.

Mechanical properties i.e. the creep resistance at high temperatures (HT) and the fracture toughness at room temperature (RT) of *in-situ* NiAl-Mo composites were characterized by tensile creep (along the growth direction) and flexure (four-point bending, vertical to the growth direction) tests, respectively. The oxidation resistance of NiAl-9Mo composites was examined by cyclic oxidation tests at 1000°C for 1000 hours.

In the current study, a steady-state creep rate of 10^{-6} s⁻¹ at 1100°C under an initial applied tensile stress of 150MPa were measured. The flexure tests sustained a fracture toughness of 14.5 MPa·m^{0.5} at room temperature. Compared to single or polycrystalline NiAl and other NiAl alloys, these properties suggested a considerable improvement. However, the oxidation resistance of as-produced NiAl-9Mo composites is somewhat deteriorated in comparison with NiAl and other NiAl alloys, e.g., IP75 alloy.

The mechanisms for RT toughening, HT strengthening and HT oxidation of *in-situ* NiAl-9Mo composites were discussed based on different models.

L-SN 05

Discrete dislocation dynamics modeling of loading orientation effect on the low stress creep of single crystal Ni base superalloys

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Single crystal Ni base superalloys are used in the blades of hot gas turbines where they operate at elevated temperatures in the creep deformation range. It is known that creep is governed by dislocation glide and climb processes which have been observed in the transmission electron microscopy. Climb process is indeed a diffusion control process that increases exponentially with temperature. In the present study we use discrete dislocation dynamics simulation to study the evolution of dislocations in a typical γ/γ' microstructure of a single crystal superalloy under different loading conditions. We focus on the early stages of creep, where dislocation plasticity is confined to narrow γ channels. A hybrid glide-climb mobility model (S.M. Hafez Haghighat, G. Eggeler, D. Raabe, Acta Mater. 2013) is used to conduct the interaction of dislocations with γ' particle. The influence of the loading direction, namely the [111] and [100], on the creep rate and its resulting microstructure is considered. It appears that at a stress of 200 MPa applied along the [111] direction the creep rate is limited due to the pinning of dislocations by γ' particle. In the [100] loading direction, however, creep strain increases monotonically due to the propagation of dislocations in the channel oriented perpendicular to the loading direction, where a dislocation network may form in agreement with experiments. The dislocation microstructure, contrary to the [111] loading direction, is anisotropic along different γ channels. These results rationalize the previous experimental findings of the effect of loading misorientation on the low stress creep of single crystal Ni base superalloys.

L-SN 06 – withdrawn

P-TA 01

Hot corrosion and interdiffusion behaviour of aluminide coated Ti-40Al-2Cr-2Mo intermetallic alloy at 800°C
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Intermetallic alloys of the Ti-Al system have been developed to have combination of properties suitable for high temperature applications. The applications of γ -TiAl, however, has been reported faced problems as this materials is still relatively brittle at room temperature and prone to hot cracking during casting. Reducing slightly the aluminium content of the alloy to give two phase of α_2 -Ti₃Al and γ -TiAl (simplified as α_2 -Ti₃Al/ γ -TiAl) has been reported give better mechanical properties at room as well as high temperatures, eventhough it affects in reduction of oxidation resistance. To solve this problem two methos are available, *i.e.*, alloying with elements that increase oxidation resistance of α_2 -Ti₃Al/ γ -TiAl, such as chromium, and applying aluminide coatings. Pack aluminizing has been widely applied to develop aluminide coatings for high temperature alloys. During applications at high temperatures, the coating can degrade due to the interaction between the coated system and the environment especially that contains halide salt of Na₂SO₄ and NaCl. In addition, at high temperatures the alloy/coating system is metastable and as consequence, the coatings could degrade due to the interdiffusion between coating and substrate alloy.

This study investigated the hot corrosion and interdiffusion between coatings and substrate of the coated samples in salt solution of 90% Na₂SO₄ + 10% NaCl at 800°C for various times. A pack containing 20 wt.% Al, 2 wt.% NH₄Cl, and 78 wt.% Al₂O₃ was used to develop coatings. The pack aluminizing was carried out at 850°C for various times of 11, 15, 25 and 40 hours. The experiment results showed that the hot corrosion of coated samples indicates pitting types corrosion. The corrosion mechanisms have been studied primarily based on corrosion products observed using electron microscope and x-ray diffraction analysis. In addition, the interdiffusion between coating and substrate alloy at high temperatures causes degradation of coating (TiAl₃ layer) with time of exposure resulting in the interdiffusion zone of TiAl₂ layer enlargement. The interdiffusion mechanism of the coated systems has been analyzed and the growth of this TiAl₂ layer at 800°C generally follows kinetics equation of $x = kt^{1/n} + C$.

P-TA 02

Combination of CVD coatings and halogen effect to prevent high-temperature embrittlement in titanium aluminides
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Intermetallic TiAl-based alloys represent an important class of high temperature structural materials providing a unique set of physical and mechanical properties that can lead to substantial payoffs in industrial applications, e. g. for turbine blades or turbolader wheels. At less than half the weight of nickel-base alloys and with excellent high temperature properties, their potential for these applications is considerable. Because of their insufficient oxidation resistance and embrittlement at higher temperatures (> 700°C) the surface of titanium aluminide alloys has to be modified in order to effectively replace the heavier nickel-base alloys currently in use.

To overcome such shortcomings, a combination of Al enrichment plus additional fluorine treatment is used to protect the alloy against the rapid diffusion of oxygen at high temperatures which leads to structural damage of the material. Indeed, Al-rich coatings plus fluorine are expected to promote the formation of a protective alumina layer at high temperatures which not only protects the alloy from oxidation, but also impedes embrittlement at high temperatures. To achieve this aim advanced coatings are produced either by pack cementation or by metal-organic chemical vapor deposition and additionally fluorine is deposited by spraying with a fluorine polymer. The coating effect on the oxidation behavior of the titanium aluminide alloys is examined under isothermal and thermocyclic conditions (800-1000°C) in air and the mechanical properties of coated and uncoated samples after atmospheric exposure are compared using 4-point bend tests to investigate the room temperature strength and ductility.

P-TA 03

Environmental protection of a β -stabilized γ -TiAl alloy by a combination of the halogen effect with thermal barrier coatings

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The halogen effect, in particular when fluorine is applied, offers an excellent oxidation protection for light-weight intermetallic titanium aluminides at temperatures up to 1050°C by formation of a protective α -alumina layer. This thermally grown layer provides an optimal basis for the application of a ceramic thermal barrier coating (TBC) without the necessity of additional Al-enrichment as bond coat, offering an innovative method to install TBCs on γ -TiAl alloys. A novel β -stabilized γ -TiAl alloy, namely TNM-B1, was chosen for the study. Different fluorination methods in combination with zirconia topcoats produced by electron-beam physical vapour deposition at 900°C and 1000°C were tested. When thermally cycled at 900°C in air, thermal barrier coatings of yttria partially stabilized zirconia deposited on γ -TiAl samples treated with different fluorination methods exhibited lifetimes exceeding the maximum exposure time period of 1000 1-h cycles. It was found that samples fluorinated with a fluorocarbon polymer is most promising at temperatures of as high as 1000°C under these conditions. Further thermocyclic tests at 900°C under more aggressive atmospheres containing water vapor also confirmed slow oxide growth rates and a good adherence of the TBCs. The study demonstrates that environmental protection on a β -stabilized γ -TiAl alloy can be achieved applying the halogen effect in combination with a TBC-system.

P-TA 04

Phases evolving in Ti-Al-Cr-Zr oxidation protective coatings on γ -TiAl alloys during thermal cycling at 1000°C

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For applications at service temperatures above 800°C, γ -TiAl based alloys most likely need protective layers to prevent oxidation. As reported in a previous paper, coatings of composition Ti-49Al-34Cr-4Zr: Y possess excellent oxidation resistance at 1000°C in air. We used X-ray diffraction, SEM and TEM to characterize the evolving phases in such a coating deposited on a Ti-45Al-8Nb alloy. After 1000 cycles of 1 hour at 1000°C, a threefold layer structure develops. Underneath a corundum scale, a discontinuous layer of a C14 type Laves phase (Ti-35.9Al-27.8Cr-4.4Zr-10.3Nb) is formed, requiring substantial outward diffusion of Nb from the base alloy. Between this phase and the TiAl substrate, a layer of a phase of unknown structure is present (U-phase). The outer part of this layer exhibits pores as well precipitates rich in Zr, Y and O, in contrast to its inner part. The composition of the U-phase as determined by EDX is Ti-30.2Al-19.5Cr-2.7Zr-4.7Nb. The SAED diffraction pattern of this phase can be indexed utilizing an orthorhombic body centered unit cell ($a=3228$ pm, $b=520$ pm, $c=1340$ pm). Additional ordering is indicated by infinite rods of intensity in reciprocal space running parallel to the a-axis. In sections perpendicular to the a-axis the resulting sharp spots are indexable by doubling the b-axis virtually conserving the body centered Bravais lattice. The substrate below the coating is enriched in Cr and slightly depleted in Nb. The prevailing phase present in this region is γ -TiAl, albeit small patches of the α_2 -Ti₃Al phase are also observed adjacent to the U-phase.

P-TA 05

Effect of tungsten on the oxidation behavior of orthorhombic titanium aluminideD.-G. Lee¹, J. H. Shon², Y. Lee¹, M.-H. Oh³, S. Emura⁴¹KIMS, Changwon, South Korea²POMIA, Pohang, South Korea³Kumoh National Institute of Technology, Gumi, South Korea⁴NIMS, Tsukuba, Japan

Ti-Al compounds and alloys has been expected to be one of the high temperature materials as well as Ni superalloys, but also be strictly restrained to be applied in special fields. In order to make it possible to be used in various fields, orthorhombic titanium aluminides has been considered one of the promising high temperature materials. For some orthorhombic alloys, a better fracture toughness, higher ductility and specific yield, lower coefficient of thermal expansion than for γ TiAl based alloys as well as for α_2 Ti₃Al alloys has been reported. Compositions of the test alloys in this study are Ti-22Al-27Nb (at.%) and Ti-22Al-20Nb-2W (at.%). Tungsten element is very strong β stabilizer and to improve corrosion resistance and creep. Two orthorhombic alloys were investigated with respect to their oxidation resistance at 800°C in air using TGA (thermo gravimetric analysis). The oxidation kinetics are discussed with regard to the influence of the alloying elements. The parabolic rate constant of three orthorhombic alloys are discussed in consideration of the literature data. Oxidation curves indicated that tungsten-involved Ti-22Al-20Nb-2W has good oxidation resistance and less scale spallation, but in all cases, the mixed oxide scale consists of TiO₂, Al₂O₃.

P-TA 06

Environmental protection of γ -TiAl alloys – Influence of coatings on oxidation resistance and fatigue behaviorN. Laska¹, R. Braun¹¹German Aerospace Center, Institute of Materials Research, Cologne, Germany

Intermetallic titanium aluminide alloys are mature light-weight materials for high temperature applications substituting Ni-based superalloys experiencing temperatures up to 750°C. To raise their service temperatures beyond this limit, protective coatings are required; additionally, thermal barrier coatings (TBCs) can be used with internally cooled components as widely applied on Ni-based superalloys.

