22 $2d^2 4s^2$

**Ti**

Titanium

47.867

13 $3s^2 3p^1$

**Al**

Aluminium

26.981

# IWTA2023

7th International Workshop on Titanium Aluminides

June 11-16, 2023, Toulouse, France

## Programme & Abstracts

# PROGRAMME OVERVIEW

| Sunday June 11 | Monday June 12 | Tuesday June 13 | Wednesday June 14 | Thursday June 15 | Friday June 16 |
|---|---|---|---|---|---|
| | 08:30 - 10:10 | 08:30 - 09:50 | 08:30 - 09:50 | 08:30 - 09:50 | 08:30 - 10:00 |
| | Processing & Applications | Fundamental | Fundamental | Additive Manufacturing | Alloy Properties |
| | | *Coffee Break* | *Coffee Break* | *Coffee Break* | |
| | *Coffee Break* | 10:20 - 11:20 | 10:20 - 11:50 | 10:20 - 11:20 | *Coffee Break* |
| | 10:40 - 12:10 | Additive Manufacturing | Alloy Properties | Processing & Applications | 10:30 - 12:00 |
| | Additive Manufacturing | 11:20 - 12:30 | | 11:20 - 12:30 | Summary & Discussion |
| | | Posters | *Buffet 12:00 - 13:00* | Posters | *Buffet & Departure 12:00 - 13:30* |
| | *Lunch 12:30 - 14:00* | *Lunch 12:30 - 14:00* | 13:00 | *Lunch 12:30 - 14:00* | |
| | 14:00 - 15:40 | 14:00 - 15:40 | Tour & Banket | 14:00 - 15:40 | |
| | Processing & Applications | Processing & Applications | | Processing & Applications | |
| | *Coffee Break* | *Coffee Break* | | *Coffee Break* | |
| | 16:10 - 17:50 | 16:10 - 18:00 | | 16:10 - 18:00 | |
| | Fundamental | Alloy Properties | | Fundamental | |
| 17:00 | | | | | |
| Registration | | | | | |
| 18:00 | | | | | |
| Welcome Party | | | | | |

# PROGRAMME

## Sunday, June 11

| Registration & Welcome party at the Mercure Hotel | | | | pages |
|---|---|---|---|---|
| From 17:00 | *Registration* | From 18:00 | *Welcome Party* | |

## Monday, June 12

| Session 1 - Processing & Applications | | | | *Chairman: Volker Guether* | |
|---|---|---|---|---|---|
| 8:30-8:40 | *Welcome* | | | | |
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| 9:20-9:50 | *Invited* | Pierre | Sallot | *γ-TiAl alloys for aeronautical applications: a status of their industrial implementation, development opportunities and* | 22 |
| 9:50-10:10 | *Oral* | Masao | Takeyama | *Revolutionary Approach to Develop Novel TiAl Alloys for LPT Blades under SIP Program in Japan* | 23 |
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| Session 2 - Additive Manufacturing | | | | *Chairman: Marc Thomas* | |
|---|---|---|---|---|---|
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| 11:10-11:30 | *Oral* | Florian | Pyczak | *Insight into Additive Manufacturing by combining in situ diffraction and modelling* | 25 |
| 11:30-11:50 | *Oral* | Marcus | Rackel | *TiAl powder based Composite Extrusion Modeling* | 26 |
| 11:50-12:10 | *Oral* | Yoshihiko | Nagata | *Investigation of alloy composition and identification of process challenges for additively manufactured gamma-* | 27 |
| | | | ***Lunch: 12:30-14:00*** | | |

| Session 3 - Processing & Applications | | | | *Chairman: Wilfried Smarsly* | |
|---|---|---|---|---|---|
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| 14:30-15:00 | *Invited* | Serjejs | Spitans | *FastCast - levitation melting & high-quality TiAl casting up to 600 g* | 29 |
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| | | | ***Break: 15:40-16:10*** | | |

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| 17:30-17:50 | *Oral* | Frank | Stein | *Stability, Composition Range, and Phase Equilibria of the Nb-stabilized, TiAl-based phases ωo and O* | 35 |

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| **9:00-9:30** | ***Invited*** | **Maria** | **Nó** | *High-Temperature Microstructure Evolution of an Advanced Intermetallic Nano-Lamellar γ-TiAl Alloy* | **37** |
| **9:30-9:50** | ***Oral*** | **Michael** | **Musi** | *The effect of zirconium on phase stabilities and microstructure in the Ti-(42-46 at.%)Al alloy system* | **38** |
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| **Session 6 - Additive Manufacturing** | | | | ***Chairman: Kui Liu*** | |
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| **10:50-11:20** | ***Invited*** | **Karin** | **Ratschbacher** | *Production of intermetallic, spherical powders* | **40** |
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| **14:30-15:00** | ***Invited*** | **Akira** | **Fukushima** | *Development of TiAl turbine blade with metal injection molding process* | **42** |
| **15:00-15:20** | ***Oral*** | **Arnaud** | **Fregeac** | *FAST/SPS: NEW industrial post-process for full densification of 3D TiAl complex shape from additive manufacturing* | **43** |
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| **17:00-17:20** | ***Oral*** | **Alexander** | **Donchev** | *High Temperature Oxidation of γ- TiAl Alloys: Effect of Zr* | **47** |
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| **15:00-15:20** | ***Oral*** | **Kui** | **Lui** | *Cost-effective Vacuum induction melting of gamma TiAl and NiTi alloy* | **64** |
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| **11:40-12:00** | **Concluding remarks** | | | | |
| ***Buffet: 12:00-13:30 & Departure*** | | | | | |

22 $2d^2 4s^2$

# Ti

Titanium

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# Al

Aluminium

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# IWTA2023

7th International Workshop on Titanium Aluminides

June 11-16, 2023, Toulouse, France

# Table of contents

# Welcome message

Dear colleagues and friends,

On behalf of the International and Local Organizing Committees of the IWTA-2023, I am very pleased to welcome you all to Toulouse.