In present work, the oxidation behaviour of the γ -TiAl based alloy Ti-45Al-8Nb (at.%) with oxidation protective coatings was studied performing cyclic oxidation tests at 850°C in air up to 2000 cycles of 1 h dwell time at high temperature. The 10 μ m thick Ti-Al-Cr-Y and Ti-Al-Cr-Zr coatings were deposited using magnetron sputtering. Furthermore, the lifetime of a TBC system on the γ -TiAl alloy was determined conducting thermal cycling tests at 850°C. The TBC system consisted of a Ti-Al-Cr-Zr bond coat and an yttria partially stabilized zirconia (YSZ) top coat, the latter produced by electron-beam physical vapour deposition. Both intermetallic layers provided oxidation protection to the γ -TiAl alloy. Particularly, the Ti-Al-Cr-Y coating exhibited excellent oxidation resistance associated with the reactive element effect of the yttrium addition. Lifetimes exceeding 2000 cycles were determined for the TBC system thermally cycled at 850°C. The fatigue behaviour of the Ti-45Al-8Nb alloy with protective coatings was investigated after isothermal exposure to air at 850°C for 300 h. Using tension specimens, fatigue tests were carried out at room temperature applying an R-ratio of R = -1 up to 5 million cycles. Both oxidation protective coatings reduced the fatigue strength in comparison to the bare substrate material. The degradation was slightly more severe for γ -TiAl samples with Ti-Al-Cr-Zr bond coat and YSZ topcoat.

P-TA 07

Fatigue properties of high Nb γ -TiAl obtained by additive manufacturing with Electron Beam Melting (EBM)
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γ titanium aluminide based alloys have become an important candidate for high temperature structural applications in the aircraft industry to replace current nickel-based superalloys as the material of choice for low-pressure turbine blades. Although such materials appears very promising for the turbine engine industry, optimizing the performance improvements requires more advanced approaches to understand and address the specific fatigue properties of these materials to assure adequate reliability in structural applications. Additionally, it's now becoming more and more important to explore the possibilities offered by novel process routes like additive manufacturing methods, with the aim of maximising the benefit given by higher content of alloying elements, with special interest in the effect of refractory elements. The fatigue properties of a high Nb containing alloy obtained by electron beam melting (EBM) with a additive manufacturing process have been examined by conducting high cycle fatigue tests performed at different loading ratios, both at room temperature and at high temperatures, comparable to those experienced by the components during service. Fatigue crack growth threshold tests were also performed, with the purpose of comparing the fatigue properties of high Nb containing alloys with those of second-generation γ -TiAl alloys. Finally, specimens with artificial defects have been used to conduct fatigue tests with the objective of studying the growth behavior of small cracks, in view of modeling the fatigue threshold behavior and quantitatively assessing the mechanical performance advantage of the high Nb γ -TiAl over the second-generation TiAl intermetallics.

P-TA 08

Study of the failure behaviour of a titanium aluminide alloy during hot formability
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Intermetallic γ -TiAl based alloys of the TNM™ alloy family are innovative high temperature materials with high specific mechanical properties at high temperatures. On the other hand, ductile failure can limit the hot formability of γ -TiAl components. In this work, the analysis of the damage behaviour is performed by means of failure modeling coupled to finite element methods (FEM) simulations of the hot deformation of compression samples with the package DEFORM-2D. Various ductile macromechanical damage models are implemented, such as Cockcroft & Latham, normalized Cockcroft & Latham, Brozzo, Freudenthal and Ayada. The models are experimentally validated with hot deformation behaviour of this TNM™ material by compression tests using a Gleeble®3800 thermomechanical simulator. Thus, material dependent parameters such as flow curves are obtained from the compression tests and implemented in DEFORM-2D. Additionally, metallography is performed on heat-treated, deformed and fast cooled specimens to acquire knowledge of the damage developed during hot deformation. Microstructural changes including shear bands, pores and cracks are investigated after different hot deformation conditions by light optical- and scanning electron microscopy.

P-TA 09

Microstructure – Property relationship in a near net shape forged γ -TiAl based alloyA. Gaitzenauer¹, H. Clemens¹, P. Voigt², R. Hempel², S. Mayer¹¹Montanuniversität Leoben, Physical Metallurgy and Materials Testing, Leoben, Austria²Titanium Solutions GmbH, Bremen, Germany

Excellent mechanical properties, including low density, high specific strength as well as good creep properties up to high temperatures are the reason why intermetallic TiAl alloys have found applications in aero- and automotive engines. The investigated TiAl alloy with a nominal composition of Ti-43.5Al-4Nb-1Mo-0.1B (at.%) is a complex multi-phase γ -TiAl based alloy consisting of lamellar α_2 -Ti₃Al/ γ -TiAl colonies and a low volume fraction of β_0 -TiAl located at the colony boundaries. In this paper a novel processing route, which is a combination of a one-shot hot-forming step and a subsequent controlled cooling treatment is presented, leading to improved mechanical properties like an increase in hardness of about 400 HV compared to a value of 350 HV of the hot-isostatically pressed pre-material.

Using scanning and transmission electron microscopy the influence of the microstructural parameters on the properties was determined. Thereby, the mean lamellae spacing of about 40 nm leads to an increase in hardness and strength according to a Hall-Petch relationship. Tensile and creep tests show the capability of the alloy for use as turbocharger wheels in Diesel engines.

P-TA 10

Microstructure and tensile mechanical properties of boron-containing β -solidifying γ titanium aluminide alloysV. Imayev¹, R. Imayev¹, T. Nazarova¹, R. Gaisin¹, E. Gaisina¹¹Institute for Metals Superplasticity Problems of Russian Academy of Sciences, Ufa, Russia

The work summarizes the results concerning β -solidifying γ titanium aluminide alloys in the composition range of Ti-(43-45)Al-(3-7)(Nb,Mo,Cr,B) (at.%). The effect of heat treatments including the β heat treatment on formation of relatively fine and homogeneous microstructures was studied. The microstructure refinement in such alloys is associated with borides, which act as nucleation sites for new α grains in the course of the solid-state β - α phase transformation. Air and furnace cooling applied after annealing in the β phase field resulted in refinement of the microstructures. However furnace cooling led to more equilibrium microstructures with lamellar colonies having nearly random lamellae orientations and more equiaxed forms in comparison with those obtained after air cooling. Tensile mechanical properties of the alloys were investigated in the cast and heat treated conditions. On this basis, optimized heat treatments are proposed for the as-cast β -solidifying alloys. The alloys under study have improved hot workability. It was shown that two-stage forging followed by heat treatment provided the duplex microstructure, which led to appreciably higher ductility/strength properties in comparison with those corresponding to the refined lamellar structure. The obtained results are discussed in terms of relationship between microstructure and mechanical properties.

P-TA 11

Solid state diffusion bonded Ti/Al-6081 alloy based multilayer compositesY. Fouad¹, B. Rabeeh¹¹German University in Cairo, Engineering materials, Cairo, Egypt

The application of hot uniaxial pressing has been carried out in the temperature range of 450-600°C for 0.5, 1 and 2 hour at uniaxial pressure of 50 MPa in vacuum. Ti/Al 6081/Ti foil-foil layup are hot pressed for a lamellar composite structure symmetrically arranged. Inter diffusion bonding between commercially pure titanium and aluminum 6081 is established. The microstructure of the diffusion zone has been scanning electron microscopy (SEM). The inter diffusion of the diffusing species across the interface has been evaluated by energy dispersive X-ray spectroscopy EDX. The reaction products formed at the interface have been identified and analyzed. It has been observed that the diffusion zone is dominated by the presence of the low melting depressant elements in Al-alloy that segregate into Ti side and induce the solid solution of β -Ti close to the titanium. Synergetic effect of alloying elements segregation along with its diffusion mechanisms induce me-

chanical bond along interface. It has been observed that the interfacial bond strength (~ 200 MPa) is highest for the couple processed at 575°C and this value decreases with rise in joining temperature. The variation of strength of the transition joints is co-related with the microstructural characteristics of the diffusion zone. The application of Al 6081 interlayer bonding to pure titanium introduced with different micro constituents. The control of interphase is dominant for composite processing.

P-TA 12

Cyclic plasticity and strain localization in cast γ -TiAl-2Nb alloy

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Cyclic strain localization and observation of surface relief in nearly lamellar cast γ -TiAl based alloy Ti-48Al-2Nb-2Cr-0.82B (all in at.%) cycled in strain control were experimentally studied. Hysteresis loop area, the cyclic hardening curves, the cyclic stress-strain curve and V_H parameter were measured. Surface relief of cyclically strained specimens was observed using scanning electron microscopy. The variations of the V_H parameter indicate the cyclic slip localization. The localization has a pronounced effect on surface relief formation namely extrusions. Persistent slip markings (PSMs) are formed on the surface along interlamellar interfaces γ/α_2 and γ/γ . These locations were nuclei for fatigue cracks in the interior of grains. The surface FIB prepared lamella from PSMs showed intrusions along many interfaces γ/γ . Experimental results concerning the loop shape parameter and surface relief are used to discuss the role of cyclic slip localization on the cyclic stress-strain response and fatigue crack initiation. Cyclic strain controlled multiple step tests in air have been performed on cylindrical specimens at 23 and 750°C . The cyclic plasticity describe by the internal and effective cyclic stress components which were derived from the hysteresis loops analyzed according to the statistical theory of hysteresis loop. Cyclic response at both temperatures was compared and discussed in relation to changes of internal and effective stress components and dislocation modes in g phase observed and referred in the literature.

P-PM 01

Synthesis and characterization of polymer nanocomposites containing Fe-40 at.% Si powder particles prepared by high energy ball millingL. Faghi¹, S. Triaa¹, M. Azzaz¹, F. Siahmed¹¹ University of Sciences and Technology HOUARI BOUMEDIENE, Algiers, Algeria

Fe-Si alloys are widely used as transformer magnets and magnetic cores because of their excellent soft magnetic properties. Fe₆₀Si₄₀ powders have milled in a high energy planetary ball mill (Retsch PM400) under argon atmosphere at different time of milling. The metal powders obtained have average diameter d₅₀ of 2.5 to 6 μm. The introduction of Si into Fe can result in a decrease of magnetic anisotropy (therefore leading to decrease of coercivity).

The nanocomposite magnetic cores were made from the Fe₆₀Si₄₀ powder obtained by high energy ball milling for different milling time. The powders particles were mixed with unsaturated polyester (UP) to obtain toroidal cores. Polymerization process was made under magnetic field H= 500 A/m, and ensured a preferential orientation of a powder particles. Influences of the metallic powder fraction on soft magnetic properties as well as thermal increase under isothermal conditions were investigated along with the possibility to control these properties with the size and amount of powder fraction. It was also found that the soft magnetic properties of the polymer composites can be controlled in a wide range and depends on mass fraction of the metallic powder Fe₆₀Si₄₀ in the composite, on shape and size of the powder particles and their orientation in composite.

P-PM 02

Microstructural characterization of metal-intermetallic functionally graded materials fabricated by using Laser Engineered Net Shaping (LENS)M. Ziętała¹, T. Durejko¹, J. Bystrzycki¹¹Military University of Technology, Department of Advanced Materials and Technology, Warsaw, Poland

Functionally Graded Materials (FGMs) belong to a class of advanced materials characterized by a variation in properties as the dimension varies. The overall properties of FGMs are unique and different from any of an individual material that it forms. Among a variety of different intermetallics that can be used in heterogeneous metal-intermetallic materials, Fe₃Al-based intermetallics are of special interest as possible replacements for various types of stainless steel because of their outstanding corrosion resistance at high temperature. The metal-intermetallic FGMs characterized by continuous and smooth variations in composition, which results in a gradual transition of the properties from the intermetallic to the steel, may offer the unique benefits of excellent oxidation and sulfidation as well as wear resistance as compared to stainless steel. This work presents our recently obtained original results on the intermetallic-stainless steel FGMs fabricated by the LENS technology. LENS is the leading additive manufacturing technology for the production of high-performance metal parts. The microstructure and mechanical properties of the intermetallic-stainless steel FGMs were investigated. The LENS system utilizing a high-power laser together with the iron aluminide and stainless steel powders was used to build fully dense functionally graded blocks and tubes with continuous gradient. These parts with continuous gradient were deposited layer by layer under the control of special software that monitors a variety of process parameters to ensure geometric and mechanical integrity as well as continuous gradient of both components. The microstructural and phase analysis were carried out by SEM, XRD and EBSD. Chemical homogeneity after laser deposition was analyzed using an EDS coupled with SEM. The internal integrity of builds was investigated by using X-ray tomography. Based upon the obtained chemical and phase compositions combined with microstructure, their effect on the hardness behavior of the functionally graded materials will be shown and discussed.