Building on the resounding success of IWTA-2016 held in Tokyo, Japan, a new workshop planned and organized by Bernard Bewlay and General Electric was scheduled to take place in the US in 2020. Unfortunately, because of the Covid pandemic, this event, as well as a second proposal in 2021, were cancelled. In May 2022, the international committee requested the Toulouse group to host a workshop in 2023. Guy Molénat, Jean-Philippe Monchoux, Muriel Hantcherli, and I agreed to take up the challenge. We have put in a great deal of effort in the last year, and our reward today, is your presence in Toulouse, coming from France, Europe, Asia, and America; we are now close to one hundred in strength. This would not have been possible without the solid support of the international committee, with whom we met once a month via a video conference that took place spanning early hours in the US, late evening in Asia, and coinciding with the afternoon siesta time in Europe. To this effort, my thanks go to Guether Volker, Helmut Clemens, Masao Takeyama, Bernard Bewlay, Rui Yang, John Lewandowsk and Hirotoyo Nakashima.

**IWTA Workshops** serve to provide a suitable platform for researchers and professionals working both in academia and in industries worldwide, to discuss and exchange views on the recent progress and future perspectives on the properties, microstructure, technologies, processing and development of Titanium Aluminides. IWTA Workshops are unique in that they bring engineers and researchers together for a week of intense discussions on TiAl alloys in a relaxed setting. Another important aim of IWTA is to welcome our new and young colleagues into the community.

**IWTA-2023** is the seventh in the well-known series of meetings that have marked the history of TiAl alloys. Let us remember that General Electric revealed the use of TiAl blades in its aviation engines in Bamberg in 2006. Many advances have resulted from our exchanges at the various workshops. IWTA-2023 will undoubtedly make its mark in the history of TiAl alloys, within the context of the ongoing energy transition and the associated breakthroughs in terrestrial and aeronautical engines. These breakthroughs are anticipated to create an increased demand for materials that are both lightweight and capable of high performance at high temperatures.

We would like to express our gratitude to **our sponsors and partners** who have helped to organize IWTA-2023, which was made possible by the active participation of the members of the international committee. Their logos are displayed on the back cover of this book. To them, we say Thank you very much!!!

During this week, we offer you a balanced program that includes keynote lectures, invited talks, oral presentations, and poster sessions. With a visit to Airbus and dinner at a vineyard, we look forward to an exciting Wednesday afternoon and evening. Don't miss these!!!

Finally, we would like to thank all the speakers, presenters, and participants, whose participation is undoubtedly key to the success of IWTA-2023.

Enjoy the workshop and enjoy your stay in Toulouse,

Alain Couret

# Organising Committee

**Bernard Bewlay**, GE Global Research, USA
**Helmut Clemens**, Montanuniversität Leoben, Austria
**Alain Couret**, CEMES-CNRS, France
**Volker Güther**, GfE Metalle und Materialien GmbH, Germany
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**Masao Takeyama**, Tokyo Institute of Technology, Japan
**Rui Yang**, Institute of Metal Research, China

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**Muriel Hantcherli**
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**Jean-Philippe Monchoux**
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CEMES
CENTRE D'ÉLABORATION DE MATÉRIAUX ET D'ÉTUDES STRUCTURALES

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# General information

## Venue

**Hotel Mercure Toulouse Centre Compans**

| | |
|---|---|
| Boulevard Lascrosses | GPS: 43.610705, 1.434822 |
| 8 Esplanade Compans Caferelli | +33 561 11 09 09 |
| 31000 Toulouse, France | H1585@accor.com |

Metro: Compans-Cafarelli station, line B (station right in front of the hotel)

From the Toulouse-Blagnac airport:
Aerobus every 20 minutes, 15-minute drive, 9 €, stop right in front of the hotel | 8 km by car

From the Matabiau main railway station:
3 metro stations away, one change, 15 minutes | 2.2 km by car

# Social program for Wednesday afternoon, June 14

1pm: Departure by bus from the Mercure hotel

1.30pm: arrival at Aeroscopia and identity check

2.00pm-3.30pm: Group A (see letter indicated on the badge) visits the Airbus assembly line

2.30pm-4pm: Group B visits the Airbus assembly line

Waiting for and returning from the Airbus visit: each group visits the Aeroscopia museum (unaccompanied tour)

5pm: departure for Château Lastours

6pm-7pm: visit of the vineyards, the cellars and wine tasting in three groups (see number 1, 2 or 3 indicated on the badge)

7pm: diner

## Visit of the Airbus assembly line

AIRBUS

https://www.manatour.fr/en/airbus

Meeting address:
Aeroscopia museum, see below

## Visit of the Aeroscopia museum


aeroscopia

www.aeroscopia.fr

Allée André Turcat, 31700 Blagnac
Tram : Line 1 / Beauzelle Aeroscopia stop

## Diner in the vineyard of Château Latours

CHATEAU LASTOURS
www.chateau-lastours.com

81 310 Lisle-sur-Tarn (60 km from the hotel)