P-PM 03

Intermetallic matrix of multicomponent high entropy alloys synthesized by Laser Engineered Net Shaping (LENS) I. Kuncce¹, M. Polanski¹, J. Bystrzycki¹

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Two multicomponent high entropy alloys such as ZrTiVCrFeNi and Zr-free TiVCrFeNi were directly synthesized from elemental powders by using Laser Engineered Net Shaping (LENS). The crystal structure, chemical composition and microstructure of as-fabricated and heat-treated alloys were investigated. Both alloys are considered to be a material for hydrogen storage. The phase analysis was performed with X-ray diffraction. Microstructural characterization was carried out with a Scanning Electron Microscope (SEM) and a chemical analysis by using Energy Dispersive Spectroscopy (EDS). The ZrTiVCrFeNi alloy after synthesis and annealing exhibits a two-phase structure, i.e., there is a dominant C14 Laves phase matrix with a minor amount of the hexagonal close packed (A3) solid solution. The TiVCrFeNi alloy after synthesis exhibits a three-phase structure which is also stable during additional heat treatment. This alloy possesses a dominant C14 Laves phase matrix and a minor cubic face centered and hexagonal solid solutions. Although the negative entropy of mixing in multicomponent equiatomic alloys as well as rapid cooling ($\sim 10^3$ Ks⁻¹) should prevent the formation of intermetallic compounds, the obtained results have shown that differences of the elements' atomic size might have the strongest influence on the intermetallic phase formation.

P-PM 04

Synthesis of intermetallics by using Laser Engineered Net Shaping

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The Laser Engineered Net Shaping (LENS) is a rapid additive manufacturing technology that allows in a short time to produce the final part. The LENS technique is especially recommended for producing of components from stainless steels, Ni-based alloys, Ti-based alloys, refractory metals and intermetallics that are difficult or impossible to produce through conventional metal forming techniques. Since the LENS technique uses a powder feedstock, it allows depositing a blend of elemental powders and *in-situ* alloys creation. The present work presents a possibility of synthesis of different intermetallic compounds such as TiFe, Ti(Fe,Ni), and ternary Fe-Al-Ti Laves and/or Heusler phase from a feedstock comprising elemental powders using the LENS process. The LENS MR7 system with the maximum laser power of 500 W and 4 powder feeders was used to fabricate binary and ternary intermetallic samples with different chemical compositions. The microstructural and phase analysis were carried out by a light optical microscopy, scanning electron microscopy, X-ray diffraction analysis and electron backscattering diffraction. Chemical compositions after laser deposition and additional heat treatment were analyzed using an energy dispersive X-ray spectrometer coupled with SEM and a wavelength dispersive X-ray fluorescence spectrometer. The obtained results show that the binary FeTi and ternary Ti(Fe,Ni) and Fe-Al-Ti intermetallic alloys can be synthesized by using the LENS technique from elemental powder mixtures.

P-PM 05

Mechanically alloyed FeAl intermetallic matrix composites reinforced with Ni₈₀Nb₂₀Zr₂₀ amorphous particles

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In the present study, both Ni₈₀Nb₂₀Zr₂₀ amorphous alloy and FeAl intermetallic compound powders were synthesized by using a mechanical alloying technique. The Ni₈₀Nb₂₀Zr₂₀/FeAl intermetallic matrix composite powders with 10~50 wt.% amorphous Ni₈₀Nb₂₀Zr₂₀ alloy powders were prepared by mixing the corresponding amorphous and FeAl powders. The Ni₈₀Nb₂₀Zr₂₀/FeAl intermetallic matrix composite powders were then consolidated into bulk discs spark plasma sintering (SPS) methods. The DSC result shows that the thermal stability of powders and bulk samples were affected by the milling time. The Vicker's microhardness of bulk samples can be promoted by increasing the amount of Ni₈₀Nb₂₀Zr₂₀, milling time and SPS time. For 45 min. ball-milled Ni₈₀Nb₂₀Zr₂₀/FeAl intermetallic matrix composite powders with 50 wt.% Ni₈₀Nb₂₀Zr₂₀, a significant increase in hardness ($\sim 49.6\%$) was achieved for Ni₈₀Nb₂₀Zr₂₀/FeAl intermetallic matrix composite disc. Based on microhardness results, the corrosion behavior of 40 wt.% Ni₈₀Nb₂₀Zr₂₀/FeAl intermetallic matrix composite discs in four different corrosive media was studied using the potentiodynamic method. The resultant polarization curves indicated the Ni₈₀Nb₂₀Zr₂₀/FeAl intermetallic matrix composite has poor corrosion resistance either in strong acid or strong alkaline solutions. However, it is noted the alloy exhibits the best corrosion resistance in 3.5 wt.% NaCl.

P-FA 01

Micro-abrasive wear behavior of iron aluminidesR. Câmara Cozza¹, C. G. Schön²¹CEETEPS, FATEC-Mauá, Mauá, Brazil²Escola Politécnica da Universidade de São Paulo, Metallurgical and Materials Engineering, São Paulo, Brazil

The rotative ball micro-scale abrasive wear test has gained large acceptance in universities and research centers, being widely used in studies on the micro-abrasive wear of materials. Two wear modes are usually identified in this type of test: "rolling abrasion", which originates from abrasive particles rolling over the surface of the tested specimen, and "grooving abrasion", which is observed when the abrasive particles slide over the surface. The wear mode has a significant effect on the overall behaviour of a tribological system. The present work investigates micro-abrasive wear behaviour of two carbide-reinforced iron aluminide alloys, seeking for a relationship between wear volume, friction coefficient and test temperature in ball-cratering wear testing. Experiments were conducted with an AISI 52100 steel ball and abrasive slurries prepared with black silicon carbide particles and glycerin. The abrasive slurry was continuously agitated and manually fed to the specimen-ball contact, with the help of a dropper, at a rate of one drop every 20 s. Different values of normal force, sliding distance, abrasive slurry concentration and temperature were selected for the tests. Tangential and normal forces were monitored throughout the tests and their ratio was calculated to provide an indication of the friction coefficient acting in the tribological system "*ball – abrasive particles – specimen*". The results have shown that: 1) temperature plays an important role in the selection of micro-abrasive wear modes; 2) detailed analysis conducted by SEM has indicated the occurrence of micro-rolling abrasion at room temperature; 3) with increasing temperatures there is a transition towards predominance of "grooving abrasion", decreasing wear volume and increasing the friction coefficient; 4) the predominance of "grooving" or "rolling" abrasion is the main factor influencing wear volume and friction coefficient.

P-FA 02

Investigation of fracture in Fe₃Al-based alloys with vanadium and carbon additions by small punch testF. Dobeš¹, P. Dymáček^{1,2}¹Institute of Physics of Materials AS CR, Brno, Czech Republic²CEITEC – IPM, Brno, Czech Republic

Creep resistance of iron-aluminium-based can be improved by introducing second-phase particles, e. g., by additions of carbon and carbide-forming elements. The presence of carbides may negatively influence the ductility of alloys. In the present study, the effect of vanadium and carbon additions on ductility and fracture of Fe₃Al-type alloy was investigated. The constant-deflection mode of the small punch test was used to compare fracture properties and their temperature dependence in the alloys with different amounts of carbon. Three main quantities were evaluated: maximum force, fracture deflection and fracture energy. Fractographic observations of ruptured specimens were performed with scanning electron microscope. Temperature dependence of small punch test quantities was related to the microscopic image of the fracture. Equivalent fracture strain was evaluated from both small punch test data and microscopic observation of fractured specimens.

The relation between equivalent fracture strain and small punch fracture energy was examined. Fracture toughness was calculated from the equivalent fracture strain using previously proposed equations. Temperature dependence of calculated fracture toughness supports the conclusion that the ductility of investigated alloys is not deteriorated by the presence of carbides.

P-FA 03

Microstructure and mechanical properties of Fe-Al-Ti-B-based alloys with addition of Mo and WX. Li¹, P. Prokopčáková², M. Palm¹¹Max-Planck-Institut für Eisenforschung GmbH, Düsseldorf, Germany²Technical University of Liberec, Dept. of Material Science, Liberec, Czech Republic

Fe₃Al-based alloys are considered as promising for application as structural materials at elevated temperatures and in hostile environments. Previous studies have shown that additions of B and Ti lead to the formation of fine TiB₂ precipitates, which inhibit grain growth and improve ductility and strength of Fe-Al alloys. W, as the strongest solid solution hardener, improves the strength of these alloys and Mo increases the wet corrosion resistance of Fe-Al alloys.

Four quinary Fe-26Al-0.5Ti-1B-X (X= Mo, W) alloys were investigated with respect to their microstructures and mechanical behaviour. Alloys were produced by vacuum induction melting under argon and one part of each alloy was homogenised at 1000°C for 100 h. Structural characterisation was carried out by light optical microscopy, scanning electron microscopy, electron probe microanalysis, X-ray diffraction and differential thermal analysis. The mechanical properties were determined by microhardness measurements, compression tests from room temperature to 1000°C, four-point bending tests and creep tests at 650°C.

The investigated alloys show a Fe₂Al matrix and – instead of TiB₂ – coarse complex borides, which are unevenly distributed along the grain boundaries. The critical temperatures T_c^{D03-B2} and T_c^{B2-A2} increase due to the addition of Mo (+Ti) and W (+Ti). The temperatures of the maximum of the yield stress anomaly (YSA) of the investigated alloys with additions of Mo and W are shifted to lower temperatures. Hence, this is further proof that there is no relation between the YSA and T_c^{D03-B2} .

No marked brittle-to-ductile transition temperature is observed for all four investigated alloys. All investigated alloys show a better creep resistance than binary Fe-26Al and other Fe₃Al-based alloys strengthened by borides or carbides. The alloy with 4.2 at.% Mo shows the best creep resistance due to the presence of the D0₃ structure at 650°C, instead of the B2 structure in the other investigated alloys.

P-FA 04

Precipitation and transformation kinetics in Fe-Al-Ta alloysM. Palm¹, P. Prokopčáková²¹MPI für Eisenforschung GmbH, Düsseldorf, Germany²Technical University of Liberec, Dept. of Material Science, Liberec, Czech Republic

Fe-Al-Ta alloys with about 25 at.% Al have a high strength and creep resistance up to 800°C. The Fe-Al matrix is strengthened by nanometre-sized coherent precipitates of the L2₁ ordered Heusler phase Fe₂TaAl. Though this strengthening mechanism is known for some time, the stability of the Heusler phase in dependence of composition, time and in relation to the Laves phase (Fe,Al)₂Ta are not known.

Therefore, a systematic study of the time-temperature-transformation (TTT) behaviour of the Heusler phase has been conducted.