# Abstracts

## Keynotes,

## Invited Talks & Oral Presentations

***(in chronological order – see programme)***

# The Science and Technology of TiAl Alloys For Commercial Aircraft Engines

B. P. Bewlay[1] and M.J. Weimer

[a] *GE Research, One Research Circle, Niskayuna, NY, 12309, United States*

**Abstract**

The present presentation will discuss development of the science and technology of Titanium aluminide (TiAl)-based alloys that has culminated in their use in commercial aircraft engines. Titanium aluminide (TiAl)-based alloys have been developed for high performance applications in the aerospace industry. Within the last decade, TiAl alloys have been introduced into service in a range of commercial aircraft engines as a new lightweight low pressure turbine (LPT) blade material class. The TiAl alloy 48-2-2 is extensively used in GEnx™, LEAP-X™, and GE9X™ engines. In addition, a new β-stabilized TiAl alloy (TNM) has been introduced as a LPT material for PW1100G™ engines. The mechanical properties of TiAl alloys are sensitive to their microstructure, which is controlled by the alloy chemistry and processing schemes. This review will focus on room and elevated temperature mechanical and environmental properties exhibited by second and third generation TiAl alloys. In terms of component manufacturing, conventional casting, near-net-shape casting, high-temperature forging, and additive manufacturing approaches will be discussed.

[1] Corresponding Author. Tel. +1 (518) 387-6121;. *E-mail address*: bewlay@ge.com.

# γ-TiAl alloys for aeronautical applications: a status of their industrial implementation, development opportunities and challenges

Pierre SALLOT[1*]

*Safran Tech, Materials and Processes, Rue des Jeunes Bois, Châteaufort, 78114 Magny-Les-Hameaux, France*

*corresponding author*

Titanium aluminide has been for years identified as a solution for weight reduction in the LPT (Low Pressure Turbine) of a turbo-engine. The improved specific mechanical properties of these intermetallic alloys, when compared to Ni-based superalloys, have pushed Safran and other major aero-turbine producers to introduce them in their newest generations of turbines. Today, TiAl LPT blades are a reality for mass production.

In the present study, several processing routes to produce complex TiAl parts are investigated, from isothermal forging to powder metallurgy, and their impact on alloy properties compared. It will be used as a basis for discussion on material developments, but it will emphasize as well limitations to overcome for next generation applications. To support it, metallurgical characterizations as well as specific mechanical trials will be used to exemplify these points, with a focus on oxidation resistance.

A specific part of the presentation will be dedicated to the introduction of a new Powder Metallurgy route using Spark Plasma Sintering and its application to industrial parts. Material microstructures and properties will be compared to other processes, but also its energy consumption will be discussed.

In term of perspectives, remaining challenges in the development of these alloys will be emphasized and discussed.

# Revolutionary Approach to Develop Novel TiAl Alloys for LPT Blades under SIP Program in Japan

Masao Takeyama*

*Department of Materials Science & Engineering, School of Materials and Chemical Technology, Tokyo Institute of Technology, Tokyo, Japan*

*corresponding author

In the past decade, two consecutive five-year national projects of "*Structural Materials for Innovation* ($SM^4I$)" (2014-2018) and "*Materials Integration for Revolutionary Design System of Structural Materials*" (2018-2022), mainly focusing on jet engine parts, were conducted in Japan under the *Cross-ministerial Strategic Innovation Promotion Program* (SIP). The R & D of TiAl blades were one of the main subjects in the projects, and I was committed to them as a technical leader and took responsibility for the novel alloy design of titanium aluminides for LPT blades in jet engines, in collaboration with industries and other universities, based on our newly integrated design principles. In the first project, the focus was placed on the innovative wrought/cast TiAl alloys, whereas the powder processed alloy development, such as MIM (Metal Injection Molding) and AM (Additive Manufacturing), was focused in the second program, in terms of the integrated inverse design approach. In both programs, we have successfully developed novel alloys superior to the currently existing alloys.

In this talk, although detailed approaches and process developments will be emphasized by the individual presentations from academia and industries in this workshop, I will introduce the whole images of industry-academia collaboration team roles and several important points for the revolutionary alloy developments. Regardless of the different fabrication processes of cast, wrought, MIM and AM, an underlying principle is in the reliable multi-component phase diagrams, especially oxygen. You must realize how oxygen level is so sensitive to change the phase relationship among the phases concerned in the developed alloys, resulting in significant change in microstructure even the major alloy elements as well as the heat treatment routes are the same. Our integrated computational inverse alloy design system consisting of two modules of MPM (mechanical-property prediction module) and MDM (Microstructure Design module) will also be touched, in addition to the importance of introducing bcc β-Ti phase in the microstructure for mechanical property improvements.

A part of this study has been carried out under the research of SIP in JST (Japan Science and Technology Agency).

# Microstructure Design of Titanium Aluminides by Additive Manufacturing

Christoph Leyens[1,2*], Juliane Moritz[1,2], Axel Marquardt[1,2], Mirko Teschke[3], and Frank Walther[3]

[1]*Technische Universität Dresden, Institute of Materials Science (IfWW), Helmholtzstr. 7, 01069 Dresden, Germany*
[2]*Fraunhofer Institute for Material and Beam Technology IWS, Winterbergstr. 28, 01277 Dresden, Germany*
[3]*TU Dortmund University, Department of Materials Test Engineering (WPT), Baroper Str. 303, 44227 Dortmund, Germany*

*corresponding author*

Titanium aluminides, including the TNM-B1 alloy (Ti-43.5Al-4Nb-1Mo-0.1B), have been attracting continuous research interest due to their potential for lightweight high-temperature applications. Nevertheless, conventional processing of titanium aluminide alloys remains challenging as a consequence of their pronounced brittleness. Additive manufacturing can be a viable alternative for the near-net-shape production of titanium aluminide components. Electron beam powder bed fusion (PBF-EB) is particularly suitable for this purpose, as the high process temperatures above the brittle-to-ductile transition temperature allow crack-free processing. However, due to the processing under vacuum and the different vapor pressures of the alloying elements, aluminum tends to evaporate from the melt. The extent of the occurring aluminum loss is closely related to the selected process parameters. Pronounced aluminum evaporation does not only have an impact on the overall chemical composition of the component, but can also affect phase transformation temperatures and microstructure evolution during heat treatment.

While these evaporation effects are undesirable in many cases, the aluminum content can also be specifically adjusted by an adequate selection of the melting parameters in PBF-EB. By controlling the aluminum content in different areas of a component, tailored microstructures can be achieved via subsequent two-step heat treatments (see Figure 1) [1]. For example, this might enable the adjustment of a fully lamellar microstructure with high creep resistance in the blade and a more ductile nearly lamellar γ-microstructure in the blade root within the same turbine blade component [2]. This contribution will provide a detailed insight into the correlation between the PBF-EB process conditions and the resulting microstructures with respect to a targeted microstructure design. Furthermore, challenges regarding microstructural homogeneity originating from the layer-wise manufacturing as well as ways to overcome these issues will be addressed.

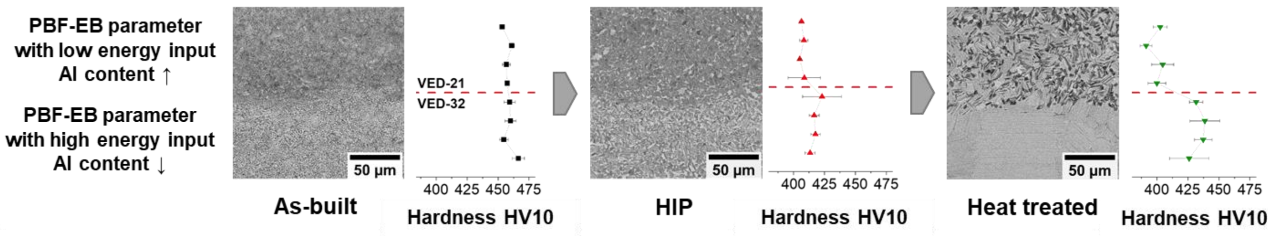


Figure 1: Local adjustment of microstructures and properties within the same component via PBF-EB process parameter variation and subsequent heat treatment [1]

References

[1] J. Moritz, M. Teschke, A. Marquardt, L. Stepien, E. López, F. Brueckner, F. Walther, C. Leyens, Locally Adapted Microstructures in an Additively Manufactured Titanium Aluminide Alloy Through Process Parameter Variation and Heat Treatment, Adv Eng Mater. (2022) 2200917. https://doi.org/10.1002/adem.202200917.

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# Insight into Additive Manufacturing by combining in situ diffraction and modelling

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Additive manufacturing (AM) is an important up-coming processing route for TiAl alloys. It provides inherent advantages like its near net shape processing capability and the good chemical homogeneity of the parts produced by powder based AM methods. Due to the low ductility of TiAl alloys, compared to conventional alloys, the high heating and cooling rates experienced during AM can cause cracking and damage. The complex phase diagram of TiAl alloys exacerbates this problem. It shows a number of phase transformations between melting point and room temperature, which may be associated with volume change or volume misfits between phases. At lower temperatures, TiAl parts are too brittle to withstand the stresses caused by those volume changes and misfits and will fail during AM, making pre-heating essential.

In this contribution, the results of the characterization by high-energy X-ray diffraction of TiAl alloys under AM conditions are presented. Directly after solidification, the phase constitution of TiAl resembles very closely the phase constitution directly at the liquid-solid transition. The final microstructure and phase constitution forms in the layers below the surface. These experience a cyclic heat treatment due to the melting and solidification of the on top powder layer. The maximum temperature of this thermal cycle decreases with increasing distance from the top layer. From the development of lattice constants and phase fractions, it is possible to reconstruct the internal processes in the material during this complex heat treatment scheme. However, the phase constitution and microstructures are far away from equilibrium. Only by a combination of direct measurements and modeling one can unveil the situation in the material and acquire the knowledge necessary to optimize the process or adapt the material to the process.

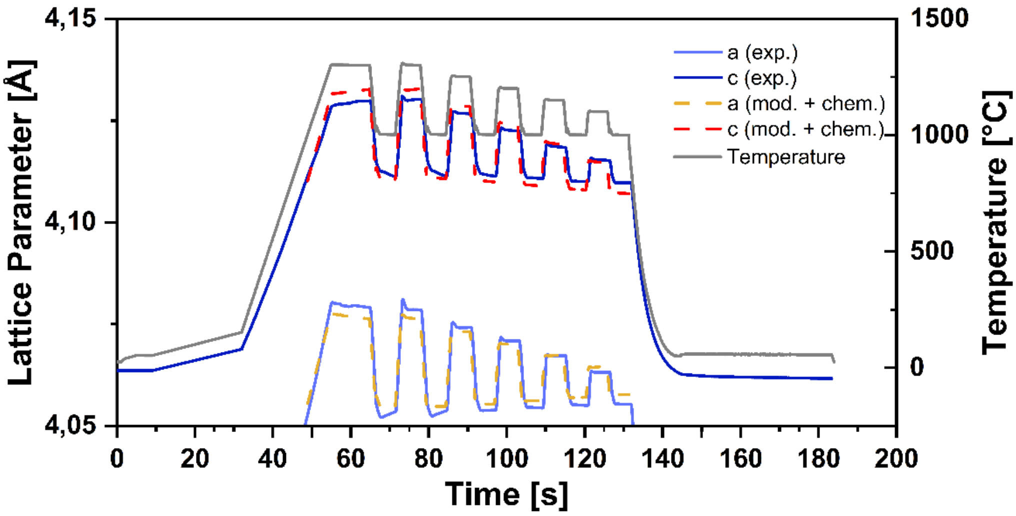


Figure 1: Temperature profile of a cyclic heat treatment to simulate AM (grey solid line) and measured lattice constants of γ phase (solid blue lines) in comparison with model predictions (dashed red lines)