Several alloys with Al contents between 23-31 at.% and 1.5-2.2 at.% Ta have been produced by vacuum induction melting. Alloys in the as-cast condition and after heat treatment between 600 and 750°C for 1, 10, 100 and 1000 h have been analysed by metallography, scanning electron microscopy, electron back scatter diffraction, electron probe microanalysis and X-ray diffraction. Systematic changes in the microstructure are observed and these and their effect on the creep behaviour will be discussed.

P-FA 05

Oxidation and parabolic rate constants of Fe-Al and Fe-Al-X alloysM. Palm¹¹MPI für Eisenforschung GmbH, Düsseldorf, Germany

Long term oxidation in synthetic air has been carried out on Fe-Al alloys with Al contents between 5 and 40 at.% Al in order to determine the parabolic rate constants for the growth of stable Al₂O₃ scales at 700 and 900°C.

Five binary Fe-Al alloys were produced by induction melting under argon. Samples cut by electrical discharge machining were ground to 1200 grit surface finish. Thermogravimetric analysis was carried out using a Setaram SETSYS 16/18 thermobalance with continuous recording of the mass gains. The oxidation behaviour was tested in synthetic air for about 1000 h.

From these experiments the duration of the initial metastable oxidation, parabolic rate constants for stable oxidation and the critical Al content necessary for the formation of Al₂O₃ have been determined. The results are compared with those obtained for various Fe-Al-X alloys.

P-FA 06

Interface evolution in an ion beam sputtered Fe/Al multilayer after annealing – influence on magnetic propertiesR. K. Brajpuriya¹, A. Vyas¹, T. Shripathi², O. H. Seeck³, N. Lakshmi⁴¹AMITY University Haryana, Department of Physics, Amity School of Applied Sciences, Pachgaon, Manesar (Gurgaon), India²UGC-DAE CSR, University Campus, Indore, India³HASYLAB am DESY, Hamburg, Germany⁴Mohanlal Shukhadia University, Department of Physics, Udaipur, India

Structural and magnetic properties of ion beam sputtered Fe/Al multilayer samples (MLS), with an overall atomic concentration ratio of Fe:Al=3:1, have been studied as a function of annealing temperature using XRD, XRR, TEM, and VSM. The structural studies show the formation of thin intermixed FeAl layer at the interface during deposition due to the diffusion of Al in to Fe layers, the nucleation and precipitation of disordered FeAl layer, and its subsequent growth to Fe₃Al at higher temperature. The results were also supported by TEM measurements. Magnetization decreases with increase in temperature due to increase in anti-ferromagnetic interlayer coupling and formation of various FeAl phases at the interfaces. The Curie temperature (T_c) is found to be 561°C and is much less than that of bulk-Fe.

P-SN 01

Features of phase composition of intermetallic alloys based on Ni₃AlE. Arginbaeva¹, O. Bazyleva¹¹FSUE "VIAM", Moscow, Russia

For new generation of aviation gas-turbine engines (GTE) an actual task is development of new heat resisting constructional materials with working temperatures up to 1250°C, low density and cost. Heat resisting high-temperature alloys based on Ni₃Al differ from superalloys by economic chemical composition, lowered density (~8000 kg/m³) and high resistance to oxidation. Increase of high-temperature strength of intermetallic alloys based on Ni₃Al is provided by alloying with elements which are dissolved both in γ, and in γ'-phases, changing their parameters of lattice, and also microalloying rare-earth elements, which have considerable difference in nuclear sizes in comparison with aluminum and nickel. Features of alloying and the main properties of intermetallic VKNA/VIN alloys developed by Federal State Unitary Enterprise VIAM are presented. In comparison with applied superalloys, alloys of similar appointment, VKNA/VIN alloys provide increase of working temperature of parts of hot path of GTE on 50-100°C, decrease of cost and labor inputs of production of engines for 15-20%.

It is important to carry out an assessment of condition of structure and phase structure of alloy after tests for hot-resistance for forecasting of stability, behavior of material in use, and also for further improvement of chemical composition. The intermetallic alloys of VKNA-25 and VIN2 containing Re were chosen as a material of research. Researches of samples of alloys after tests for tensile and hot resistance were made.

By the metal physical analysis it is established, in a cast condition alloys have balanced phase and chemical composition, TCP are absent. Dendritic liquation caused by the nature of crystallization of intermetallic alloys, leads to that there is allocated phase with high content of aluminum in interdendritic spaces, the phase is nonhomogeneous and mixed with secondary γ'- phase.

The microstructure, phase structure and mechanical properties of alloys in a cast condition and after heat treatment were analysed. It was showed by SEM of samples of intermetallic alloys after tests, it wasn't revealed considerable changes of phase structure thanks to thermally stable matrix of intermetallic alloys. It was showed that heat treatment leads to decrease of dendritic liquation, dissolution of phase with high content of aluminum and to additional allocation of intermetallic phases. These structure changes results in long-rupture strength of view intermetallic alloys at temperatures of 1100-1200°C.

P-SN 02

Formation of the metastable phase compositions of Ni₇₅(Al,V)₂₅ and Ni₇₅(Al,V,Y)₂₅ (Y: Ti, Zr) alloys prepared by the CCLM and subsequently intensively deformed by the HPTT. Czepepe¹, Z. Świątek¹, A. Sypień¹, G. Garzeł¹, A. Wierzbicka-Miernik¹, L. Litynska-Dobrzynska¹, Ł. Major¹, G. Korznikova², A. Korznikov²¹Institute of Metallurgy and materials Sciences PAS, Krakow, Poland²Institute of Metals Superplasticity Problems RAS, Ufa, Russia

At temperature below 1273K the Ni₇₅(Al,V)₂₅ alloys are subjected to eutectoid decomposition, revealing in the equilibrium state characteristic microstructure resulting from the formation of the mixture of Ni₃Al and Ni₃V phases¹. The structures of this phases L₁₂ and D0₂₂ are highly ordered, dens packed, with high degree of coherence at the interfaces^{1,2}.

The path of the formation of such microstructure remains not completely clear, depending on the long time annealing, which finally leads to the lamellar type microstructure¹, revealing then good creep properties². The question of the present paper concerns phase composition, microstructure and properties achieved in alloys made by the cold crucible levitation melting, including directly cooling down in the copper levitator as well as their properties after further intensive plastic deformation.

The paper presents results of the investigation of the phase composition and microstructure of the Ni₇₅(Al,V)₂₅ and Ni₇₅(Al,V,Y)₂₅ (Y: Ti, Zr) alloys, cooled directly in the cold crucible levitation melting process and after intensive plastic deformation by the high pressure torsion. In case of the Ni₇₅(Al,V)₂₅ alloys after CCLM and crystallization, the Ni₃V phase, which should be one of the products of the eutectoid decomposition was not formed, but Ni₃Al phase and some amount of Ni(Al,V) solid solution were discovered.

In spite of the metastable phase composition, the alloys revealed high ductility in the compression test. The intensive deformation by the HPT increased metastability of the structure, leading to the Ni_3Al single phase composition. The addition of Ti (2.5 and 5 at.%) to the $\text{Ni}_{75}(\text{Al},\text{V})_{25}$ alloys was favorable for the Ni_3V phase precipitation and Ni_3Ti phase formation, however the Ni_3Al phase was still dominant. The solidus temperature of the alloys was lowered by the Ti additions. In case of alloys with the 2 and 4 at.% Zr additions, it was found, that even such small amount of Zr changed the phase equilibrium. In place of the $\text{Ni}_3\text{Al-Ni}_3\text{V}$ eutectoid phase mixture, the cubic $\text{Ni}_3(\text{Al})$ and Ni_7Zr_2 phases were identified in the alloys with higher Al content, while the tetragonal Ni_3V and Ni_7Zr_2 phases were formed in case of alloys with lack or low content of Al. Both microstructural and DSC investigations suggested that Zr additions shifted the eutectoid composition of $\text{Ni}_{75}(\text{Al},\text{V},\text{Zr})_{25}$ alloys to the smaller vanadium contents than in the $\text{Ni}_{75}(\text{Al},\text{V})_{25}$ alloys.

Conclusion: The $\text{Ni}_{75}(\text{Al},\text{V})_{25}$ alloys, both crystallized with relatively high rate and intensively plastically deformed revealed metastable phase composition, predominantly Ni_3Al phase. Even small additions of Ti and Zr were more favorable for the equilibrium $\text{Ni}_3\text{Al-Ni}_3\text{V}$ phase composition, leading however to the formation of the Ni_3Ti or Ni_7Zr_2 intermetallic phases.

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P-SN 03

The effect of alloying high-melting point elements on the microstructure and mechanical properties of nickel intermetallic alloy with polycrystalline structure

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The successful solution of development advanced gas turbine engine is impossible without using of new materials and manufacturing technologies. This refers to the nozzle and rotor blades, combustion chamber. To improve the efficiency of the cooling system combustion chamber requires materials with high working temperature (1200°C and above).

During recent years the high-temperature strength properties of the nickel base alloys have been improved due to increase in their composition of the total content of high-melting alloying elements decelerating diffusion processes that leads to increase in density of the alloys. That promoted the search for heat-resistant structural materials for the new generation of aircraft engines and stationary power plants of different purposes, which could be used at temperatures above 1100°C and which have more efficient composition, lower density and cost. Those high-temperature alloys could be made in the frame of well-proven technology of manufacture and treatment of the commercial nickel base superalloys are of a particular interest. Refractory nickel aluminides, are the most able to satisfy the requirements above.

Intermetallic nickel alloy VKNA-4UR was selected for research as a potential material for the manufacture of thin-walled details of the nozzle and the afterburner. This alloy is intended for the manufacture of details with a polycrystalline structure.

However, in the long-term rupture strength at 1100°C, the alloy does not satisfy the requirements of materials for details afterburner. To solve this problem and improve the mechanical properties we have changed the composition of the alloy adding high-melting point elements.

At high temperatures, the strength of materials depends only the rate of diffusion processes in the bulk grain, consisting of ($\gamma' + \gamma$) phases. Alloying with high-melting point elements can slow down the diffusion of elements in the bulk grain and at interfaces γ'/γ , which are closed around the γ -precipitate and do not create a continuous diffusion paths in the cross section of the samples.

As a result of the study, we increased long-term rupture strength values at 1100°C by 10-15%. Compared with basic alloy VKNA-4UR, these results showed a large improvement of mechanical properties, especially the long-term rupture strength at 1100°C, which makes this composite as a potential candidate material for thin-walled details of the nozzle and the afterburner.

Improving long-term rupture strength, ductility and tensile strength of the alloy while maintaining the density and heat resistance provides: increase of resource details 1.2-1.5 times, reduction of emissions by 25-35%.

P-SN 04

Structural perfection of cored turbine blades of single-crystalline CMSX-4 superalloyJ. Krawczyk¹, W. Bogdanowicz¹, R. Albrecht¹, J. Sieniawski², K. Kubiak², A. Onyszko²¹University of Silesia, Institute of Materials Science, Chorzów, Poland²Rzeszów University of Technology, Department of Materials Science, Rzeszów, Poland

The turbine blades are essential elements of the power-station turbines and jet engines. The nickel-based superalloys, contained about 70% of cubic γ' phase possess high temperature strength properties and resistance to creep and corrosion. The cored turbine blades of CMSX-4 superalloys were obtained by the Bridgman method in Research and Development Laboratory for Aerospace Materials in Rzeszów University of Technology. Analysis of the macro- and microstructure defects in turbine blades may allow for the elimination of the casting defects. The classic Laue diffraction method and the X-ray mapping performed by original OD-EFG diffractometer (EFG Company, Deutschland, Berlin) were used for crystal orientation study. The orientation maps were created for the different parts of the cored turbine blades. Additionally the maps of γ' lattice parameter were obtained for analysing the parameter constancy in the blades volume. The effect of misorientation angle on lattice parameter of γ' phase were analysed. The structural perfection of the blades were analysed by the OD-EFG diffractometry mapping method and the X-ray topography. The X-ray topograms were compared with SEM images of macro- and microstructure of cored blades. Disturbances of the main structure parameters in the cores areas of blades were studied.