# TiAl powder based Composite Extrusion Modeling

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In this paper, a first attempt was made to process TiAl alloys using the *C*omposite *E*xtrusion *M*odeling (CEM) process. The achieved properties are compared to those of material made using a standard *M*etal *I*njection *M*oulding (MIM) process route.
One advantage of the CEM process is that the existing technical infrastructure of the well established MIM processing route can be used. The CEM process combines this with the advantages of additive manufacturing processes such as high geometrical degree of freedom and optimised material use. Thus material and machine costs can be reduced. Furthermore, process induced residual stresses and their negative influence on components properties are negligible compared to powder bed-based additive manufacturing processes such as laser or electron beam fusion.
The first step in the process was the production of spherical TNM alloy powder (Ti-43.5Al-4Nb-1Mo-0.1B, in at.%) at Helmholtz-Zentrum Hereon using the *E*lectrode *I*nduction Melting Inert *G*as *A*tomization process. After sieving, the powder fraction < 25 µm was used for the preparation of a paraffin based binder feedstock. Thereafter, the feedstock was granulated to 1–3 mm diameter particles and dog bone shaped samples were produced, using an AIM3D ExAM 255 printer for CEM processing and an Arburg 370A injection moulding machine for MIM processing. Subsequently, all the samples produced were thermochemically debinded and then sintered in an inert argon gas atmosphere. The resulting microstructures and phases were analysed using SEM and EBSD, as well as high energy X-ray diffraction.
For both the CEM and MIM production techniques, tensile tests were performed at room temperature. In addition, compression creep tests were carried out at two different temperatures, 700 °C and 800 °C, under 150 MPa load.

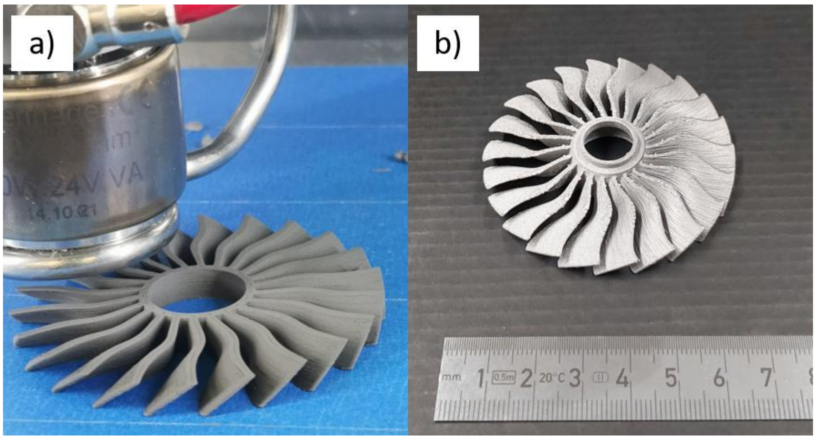


***Figure 1:*** a) CEM printing of a TNM demonstrator part. b) Finished demonstrator part made out of TNM alloy after printing, thermochemical debinding and sintering.

# Investigation of alloy composition and identification of process challenges for additively manufactured gamma-titanium aluminides

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Gamma titanium aluminides (γ-TiAl) has been expected to replace existing Ni-based superalloys (Alloy 718) due to its low density and superior specific strength at high temperature. In an effort to make γ-TiAl alloys more widely applicable as structural materials, development projects have been vigorously pursued over the past 30 years in Europe, the United States, and Japan, mainly by the aeroengine industry, the materials industry, and research institutes. In particular, the project conducted by GE in 1995 succeeded in the ground test of the engine using TiAl alloy for LPT blades, which gave a great impetus to the practical use of TiAl. IHI has accumulated knowledge of precision casting technology to ensure a practical level of quality.

Since 2000, there has been a growing demand from the automotive industry for turbocharger blades made of TiAl alloy instead of Alloy 713C, and IHI has developed a fully lamella-TiAl alloy with improved creep strength in the temperature range of 1175K to 1275K. Utilizing these advanced technologies and building a supply system, IHI began mass production in 2004 and has produced well over 160,000 units.

In order to expand the application of TiAl beyond aircraft engine blades and turbocharger airfoils, we believe that a new manufacturing method, especially additive manufacturing (AM), will be effective. In this presentation, we will discuss the alloy design guideline based on the reintroduction of elements whose addition was restricted by conventional production methods due to segregation problems. The degradation phenomenon of TiAl airfoils used for more than 10 years as turbochargers for vehicles will also be discussed. Furthermore, in the AM process development, we will focus on the electron beam melting (EBM) method and extract engineering issues that affect the process and quality stability. For example, we will investigate the smoke phenomenon characterizing the dependence of each powder particles variable, and deepen the discussion and sharing of results with the participants to work towards a solution.

# Superior Productivity and Properties of Newly Designed Wrought and Cast TiAl Alloys for Jet Engine Blade Components

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TiAl alloys are light-weight heat resistance material and have been applied to low pressure turbine blades of civil aircraft engines represented by GEnx, LEAP and GE9X. The possibility of TiAl alloys spread more in comparison with these conventional alloys. In this study, TiAl alloys for forging and casting were developed according to established alloy design method. These alloys, developed for next-generation jet-engine, have a good balance of strength, ductility at room temperature and productivity. Tensile, creep and high cycle fatigue tests at elevated temperature were conducted for developed alloys and their results were superior to conventional TiAl alloys, partly due to the effect of adjusting heat treatment conditions. Also, it was demonstrated that the developed alloys can be processed into low pressure turbine blade shapes without harmful defects by casting or hot die forging using conventional techniques and equipment (not isothermal forging). These results will be able to achieve lower cost and expand the jet-engine application range of TiAl alloys. The details of the processing route and performances on the developed wrought and cast alloys will be presented.

This work was supported by Council for Science, Technology and Innovation(CSTI), Cross-ministerial Strategic Innovation Promotion Program (SIP), “Structural Materials for Innovation”(Funding agency : JST).

# *FastCast* - levitation melting & high-quality TiAl casting up to 600 g

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Ti-alloys are highly reactive in liquid phase and must be melted in water-cooled copper crucibles. A thin skull layer that forms at the cold wall prevents contamination, however, results in huge thermal losses, low melt superheat and "cold-runner" defects in castings. Because of that, complex geometry turbocharger wheels and blades from hard-to-cast γ-TiAl are mostly machined.

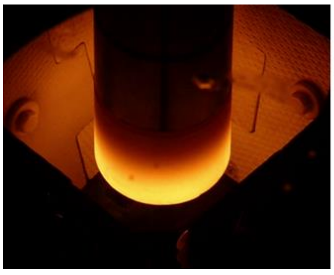
Levitation melting, known since 1920's, utilizes EM-field to confine metallic samples in a contact-free condition and has evident advantages in superior purity, no material loss in the skull and significantly higher superheat. However, in conventional axisymmetric implementation only small samples up to 50 g can be levitated and investment casting scale-up needs remain unsatisfied.

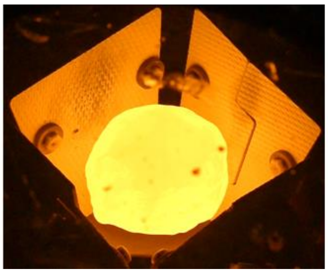
A novel levitation melting method that utilizes two horizontal and orthogonal EM-fields of different frequencies has been developed to solve the scale-up issue. Numerical modelling has been used to design a new melting unit and to integrate it in a pilot investment casting furnace called *FastCast*, capable of contact-free melting and single-shot casting of Ti- and Ni-alloys up to 600 g.

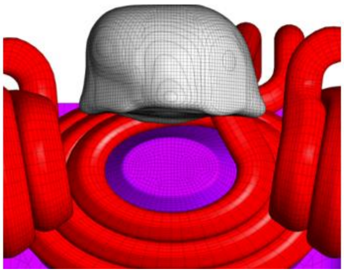
The demonstrator furnace is capable of 10 automated consecutive castings in vacuum or Argon atmosphere. Melting starts as the lower end of the vertically oriented electrode is immersed in the levitation melting unit. EM fields (installed power 180 kW) rapidly melt up to 600 g of material from the tip of the electrode and simultaneously confine the liquid metal in a contact-free condition. The electrode is moved up and detached levitated melt is rapidly superheated up to adjustable 250 C. Then EM field below the melt is reduced and the melt falls down in the awaiting preheated mold. Synchronized with the free-falling melt position, the mold is accelerated vertically down following the pre-programmed recipe to catch the melt and full-stop at the bottom of the casting chamber. Such mold movement reduces relative velocity between the falling melt and the mold at the moment of the contact, avoids splashing (>98% of material lands in the mould) and ensures smooth filling. On top of that, the mold table allows for the centrifugal casting up to 800 rpm. As the casting is accomplished, the manipulator arm stores the filled mold and takes the next preheated empty mold for the next cycle. Short melting time (<100 s) per casting is a prerequisite for high productivity.

Ti-48-2-2 turbocharger wheel castings with 0.3 mm blade thickness revealed good surface quality, high level of reproducibility and absence of cold runners even at low mould temperatures. Defect-free filling of thin and complex sections has been approved by CT-scans.

The *FastCast* pilot system is available at ALD for customer visits and trials.







# Development of TiAl alloys for gas turbine engines

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Keywords: TiAl, gas turbine blade, high-temperature strength

Research on developing new TiAl alloys for high temperature applications is introduced. At KIMS, we have developed new TiAl alloys which have excellent room temperature and high temperature properties. Especially, the new alloy showed excellent oxidation resistance in the temperature range from 900 to 1000°C by forming stable $Al_2O_3$ oxidation layer. Process developement of casting, forging as well as 3d printing on the newly developed KIMS alloys was introduced. Especially, small size turbine wheel and blade were manufactured be centrifugal casting process. The results from the testing and validation of TiAl blade were shown that KIMS alloy can be used as a turbine blade above 900°C. In addition, we proposed some underlying mechanism of high temperature strength of KIMS alloy from TEM and SEM observations. Finally, the operation test of micro gas turbine is now under examination to confirm the possibility of the application of the new alloy in the gas turbune engine.

# Microstructure and Mechanical Properties of Reactively Sintered TiAl based Alloys

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Microstructure and mechanical properties of γ-TiAl based alloys prepared by reactive sintering of pure metal powders have been investigated. Metal powders of Ti, Al and M (M: transition metal elements) were mixed to obtain a composition of Ti-45Al and Ti-45Al-4M (at. %) . Mixed powder was then heated to a temperature range from 1073 K to 1573 K by spark plasma sintering to produce sintered compacts with a diameter of 30 mm and a height of 7 mm. The mechanical properties of the alloys were evaluated by three-point bending tests. The density of sintered Ti-45Al compacts increased from 3.3 to 3.9 g/cm$^3$ as the sintering temperature increased from 1073 K to 1573 K. That of Ti-45Al-4M alloys sintered at 1573 K shows approximately 4.0 g/cm$^3$, which is considered to be close to the theoretical density. The microstructure of Ti-45Al alloy consists of $\alpha_2$-$Ti_3Al$/γ lamellar and γ grains. That of Ti-45Al-4M alloys is composed of $\alpha_2$-$Ti_3Al$/γ lamellar grains, γ grains and β grains. The bending strength of Ti-45Al alloy is about 700 MPa at room temperature and 800 MPa at 1273 K. Details of reaction sintering process of pure metal powders and microstructural formation will be discussed in presentation. This work was supported by the project, “Subsidy for Regional University / Regional Industry Creation” of the Cabinet Office, Government of Japan: "Creation of a Global Base for Advanced Metals - Next Generation TATARA Project-".