P-SN 05

X-ray topography study of single crystals superalloy turbine bladesW. Bogdanowicz¹, R. Albrecht¹, J. Lelątko¹, J. Sieniawski², K. Kubiak²¹Institute of Materials Science, University of Silesia, University of Silesia, Chorzów, Poland²Rzeszów University of Technology, Rzeszów, Poland

Conventional methods for examination of single crystal superalloys base on obtaining X-ray diffraction patterns by the Laue technique. The Laue method is an excellent technique to determine crystal orientation but the main disadvantage is the small size of studied area of sample. To avoid this limitation and obtain information about crystal orientation of whole sample surface it is necessary to apply other diffraction techniques. The technique which has been used in overall single crystal inspection is X-ray diffraction reflective topography with divergent incident beam (micro-focus X-ray source). In this case it has been possible to examine crystal orientation of each fragment of surface samples. The only limitations of this method is the difficulty in interpreting the topograms.

The plate like samples were prepared from as-cast turbine blades of CMSX-4 alloy. The *high quality topograms* obtained by the Auleytner X-ray camera equipped with micro-focus X-ray source were analyzed. Additionally the samples were studied by Laue method. X-ray topograms shows band structure parallel to the blade axis. The bands observed in the topograms create parallel set along the blade axis with similar deviation as in Laue studies. Macroscopic low angle boundaries were revealed on the topograms. The topograms also show the macroscopic deformation of some parts of the blade which was probably caused by some imperfections of casting process. For correct interpretation of topograms the microstructure of whole sample surface was examined by SEM. Additionally, the testing with the use of new mapping technique of X-ray diffractometer made by EFG company (Deutschland, Berlin) was performed.

P-SN 06

Characterization of casting defects in the single crystal turbine blade tip regionW. Bogdanowicz¹, R. Albrecht¹, J. Sieniawski², K. Kubiak²¹University of Silesia, Institute of Material Science, Chorzów, Poland²Rzeszów University of Technology, Department of Material Science, Rzeszów, Poland

Nowadays, due to improvement in the directional solidification of turbine blades techniques the grain boundaries are almost entirely eliminated, which enables obtaining the single-crystalline blades.

Therefore, it become essential to pay attention and examine more subtle defects of single-crystalline blades, especially in their high loaded parts. In the paper the investigation of the CMSX-4 single crystalline turbine blades tip region by different X-ray diffraction methods complemented by SEM investigations is presented.

By the X-ray diffraction topography method several disorientation defects were shown as well as dendritic disorder in the turbine blade airfoil samples prepared from areas near leading and trailing edges.

Additionally the samples were studied by X-ray orientation mapping and mapping of γ' lattice parameter obtained on novel diffractometer provided by EFG company (Germany, Berlin). The dendritic array was determined by SEM methods and compared with the results obtained by X-ray diffraction methods. The possible defect formation process in tick (near leading edge) and tin (near trailing edge) areas of airfoil were proposed.

P-SN 07

Structure of multi-component super-alloy systemsB. Kodess¹¹ICS&E-VNIIMS, Crystals Metrology, Aurora, United States

X-ray data have been collected for the full Ewald sphere (from 3,000 to 8,000 reflections) using Mo K α radiation for several spheres prepared from single-crystalline blades of nickel aluminide-based super-alloys. Optimal performance characteristics of these alloys are determined by different ratios of alloy components. For the refinement of atom occupancies and thermal parameters (which are computationally correlated) we use a multi-step sequential approach. This method has been successfully used earlier during the refinement of the stoichiometric ratio for vanadium intermetallics and diborides containing vacancies in certain positions [1-2]. We show also the change in the structural characteristics of the external exposure on this single crystals. The results were compared with the data of X-ray microanalysis and electron microscopy analysis, which also confirmed the possibility of redistribution of elements in different crystallographic positions in the unit cells of the two main phases of a multi-component compound during the impacts.

P-SN 08

High temperature elastic properties of the nickel-base superalloy CMSX-4K. Demtröder¹, H. Buck², P. Wollgramm², G. Eggeler², J. Schreuer¹¹Ruhr-University Bochum, Institute for Geology, Mineralogy and Geophysics, Bochum, Germany²Ruhr-University Bochum, Institute for Materials, Bochum, Germany

Today composite pseudo-single crystals of nickel-base superalloys are widely used in the blades of gas turbines and jet engines. The high temperature conditions and mechanical stress may result in microstructural changes of the turbine blades. The characteristic microstructure can be described with two different phases: the gamma phase γ and gamma prime phase γ' . The γ' -phase consists of an ordered Ni₃Al-structure, which is embedded in a disordered γ -matrix [1, 2]. This exsolution structure enables an extraordinary mechanical strength at high temperatures [3]. This shows the importance of a better understanding of the mechanical properties as a function of temperature and mechanical stresses. The mechanical properties can be characterized by the elastic constants, which can easily be determined by the innovative technique of the resonant ultrasound spectroscopy (RUS).

The elastic and anelastic properties of nickel-base superalloys of a heat treated and an as-cast CMSX-4 were closely specified by RUS measurements in a temperature range from room temperature up to 1500 K. In addition to that, the

thermal expansion was investigated with the aid of a dilatometer. Microstructural characterization was performed by using a combination of optical microscope, electron microprobe analysis and scanning electron microscopy (SEM). Strong ultrasound dissipation effects develop above about 1100 K, which could indicate an increasing number of point defects. This coincides with the dissolving of the γ' -phase at around 1100 K. Furthermore a change of microstructure during thermal treatment has also an effect on the elastic properties.

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P-SN 09

Structural and morphological analysis of plasma jet synthesized thermal barrier coatings for aerospace applications

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Thermal barrier coatings (TBC) for aerospace applications consisting of multilayers of yttrium stabilized zircon (YSZ) and (NiCo)CrAlY alloy bond layers on top of NIMONIC or stainless steel test specimens are prepared by plasma jet deposition. The effect of thermal shock at 1000°C-1100°C followed by forced air cooling and water cooling on structural and morphological stability of TBC and substrate are investigated by transmission and scanning electron microscopy, X-ray diffraction and Raman spectroscopy prior and after thermal shock tests. Preliminary results of SEM and X-ray diffraction on stainless steel specimens covered with TBC show thermal protection capability of the TBC, as well as its structural and morphological stability.

Further details regarding the deposition method, thermal shock procedure, and structural results by TEM, SEM and XRD on various TBC multilayer composition will be presented in our presentation.

P-SN 10

Effect of Re on phase stability and hardness of Mo₅SiB₂

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Recently, the partitioning behavior of Rhenium (Re) added three-phase Mo-silicide alloys consisting of Mo_{ss}, Mo₃Si, and T₂ (Mo₅SiB₂) was studied by Yang *et al.* In their study, it was found that a small concentration of Re is soluble in T₂ phase while it increases the solubility of Si in the Mo_{ss} phase. T₂ is a key phase responsible for the high-temperature strength of Mo-Si-B alloys, and Re may change mechanical properties of T₂. The aim of this study is to investigate the effect of Re on the phase stability and hardness of T₂ in the Mo-Si-B-Re system. Re was added to Mo-Si-B alloys by a conventional Ar arc-melting technique. Cast samples were homogenized at 1800°C for 24h in an Ar atmosphere. T₂ single-phase region in a Mo-Si-B-Re quasi-ternary phase diagram at 1.4 at.% Re and 1800°C were experimentally determined using by electron probe micro-analysis (EPMA). T₂ single-phase region was slightly changed by the Re addition from the Mo-Si-B ternary phase diagram. The site occupation of Re in T₂ phase was estimated by first-principle calculation and high-resolution high-angle-annular dark-field (HAADF) scanning transmission electron microscopy (STEM). The HAADF-STEM observation suggested that specific Mo sites are partly replaced by Re in T₂ phase. However, first-principle calculation suggests that Re slightly decreases the phase stability of T₂. Nano-indentation hardness of T₂ phase at room temperature was measured using a Berkovich tip. It was found that the hardness is hardly changed by Re, indicating that Re is not an effective solid solution element in T₂.

P-SN 11

Processing of Mo-Si-B samples using nitride containing powder mixtures and their upscale towards larger itemsB. Klöden¹, A. Wiltner¹, I. Schönitz¹, G. Walther¹, T. Weißgärber¹, B. Kieback¹¹Fraunhofer Institute for Manufacturing Technology and Advanced Materials IFAM, Branch Lab Dresden, Dresden, Germany

Ternary mixtures exhibiting α -Mo beside the intermetallic compounds Mo₃Si and Mo₅SiB₂ offer the ability to control high temperature mechanical properties accompanied by applications of such tools at temperatures beyond those achievable by Ni-based superalloys. A continuous α -Mo matrix as well as intermetallic Mo₃Si and Mo₅SiB₂ intercalated within are required to reach application temperatures beyond 1200°C accompanied by excellent mechanical properties. Different processing routes are described in literature. In order to reach tools exhibiting larger scales as well as different shapes applying a short processing route we used the pressureless reactive sintering of powder mixtures containing Mo, Si₃Ni₄ and BN. The powders were mixed for minutes in a ball mill making a time expensive processing step like high energy milling dispensable. During the thermal treatment of these powder mixtures the nitrides were subsequently decomposed by silicide forming reactions and N₂ realization. We have analyzed the silicide forming reactions as well as N₂ realization in detail using thermal analysis (DTA) of powder mixtures containing different amounts of Mo, BN and Si₃N₄. Results from these measurements were used to consolidate samples of different shapes. Since the N₂ has to reach the surface to leave the sample achieving high densities the processing conditions have to be scaled up respectively. The densities reached by the processing route as well as the hardness values and results originating from 3-point bending tests will be presented here. Results from XRD measurements as well as SEM images complete the analysis of the Mo-Si-B samples.

P-FD 01

Structural and stability properties of $M_{2+x}Ni_{21-x}B_6$ ($0 \leq x \leq 1$) and $M_{2-x}RE_xNi_{21}B_6$ compounds ($M \equiv Ti, Zr, Hf$; $RE \equiv Y, Sc$)
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The $M_{2+x}Ni_{21-x}B_6$ ($M \equiv Ti, Zr, Hf$) compounds are the only ternary phases appearing in the M-Ni-B phase diagrams [1]; the solubility range of M has not yet been fully clarified, although the knowledge of the B-M-Ni ternary diagrams is of fundamental importance when dealing with wettability studies of Ni-B brazing alloys on Zr, Ti and Hf diborides [2, 3]. The formation of the ternary compound at the metal/ceramic interface can in fact modify the wettability features and negatively affect the reliability of the joint. The partial substitution of the transition metal by an ion with similar size, such as Y or Sc, can help to define the stability ranges of the $M_{2+x}Ni_{21-x}B_6$ phase as a function of the mean ionic radius.

$M_{2+x}Ni_{21-x}B_6$ and $M_{2-x}RE_xNi_{21}B_6$ were synthesized by arc-melting the elemental metals in Ar atmosphere and by subsequently annealing the obtained samples in a vacuum at 900°C. Powder diffractograms were collected and refined by means of the Rietveld method, and polished surfaces of the samples were analyzed by SEM-EDS.