# Modelling of phase transformations in TiAl-based alloys

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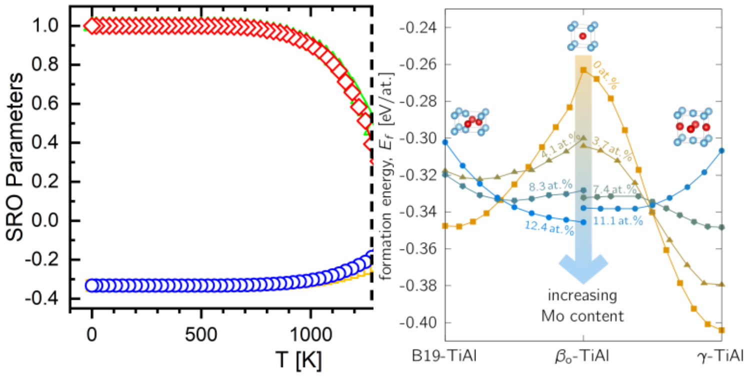
In this contribution, I will review our recent activities in applying Density Functional Theory (DFT) to phase stability problems and related phase transformations between various phases in a model TiAl+Mo system. It is experimentally interesting to include the bcc phase at high temperatures, which is, however, mechanically unstable in the binary stoichiometric TiAl scenario [1]. Alloying Mo, this phase can be stabilized concerning both phases, the hexagonal based on the $\alpha_2$-$Ti_3Al$ structure [2] and the distorted tetragonal γ-TiAl phase [3]. The DFT calculated potential energy surfaces suggest that these martensitic transformations are mostly barrier-less.

The chemical disorder was suggested as an effective stabilizing mechanism for the β-TiAl+Mo system [1]. In comparison to the ordered phases mentioned above, the chemical disorder significantly reduces the transformation driving force for both scenarios, β⟶α [2] as well as $\beta \rightarrow \gamma_{dis}$ phases [3]. Moreover, small energy barriers appear for low Mo concentrations, thus opening a possibility for metastable phases.

In the final part of my talk, I will present strategies for predicting ordering transformations [4]. The calculated ordering temperature $T_{ord}$ will be presented again for the TiAl+Mo model system for bcc $\beta \rightarrow \beta_o$ and hcp $\alpha \rightarrow \alpha_2$ (B19) transformations.




(a) (b)

(a) Example of $\alpha_2$(B19)↔$\beta_o$↔γ transformation landscape for TiAl+Mo [2, 3, 5]. (b) Ordering $\alpha_2$(B19)↔α for binary TiAl [4].

# Computational Alloy Design of TiAl alloys applicable to Powder Processes

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A microstructure design module for multi-component TiAl alloys has been developed as part of an integrated computational inverse problem approach. This module outputs the alloy composition and heat treatment conditions that will achieve the input of the volume fraction of the microstructural constituents to meet satisfy the required properties, and thus provides for faster and lower-cost alloy design. This is achieved by combining the thermodynamic calculation and kinetic modeling using the CALPHAD framework with ab-initio calculations.

With particular application to powder processes where oxygen contamination is a serious problem, the effect of oxygen in solution on the phase equilibria and phase transformation kinetics was evaluated using an experimental forward problem approach. For the phase equilibria, soft X-ray emission spectroscopy (SXES) combined with EPMA has revealed a strong α-Ti (or $\alpha_2$-$Ti_3Al$) stabilizing effect of oxygen and the resulting shift of the β-Ti + α + γ-TiAl three-phase tie-triangles towards the higher M concentration side of the Ti-Al-M-O (M: transient metal elements) systems with the oxygen addition. For the kinetics, oxygen is found to retard both nucleation and growth of cells in the $\alpha + \gamma \rightarrow \beta + \gamma$ cellular reaction. A set of databases has been constructed to reproduce these experimentally determined oxygen effects on the basis of thermodynamic descriptions and classical nucleation and growth theory. The combined computational design approach is demonstrated by designing unique high performance alloys applicable for metal injection molding (MIM) and powder bed electron beam melting processes.

This study was supported by the Cross-Ministerial Strategic Innovation Promotion Program (SIP) “Structural Materials for Innovation” from the Japan Science and Technology Agency (JST).

# First-principles study of transition-metal β stabilizers in Ti-Al-O

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In microstructures of Ti-Al alloys, the bcc β phase plays important roles as subphases in exhibiting mechanical properties to prevent plastic deformations at high temperatures. In designing microstructures, additive metallic elements at substitutional atomic sites can be effective, e.g., Cr and Nb as β stabilizers. On the other hand, oxygen atoms are included at interstitial sites in powder-processed Ti-Al alloys. These interstitial oxygen atoms also affect phase equilibria modulating microstructures that have been reported for Ti-Al-V and Ti-Al-Cr [1]. Thus, interplays between interstitial oxygen and substitutional metallic elements should be considered in understanding phase equilibria precisely. Even though knowledge of the systematic change in a transition-metal element *M* will be helpful for deeper understanding, neither experimental nor theoretical studies have been reported from this aspect. Particularly, the change in the effectiveness as a β stabilizer is one of primary interests.