$M_2Ni_{21}B_6$ compounds are reported to crystallize with the $Cr_{23}C_6$ -type structure, belonging to the $Fm-3m$ space group [4]. Nevertheless, they are sometimes reported as being characterized by the stoichiometry $M_3Ni_{20}B_6$ [5], that differs from the former for the substitution of M on one of the crystallographic sites of Ni; in this work structural and microstructural studies on the Zr-Ni-B system are presented, that show a certain solubility of Zr on the $4a$ site occupied by Ni in $Zr_2Ni_{21}B_6$; the stability of the $M_{2+x}Ni_{21-x}B_6$ phase with $0 \leq x \leq 1$ can be then assessed. Besides, a more general structural study has been undertaken with the aim to investigate the effect of the partial substitution of the transition metal (Hf, Ti, Zr) by Sc and Y on the stability and the stoichiometry of the ternary compound.

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P-FD 02

Elastic and thermodynamics properties of the B2-ErX (X=Cu, Au, Ag, Ir) type rareearth intermetallic compounds from ab-initio calculations.

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The full-potential linearized augmented plane wave (FLAPW) method were employed within the generalized gradient approximation (GGA) to investigate structural and elastic properties of ErX (X= Cu, Au, Ag, Ir) compounds. The ductility or brittleness of these intermetallic compounds is predicted, through this investigation reveals that the ErAg crystal is more ductile. Moreover, the elastic constants values and the sound velocities were discussed for longitudinal and shear waves. In addition, the chemical bonding of these compounds has been investigated in light of topological analyses approach grounded in the theory of atoms in molecules (AIM). All of the electron density critical points in the unit cell were systematically calculated in order to predict basins interaction of each atom.

P-FD 03

Phase field model for solid phase transformation driven by the elasticityO. Tschukin¹, D. Schneider¹, B. Nestler^{1,2}¹University of Applied Sciences, Institute for Applied Materials, Karlsruhe, Germany²Karlsruhe Institute of Technology, IAM-ZBS, Karlsruhe, Germany

In our work we present a new approach for small deformation elasticity for solid-solid interaction in the phase field context. We derive our model according to the classical Hadamards jump condition on the deformation and to the traction condition for the stresses at the interface. Hence we formulate a consistent description of the energy landscape through the interface between phases. Comparing to the previous models, once corresponding to the Reuss/Sachs interpolation scheme where the homogeneity of stress is conserved and twice for the Voigt/Taylor approach with homogeneity of strain, this model compound both approaches and differs the homogeneity system variable in dependence of the interface orientation. We extend our model for the situation where the phases are afflicted with eigenstrains or eigenstresses and present the comparison of the simulation results with theoretical predictions.

P-FU 01

Thermal properties of solid solutions $\text{Yb}_{14}\text{MnSb}_{11}$ and its solid solutions of $\text{Yb}_{1-x}\text{Lu}_x\text{MnSb}_{11}$
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$\text{Yb}_{14}\text{MnSb}_{11}$ compound is attributed to Zintl phases and is considered as perspective materials for transferring the energy to long distances. Zintl phases has good electrical properties with low thermal conductivity. It was shown that Seebeck coefficient can be increased using pressure. This induced us to use chemical pressure for this structure type. As a result, solid solutions with ytterbium part substitution by lutetium were obtained. From the thermo electrical material perspective, thermal stability of these objects is also functional property since while applying these materials should function at high temperatures where the processes of diffusion, volatility, oxidation can easily occur. In this work, data on thermal properties is presented: melting temperatures, thermal expansion.

Crystals were obtained by flux method using tin as solvent. X-ray diffraction analysis of the crystal samples was made using DRON-UM(R=192 mm, CuK α -radiation, Ni-filter, scintillation detector with amplitude discrimination, step 0,02° 2 θ , impulse acquisition interval in each point 3c, room temperature). Analysis of quantitative composition of samples was performed by microprobe method using JXA -8100, JEOL (Japan), with voltage 20 kV and current 30nA. Thermal analysis method was the following: inert gas pressure in chamber - 7 atm, heating rate 3000°C/min, this way the conditions of piston pressure were created that minimized the vapor loss of the volatile component from crucible to chamber. Thermal expansion was investigated in the interval of 20-750°C. Coefficients of thermal dilatation were found, Debye temperatures were calculated. These investigations have shown that:

- 1. All solid solutions are crystallized in tetragonal structure of $\text{Ca}_x\text{MnSb}_{11}$, X-ray analysis has shown the monophasic of all the samples. Microprobe analysis demonstrated availability of other phases and tin but they did not exceed 1%.
- 2. All solid solutions are melted incongruently at temperatures higher than 1500°C
- 3. All studied properties demonstrate that lutetium substitute ytterbium up to $x=0,5$ composition in crystal lattice $\text{Yb}_{14}\text{MnSb}_{11}$.
- 4. Acknowledgements This work was supported by International Science & Technology Center (ISTC), #Project T-1597

P-FU 02

The shot noise model to adjust to noise in YBaCuO HTc thin superconductors
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The investigation of the vortices dynamic in an important in order to limit as much as possible the dissipation mechanism in practical in high critical temperature superconducting materials. For this purpose, Van Gorp et al. [1] used, for the first time, in the early 1960 the measurements of the voltage noise. This researcher was able to establish the first model relating the dynamic of vortex matter to the power spectral voltage noise. This model is named shot noise model and was successful to some extent to describe the kinetic of vortices in various superconducting materials. In the case of $\text{YBa}_2\text{Cu}_3\text{O}_{7-d}$ high Tc superconductors, the voltage noise measurements were being adjusted with the shot noise model.

The measurements were performed for a high external magnetic field so that the vortices will be densely packed in order to exhibit a collective behavior. Our sample is carrying a transport dc current of 1nA. The important findings in this article are: (1) the fit of the voltage noise with the shot noise model leads to determine the time transit t and the mean velocity v of the vortices. (2) The graphic presentation of those parameters versus temperature shows that the shot noise model describes perfectly the obtained from the qualitative viewpoint. (3) the estimation of the noise in the studied materials basing on the shot noise formulas was found to be very smaller compared with the experimental results.

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P-FU 03

Positron study of small pinning sites in $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ V. Slugen¹, J. Simeg-Veternikova¹, S. Sojak¹¹Slovak University of Technology, Institute of Nuclear and Physical Engineering, Bratislava, Slovakia

High temperature superconductor $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ was irradiated by fast neutrons for generation of spherical defects with sizes of a few nanometers, which influences as additional pinning centers and enhances critical current J_c . However, also small vacancy defects are accumulated into the structure during the irradiation. These point defects are not enough detectable by transmission electron microscopy; therefore Positron annihilation lifetime spectroscopy was applied to investigate their size and concentration in this work. Two different bulk samples, pure non-doped $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ (YBCO) and multi-seed $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ doped by platinum (MS2F), were studied prior and after irradiation in TRIGA MARK II reactor in Vienna. The fast neutron fluence achieved up to $6 \times 10^{21} \text{ m}^{-2}$. After first measurements, the samples were annealed and the recombination of the small defects was also observed.

Samples demonstrated presence of Cu-O di-vacancies with concentration proportional to exposure time. After the annealing, the small defect concentration was visible reduced in the both samples. More intensive defect accumulation during the irradiation was found in YBCO, which has also better improvement of superconducting properties.

P-FU 04

On the formation of internal tin Nb_3Sn superconducting compoundA. Hangga¹, D. Risanji¹, A. Pramono²¹Institut Teknologi Sepuluh Nopember, Engineering Physics, Surabaya, Indonesia²Indonesian Institute of Sciences, Research Center of Metallurgy, Tangerang, Indonesia

Nb_3Sn is intermetallic superconducting compound having A15 structure which was formed through interdiffusion process at high temperature. It is known that for Nb_3Sn superconductors manufactured by internal tin process, the most appropriate heat treatment is still a matter of debate. Some problems arise during present fabrication, such as oxidation, Sn leakage and inhomogeneity in the strands. Our experiment results show that in the temperature range of 700-850°C for 96 hours the Nb_3Sn was already formed together with minor amounts of other compounds, such as Nb_6Sn_5 . For lower temperature and shorter time of heat treatment the more Nb_6Sn_5 compound was formed.

In order to optimize the process we employ simulation of Cahn-Hilliard equation (CH) applied in finite difference to predict and optimize the formation of Nb_3Sn compound. Several factors affecting formation of Nb_3Sn compound such as composition, geometry, temperature and time of heat treatment were involved.

P-FU 05

The effect of the mechanical alloying process on the magnetic, structural and micro-structural properties of $\text{Ni}_{63}\text{Fe}_{13}\text{Mo}_4\text{Ti}_{20}$ alloyL. Karimi¹¹Shiraz University of Technology, Shiraz, Iran

In this work, the magnetic, structural and micro-structural properties of amorphous/nanocrystalline $\text{Ni}_{63}\text{Fe}_{13}\text{Mo}_4\text{Ti}_{20}$ powders prepared by mechanical alloying (MA) are investigated. Structural and micro-structural studies were carried out by x-ray diffraction (XRD), high resolution transmission electron microscopy (TEM) and scanning electron microscopy (SEM). The ball-milling of Ni, Fe, Mo and Ti powders leads to alloying the element powders. At 120h, the Ni main peak is completely broad and the crystallite size is about 3 nm with a minute amount of Mo phase due to high melting point, hardness and low diffusion at low temperature of Mo in to Ni. The results reveal that the mechanically powders alloyed for 120h has better operating frequency and soft magnetic properties as compared with other samples. At 190h the intensity of Ni main peak is higher and the Ti peak is creating again. The inter-atomic distance between transition metals (2.545 nm) and the peak shift ($\Delta 2\theta = 1.08$ degree) are maximum at 120h milling. The magnetic measurements of sample milled for 120 h show low magnetic permeability and high operating frequencies up to 1000 Hz.

P-FU 06

Surface topography, microstructure and magnetic domains in Al for Sn substituted ferromagnetic Ni-Mn-Sn Heusler alloy ribbons

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Ternary intermetallic Heusler compounds of general formula Ni-Mn-X (Ga, Sb, In, Sn) have attracted considerable attention due to the diverse structural and magnetic properties they exhibit. Among them off-stoichiometric, Mn-rich Ni-Mn-Sn alloy has been shown to display a large negative magnetoresistance effect accompanied by the magnetic field induced reverse transformation. Large magnetic entropy changes associated with this transition have also been reported for this alloy system, rendering it promising for magnetic refrigeration applications. Recently it has been demonstrated that Al substitution for Sn may lead to an increase in the martensitic transformation temperature in these alloys. The research has focused on bulk polycrystalline as well as melt spun ribbons prepared from these materials. The latter have the advantage of faster heat exchange between the working body and the exchange fluid and thus may improve the technical characteristics of a refrigeration unit. Furthermore rapid solidification allows for avoiding of the thermal annealing step to produce a single phase, homogenous alloy and it yields highly textured polycrystalline ribbons with enhanced magnetocaloric properties along a specific direction.

Microstructure and the surface properties are therefore an important consideration with respect to practical use. This paper presents a detailed atomic force microscopy study of the $\text{Ni}_{48}\text{Mn}_{39.5}\text{Sn}_{12.5-x}\text{Al}_x$ ($x=0, 1, 2, 3$) ribbons. This is a novelty since so far the literature lacks such data. It is shown that the surface of the Al free samples is composed of two types of subgrains, which aggregate into larger grains. One type is of 25-30 nm in height and the other is up to 0.5 μm in diameter and 60-130 nm in height. The Peak Force analysis revealed that the reduced Young Modulus between the two phases differs notably. The average grain size is 550-580 nm. The analysis of Al containing samples clearly shows two distinct morphologies indicating the presence of a two phase structure. This is in accordance with X-ray diffraction and transmission electron microscopy studies, which proved the coexistence of martensite and austenite at room temperature in these samples. Depending on the phase two different types of magnetic domains were observed and they also differed in terms of a magnitude of the emitted magnetic field. A characteristic twinned plate like microstructure is also seen in the 3Al sample confirming the presence of an increased amount of martensite. The martensitic plates are 70-100 nm in size. The phase shift of 0.5° was detected while scanning this sample (MFM mode) indicating the existence of a magnetic multi-domain structure. Two different morphologies were observed on the top and bottom side of ribbons which is related to rapid quenching on a spinning copper wheel and influences the magneto-mechanical properties of these materials.

P-FU 07

Fabrication of thermoelectric modules through Ag/Sn/Ag thin-film intermetallics reactions

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Thermoelectric materials have been applied for the recycling of industrial waste heat at medium and low temperatures. For the manufacturing of thermoelectric modules, the traditional soldering method for the bonding of TE devices with electrodes has a disadvantage that the operation temperature of the finished TE modules is limited by the melting point of solder alloy. Brazing method uses filler metals with higher melting points and the manufactured TE modules can endure higher temperature. However, the thermal stress caused by the higher bonding temperature during brazing process can lead to the failure of TE modules. An alternate method through the intermetallics reactions of Ag/Sn/Ag was adopted for the packaging of TE modules. The principle of this technique uses a Sn film with a thickness of several micrometer to react with Ag layer deposited on both TE device and metallic electrode, which results in the formation of a Ag_3Sn intermetallic compound, which possesses a high melting point of 480°C. Meanwhile, the low melting point Sn thin film has been exhausted. Therefore, the finished TE modules can endure much higher temperature than that manufactured with traditional soldering method and prevent the thermal stress induced damage during the brazing process. This technology has been verified on various thermoelectric materials such as $\text{Bi}_{0.5}\text{Sb}_{1.5}\text{Te}_3$, $\text{Bi}_2\text{Te}_{2.55}\text{Se}_{0.45}$, PbSnTe , GeTe(Pb) and ZnSb .

The results indicate that sound interfaces without voids or cracks are obtained in all cases. The optimized bonding strengths for various TE materials can attain satisfactory values over 15 MPa. Furthermore, in order to extend the operating temperature range of different thermoelectric materials and increase the efficiency of TE modules for the waste heat recycling, a cascade TE module is required. The traditional method for the manufacturing of such a cascade TE module is brazing of medium or high temperature thermoelectric device following with soldering of low temperature thermoelectric device. This study has also verified that a cascade TE module can be successfully fabricated in a single stage by this innovative method through Ag/Sn/Ag intermetallics reaction.

P-FU 08

Processing and microstructures of Ti-Ta high temperature shape memory alloys

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Ti-Ta shape memory alloys (SMAs) show a martensitic transformation at temperatures close to 500K. Most importantly, they are very ductile, and in contrast to many other high-temperature SMAs (HT-SMAs), they show an excellent workability. However, the preparation of Ti-Ta SMAs is challenging because the melting point of Ta is close to the evaporation point of Ti. Furthermore, the solidification of molten Ti-Ta is associated with decomposition processes. In the present work we show, how homogeneous Ti-Ta alloys can be prepared through arc melting, heat treatments and thermomechanical processing. The resulting materials were characterized through differential scanning calorimetry, optical, scanning and transmission electron microscopy. Ti-Ta ingots have to be remelted several times to ensure a macroscopic homogeneity. Homogenization annealing at 1100°C for 25 hours is required to sufficiently reduce microscopic concentration gradients resulting from solidification. Ti-Ta alloys can be easily deformed through cold rolling. Deformation levels of up to 90% can be achieved, even without any pre-heating. In the present work, we also provide first results on the effect of omega-phase formation on martensitic transformations in Ti-Ta.

P-FU 09

On the functional properties of FeNiCoAlTa shape memory alloys

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Iron-based shape memory alloys (Fe-based SMAs) such as FeMnSi and FeCoNiTi attracted a lot of attention in the last few years due to their good workability and lower processing costs compared to conventional NiTi SMAs. Most Fe-based SMAs exhibit high theoretical transformation strains, which make them also attractive for superelastic and damping applications. However, recent studies show that the experimentally determined transformation strains of about 2% are much lower than the predicted values stemming from theoretical calculations. By contrast, superelastic strains of about 13% have been demonstrated for textured FeNiCoAlTa SMAs in single cycle tests. For actual applications, however cyclic stability is crucial. Still, data reporting on functional degradation of FeNiCoAlTa are extremely rare in open literature, and thus, solid conclusions on the role of microstructural features on cyclic stability are not possible, yet.

Therefore this study focuses on the cyclic stability of the alloy under conditions where stress-induced and thermally induced martensite forms. Cubic and dog-bone shaped specimens were subjected to iso-stress and iso-temperature experiments in order to investigate the deformation behavior under cyclic loading conditions. Specimens were cycled up to 4.5% in the superelastic regime and in-situ observations were conducted in order to evaluate variant-variant interactions and the local strain evolution on the sample surface up to 100 cycles. Temperature cycling experiments were conducted with superimposed stresses up to 600 MPa in the temperature range between -140°C and 50°C in order to identify the maximum transformation strains in both tension and compression.

P-FU 10

On the influence of inclusions on fatigue life of pseudoelastic NiTi shape memory alloysM. Rahim¹, J. Frenzel¹, M. Frotscher¹, R. Steegmüller², M. Wohlschlägel², G. Eggeler¹¹Ruhr-Universität Bochum, Lehrstuhl Werkstoffwissenschaft, Bochum, Germany²Admedes Schuessler GmbH, Pforzheim, Germany

In the present work, we investigate how oxide and carbide inclusions affect the fatigue behavior of pseudoelastic NiTi shape memory alloys (SMAs). Three types of NiTi SMA wires (high purity, O-rich and C-rich) with different oxygen and carbon impurity levels (both ranging from 0.004 to 0.05 wt.%), but very similar mechanical behavior, were prepared by arc melting and wire drawing in combination with heat treatments. Structural fatigue testing was carried out by bending rotation fatigue using electropolished samples to guaranty a good surface quality. The resulting fracture surfaces were characterized extensively through scanning electron microscopy. It was found that inclusions strongly affect fatigue performance, once other critical surface defects resulting from wire drawing are removed through electropolishing. For strain amplitudes above the transformation strain, the wire with the highest purity clearly outperforms the wires with high C- and O-levels. For strain amplitudes below the transformation strain, the high purity wire and the C-rich wire show higher fatigue lives than the material with a higher O-level. It was found that inclusions which are associated with voids (particle/void-assemblies) represent preferred crack initiation sites. The low fatigue life of oxygen-rich wires is directly related to the high frequency of this type of defect in the material.

P-FU 11

On the effect of crystallographic compatibility on hysteresis widths, transformation strains and irreversible strains in binary NiTi shape memory alloysS. Jaeger¹, J. Frenzel¹, B. Maaß¹, O. Kastner^{1,2}, G. Eggeler¹¹Ruhr-Universität Bochum, Institut für Werkstoffe, Bochum, Germany²GeoForschungsZentrum, Potsdam, Germany

Nickel-titanium (NiTi) shape memory alloys (SMAs) are fascinating materials, which exhibit a thermal (one way effect, two way effect) or a mechanical (pseudoelasticity) shape memory effect (SME). The occurring SME depends on the alloy composition and the thermo-mechanical treatment of the specimen. NiTi SMAs show superior functional and structural properties which are exploited in medical technology for stents and surgical instruments. Frenzel et al. have shown, that the Ni-concentration has a large effect on the thermal hysteresis widths [1]. Moreover, Ball and James have proposed that the width of the thermal hysteresis is governed by the crystallographic compatibility between the two phases austenite and martensite [2]. The present work shows, that the Ni-concentration also affects the mechanical hysteresis widths, as well as the irreversible and transformation strains observed in pseudoelastic loading/unloading experiments. Furthermore it is shown that an increasing quality of the crystallographic fit between the austenite and martensite lattices not only reduces the width of thermal but also the width of mechanical hysteresis. These relations governing the thermal and mechanical shape memory behaviour were established focusing on a simple binary NiTi system, where different lattice misfits were adjusted through varying Ni-concentrations.

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P-MI 01

Thermodynamic of new intermetallics for lithium-ion battery within the Cu-Li-Sn systemD. Henriques¹, V. Motalov², L. Bencze³, T. Markus¹

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Li-ion Batteries have a high potential to efficiently store electric energy. These Li-ion Batteries can be used in portable electronics, as a backup-system in case of a blackout, in mobility or aerospace application and for temporary storage of energy produced by renewable Energy. It is important to design the Li-ion Batteries for their respective application. To achieve all the requirements for Li-batteries, a systematic investigation of materials used for cathode, anode, electrolyte and separators is necessary to improve the understanding of thermodynamics, kinetics, crystal chemistry as well as micro-and nanostructures of the materials involved. Because of the high amount of potential components in Li-systems, a profound thermodynamic data and phase equilibrium information is desirable to create a concise experimental phase diagrams for the respective systems as a basis for corresponding modeling (e.g. CALPHAD). The knowledge of basic thermodynamic data in Li-systems is very weak. So it should be enhanced for advanced materials design based on thermodynamic modeling.

With the method of Knudsen Effusion Mass Spectrometry (KEMS) it is possible to determine thermodynamic activities, partial and integral molar Gibbs energy, enthalpy and entropy changes of mixing. The method of KEMS allows a qualitative and quantitative analysis of the equilibrium gaseous phase. Intensities of ions formed from the gaseous species over the condensed sample were measured by a secondary electron multiplier. From the temperature dependence of the measured ion intensities the partial pressure of the neutral molecules can be calculated as well as the thermodynamic activities. It is important to evaluate the KEMS set up accurately for Li-systems. This requires a validation by a limited number of experiments in well investigated systems. For this reason measurements within the Li-Sn system have been performed for the concentration from 0,1 mole fraction of Li to 0,81 in a temperature range between 450°C and 750°C. The measured activities are in good agreement with the available literature data and the reliability of the computed excess Gibbs-Energy (ΔG_{ex}) data could be demonstrated with literature data. Additionally the binary system of Cu-Li were measured between the concentration of 0,3 mole fraction of Li and 0,81 to obtain thermodynamic data between 400°C and 600°C. The ternary system Cu-Li-Sn is a feasible anode material. Up to now there is no phase diagram described in literature for the Cu-Li-Sn system and the available thermodynamic data for the respective system is poor. Therefore samples within this system were chosen and thermodynamic data were derived during measurements with the KEMS set up. Thermodynamic activities were measured over the solid condensed phase and over the liquid phase. Excess Gibbs-Energies (ΔG_{ex}) were computed directly. This thermodynamic data for the ternary system Cu-Li-Sn is important as a basis for calculating the phase diagram.

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P-MI 02

Nucleation and growth of the Laves phase during long term creep of 12% Cr tempered martensite ferritic steelsM. Isik¹, A. Kostka¹, G. Inden², G. Eggeler^{1,3}

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The objective of the present study is to reveal dominant processes governing nucleation and growth of intermetallic Laves phase (Fe_2Mo) during creep of tempered martensite ferritic steel X20CrMoV12-1. Prior to creep test, steel was subjected a two stage heat treatment; austenitizing at 1050°C for 0.5 h and tempering at 770°C for 4 h both followed

by air cooling. After tempering, the microstructure consists of an ultrafine grained ferritic matrix with high density of dislocations and two types of precipitates: $M_{23}C_6$ and V-rich MX. Interrupted long term creep tests were performed up to 139971 h at 550°C and 120 MPa. Thermocalc/Dictra calculations demonstrated that Laves phase is not stable in given composition (0.5 at.% Mo), while we observed large quantity of Laves phase particles (up to 1.4% volume fraction after 139971 h of creep). Investigations on shorter crept samples (2400 h) untied the contradiction; local composition variation (~15 at.% Mo) was observed in regions where Laves phase formation is thermodynamically favorable. Our results shows that the locally Mo-rich grain boundaries decorated by $M_{23}C_6$ carbides are the nucleation sites for Laves phase when TMFSs are exposed to high temperatures. Its further growth is controlled by GB diffusion. After long term creep, the size and number density of Laves phase particles reach ~400 nm and 0.13 respectively.

P-MI 03

Precipitation hardening of austenitic-ferritic steel 03Kh13N10K5M2Yu2T

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Corrosion-resistant austenitic-ferritic steels, known as Duplex stainless steels have a number of advantages over austenitic steels and are successfully used in various industries. The study of austenitic-ferritic steel 03Kh13N10K5M2Yu2T has a reduced chromium content, which makes it possible to exclude the formation of σ -phase and avoid the 475°-fragility. Low carbon and alloying of steel by titanium and aluminum contribute to the formation of hardening intermetallic phases. The use of thermoplastic processing, represented by the following: quenching in the supersaturated solid solution, deformation and subsequent aging can help to achieve high-strength state ($\sigma_b = 2500-2800$ MPa). After quenching from 1000°C steel contains approximately equal amounts of austenite and δ -ferrite. A specific feature of the steel – anomalous δ -ferrite hardness after quenching (500HV), while the hardness of the austenite 200 HV. The reason for the abnormally high hardness δ -ferrite is its decay, emitting intermetallic ordered phase (Fe, Ni) Al with lattice B2.

Cold plastic deformation of steel can lead to almost complete transformation of austenite to strain martensite. Post deformation ageing leads to an additional increase of the strength properties of steel through the course of hardening of supersaturated δ/α solid solution with the formation of the ordered intermetallic phase (Fe, Ni) Al.

P-MI 04

Effect of heat treatment on structure and phase constituents evolution in highly-doped eutectoid titanium alloys

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Recently the scientific activities in the field of searching for new alloying systems and chemical compositions of titanium alloys as well as new approaches to the development thereof have been of a particular interest due to the fact that by nowadays almost all possibilities for service properties improvement of conventional titanium alloys (which possess tensile strength of 1000-1300 MPa and plastic elongation percent of 8-15) have been completely exhausted. Among other investigations one can single out the research projects on eutectoid and hypereutectoid titanium alloys including those of Ti-Fe-Co-Sn alloying system. It has been stated that these alloys can exhibit high compression strength (~2600-2800 MPa) and high ductility level (18-24%), while no rapid solidification is required for ingots production. Our present work involves the investigations of heat treatment influence on microstructure and phase constituents in a pilot Ti-Fe-Co-Sn alloy. The results of this project will give the opportunity to set up an effective structure and properties management by means of thermal and thermomechanical treatments.

P-MI 05

Influence of intermetallics on the surface properties of an aluminium alloy of 1100 series during hot rolling and annealing
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Hot rolling is used for the reduction of the thickness under high mechanical stress and temperature. Those two parameters induce transverse cracking, voids, wear debris and precipitates to the surface and below the surface area. Those phenomena are examined in this study. One of the most demanding applications of hot rolled aluminium is the printing industry, whereby the printing quality is well connected with good surface properties such as homogeneous grain size distribution and the absence of surface defects introduced to the surface during the rolling process. Thus, the aim of this work is to understand the influence of the temperature, hot rolling and heat treatment on an AA 1100 series used as a matrix in the printing industry. Surface intermetallics, precipitates and other inclusions are examined in relation to the as cast material, after hot rolling and after annealing. We chose those stages of the process to highlight the importance and the evolution of precipitates and oxides in the surface properties. Surface analysis techniques such as Auger Electron Spectroscopy and Secondary Electron Microscopy were used. The near surface layer (1-5 μm below the surface) is known to be highly deformed making the material more susceptible to corrosion. For this reason cross sections were also prepared with the use of a Focused Ion Beam and a Cross Section Polisher. A TEM was used with a newly developed technique called Automated Crystallographic Orientation Mapping to observe small grains in such a deformed layer. The grain structure, the intermetallics, the precipitates, the oxides and their structure were all investigated for the different samples mentioned above. From this study, we aim to optimize the production line of hot rolled aluminium alloys by understanding the important relationship between the near surface microstructure and the rolling parameters.

P-MI 06

Engineered interface/interphase in Cu-Zn/Al laminates induce non traditional metal matrix composite

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Hot uniaxial pressing of Cu-Zn/Al laminates in foil-foil processing technique (Al/Cu-Zn/Al) is a target for aerospace engineering. However, delamination is a major limitation of lamellar composite structure; interface/interphase control is an objective. Mitigation of crack as well as toughening mechanisms is introduced with parametric study (temperature, pressure and holding time) and established for the control of interface/interphase kinetics. Hot uniaxial pressing parameters as well as alloying elements have a dominant effect on the kinetic of interface/interphase formation. Lamellar metal matrix composites introduced with new micro laminated composite interphase. The addition of stainless steel powder is also introduced for the interface/interphase control. Alloying element segregation with diffusion mechanisms induced the formation of localized metal flow along with the interface as well as, low melting phase (LMP). LMP induced the delocalized melting phase structures enriched with Al phase, Cr-Fe phase, and Al-Cu-Zn phase.

Alloy segregation during solidification in a semisolid state processing induces directional solidification with fibrous and bulk whiskers structural interphase. Microstructural characterization, mechanical characterization is also established via optical microscopy scanning electron microscopy, energy dispersive X-ray spectroscopy and tensile testing. Chemical and mechanical bonding via inter diffusion processing with LMD are dominant for interphase kinetics. Mechanical characterization with interfacial shear strength is also introduced with hardness testing. The interphase kinetic established through localized micro plasticity, metal flow, LMD and delocalized Al oxide and Mg oxide. The morphology of interphase is cached with interesting structures that need more studies.

P-MI 07

Microstructural and mechanical characterization of as-cast Ti-Nb-Zr alloys for biomedical applicationsT. Matković¹, L. Slokar¹, P. Matković¹¹University of Zagreb, Metallurgy, Sisak, Croatia

Titanium and its alloys have been extensively used in the medical applications because their prominent biological, mechanical and physical properties. Recent biomaterials research has been focused on developing of new β -type Ti alloys with lower E-modulus and better formability. The purpose of this work is to provide characterization of Ti-Nb-Zr alloys with emphasis in the property- microstructure- composition relationships. Six ternary Ti-rich alloys with Nb (10-30 at.%) and Zr (10-30 at.%) as the betha- phase stabilizers were produced from commercially pure materials by an arc melting method. Their casting, in the cylindrical samples, was made using the same equipment. Specimens, in as-cast and heat-treated conditions, were examined by light and scanning electron microscopy (SEM). These results suggested that alloys with 10 at.% Nb were single β -phases and that those with 20–30 at.% Nb consisted of the β -phase and the nidle like α'' - phase. The average chemical composition of both phases was established by EDX analysis and corresponded to the chemical composition of each alloy. Quantitative microstructural analysis was performed according to standard ASTM 112-96. Hardness was measured by Vickers method. The hardness values of single β -phase alloys were lower and obtained data (336 to 420 HV) were inside of limits for biomedical alloys. Finally, these alloys exhibit the β -type Ti- structure with properties which are promising for their application in biomedicine.

P-MI 08

Thermoelastoplastic and vibration characteristics of composites with interphasesY. M. Shabana¹, R. El-Adl. M.², E. I. I. Morgan¹¹German University in Cairo, Faculty of Engineering and Materials Science, Mechatronics Department, New Cairo City, Egypt²El-Mataria Helwan University, Mechanical Design Department, Faculty of Engineering, Cairo, Egypt

This paper addresses the problem of predicting the behaviors of a composite material, which consists of a matrix and a spherical inclusion coated by a multi-layered interphase, under thermal and/or mechanical loading variations and based on the micromechanics principles. The multi-layered interphase, which in general includes different properties for each layer, is modeled by applying the multi-inclusion model. The considered damage mode, which is represented by the progressive debonding of the particle from the interphase, is assumed to be controlled by a critical energy criterion and the Weibull's distribution function. The effects of the interphase parameters such as its thickness and the properties of each layer on the effective thermomechanical properties and the vibration characteristics of composites with multi-layered interphases are presented and discussed. The natural frequencies give information about the resonance avoidance whereas mode shapes give information about observability and controllability of different structures. Therefore, the natural frequencies and mode shapes as vibration characteristics are investigated based on Euler and Timoshenko theories.

P-MI 09

Mechanical properties of martensitic precipitation hardening stainless steels strengthened by Ni₃Ti and NiAl intermetallic phasesT. Horiuchi^{1,2}, K. Shimoda² and M. Yamaya²¹ Max-Planck-Institut für Eisenforschung GmbH, Düsseldorf, Germany² Hokkaido Institute of Technology, Hokkaido, Japan

Applications of martensitic precipitation hardening (PH) stainless steels have been spreading because of their excellent strength and corrosion resistance. In order to achieve further improvement of their mechanical properties, authors have developed four martensitic PH stainless steels strengthened by η (Ni₃Ti) and NiAl phases based on the commercial "Custom 465" PH stainless steel (Fe-11Ni-12Cr-1Mo-1.6Ti). Chemical compositions and temperatures for the solution heat treatment of the developed PH steels were estimated by thermodynamic calculations using Thermo-Calc. Tensile and impact tests were carried out for specimens of the steels before and after aging heat treatment. Microstructural observations and X-ray diffraction analysis were also performed. Precipitation of η and/or NiAl phases was confirmed

by transmission electron microscopy in all the steels after aging at 550°C. The tensile strength was increased after aging, especially for the steels with precipitation of NiAl phase. However, one of the steels with precipitation of η and NiAl phases did not show improved tensile properties. This steel also contained a relatively high amount of retained austenite. It thus should be crucial for the developed PH steels to control the retained austenite as well as the precipitation of η and NiAl phases.

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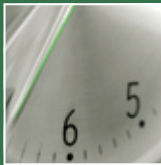
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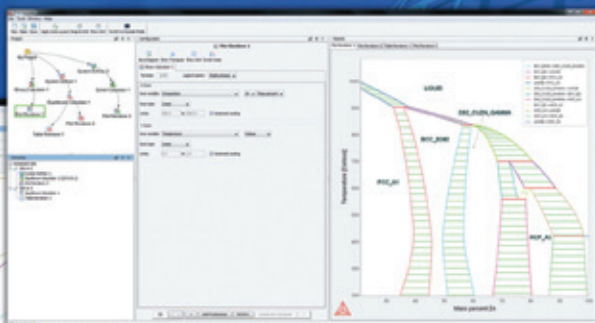
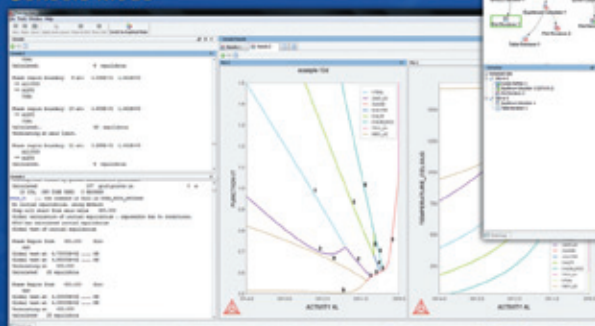
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