In this study, the structural stability of the β phase relative to the hcp α phase is examined from the viewpoint of electron theory. First-principles calculations are performed for random alloys by the supercell approach containing 64 metallic atoms on the basis of density functional theory within the generalized gradient approximation. One transition-metal *M* atom are substituted to a Ti site and one oxygen atoms are included into an octahedral substitutional site. Since the β phase is unstable without help of anharmonic lattice vibrations at finite temperatures, the structural optimization for the β phase is partial: i.e., impurity atoms and their surrounding host atoms as well as the lattice constant are optimized. Differences between the full and partial structural relaxations are confirmed to be sufficiently small for the α phase.

First, we identify that oxygen is most stable at octahedral interstitial sites consisting of 6 Ti atoms in ternary Ti-Al-O alloys. The formation energy for the oxygen interstitial is examined using total energies of ternary Ti-Al-*M* and quaternary Ti-Al-*M*-O alloys, where *M* is examined for 3*d* and 4*d* transition-metal elements. The formation energy is lower for early transition metals compared with late transition metals, which can be partly explained by high ionicity of early transition metals. Even though this tendency is common for both α and β phases, differences between the phases are also seen. Thus, we compare total energies of the α and β phases. As shown in Fig., oxygen hinders Nb from being a β stabilizer significantly. Its detailed mechanism will be clarified in the presentation.

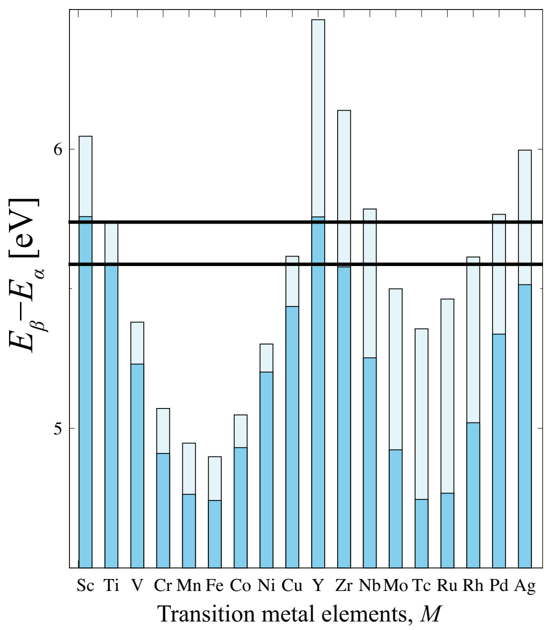


**Fig.** Energy differences per *M* atom between the α and β phases for $Ti_{0.609}Al_{0.375}M_{0.016}$ (dark bars) and $Ti_{0.600}Al_{0.369}M_{0.015}O_{0.015}$ (light bars behind dark ones). Horizontal lines represent values for $Ti_{0.625}Al_{0.375}$ and $Ti_{0.615}Al_{0.369}O_{0.015}$.

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# Stability, Composition Range, and Phase Equilibria of the Nb-stabilized, TiAl-based phases $\omega_o$ and O

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Nb is the most important alloying element in TiAl-based alloys. Depending on alloy composition and thermal history, Nb-containing TiAl-based alloys may contain the $\omega_o$ and O ternary phases. These ternary compounds significantly affect the microstructure and mechanical properties and, therefore, a detailed knowledge of their composition and stability ranges, as well as of the phase equilibria and microstructures they form with the Ti-Al phases α-Ti, $\alpha_2$ ($Ti_3Al$), $\beta/\beta_o$-Ti, and γ (TiAl) is required.

A series of ternary Ti-Al-Nb alloys containing 17.5 to 45 at.% Al and up to 25 at.% Nb as well as some solid-solid and solid-liquid diffusion couples were prepared and annealed between 700 and 1000°C for up to 1500 h. The resulting microstructures and the types and compositions of phases were analyzed by scanning electron microscopy (SEM), electron probe microanalyses (EPMA), and high-energy X-ray diffraction (HEXRD) yielding isothermal sections at 700, 800, 900, and 1000°C [1,2]. In addition, the phase transformations involving the decomposition/formation of the $\omega_o$ and O phase were studied by differential thermal analysis (DTA) and *in situ* HEXRD [3]. The slow kinetics of these solid state phase transformations was analyzed by DTA experiments with varying heating rates allowing the determination of the true equilibrium transformation temperatures. The dependence of the measured transformation temperatures on the heating rate varies systematically with the complexity of the transformations. Both, $\omega_o$ and O phase are stable to above 900°C. For the $\omega_o$ phase, a new formula $(Ti,Nb)_2Al$ is suggested based on the measured composition range and its crystal structure type. The results also indicate that the phase field of the $\omega_o$ phase is split into two parts at 900°C because of the growing phase field of the ordered $\beta_o$ phase.

The research was carried out within the scope of the European Union Clean Sky 2 project ADVANCE, which has received funding from the Horizon 2020 research and innovation programme under grant agreement No 820647.

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**Formation and evolution of defects that interrupt lamellar microstructure in cast TiAl alloys**

Renci Liu, Ruxin Cao, Zhichun Zhou, Yuyou Cui, Rui Yang

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Cast gamma TiAl alloys normally contain lamellar microstructure that ensures excellent resistance to fatigue crack growth and to high temperature creep of aeroengine components such as low pressure turbine blades. Extra alloying additions are sometimes necessary to improve mechanical and service performance but, if not controlled properly, they may cause undesirable phases that may interrupt or even destroy the lamellar microstructure. Typical examples include ribbon-shaped boride in boron containing gamma alloys that may cut through a lamellar grains and thus provide a short cut for crack propagation, and formation of orthorhombic phase from the ordered hexagonal phase that may disintegrate the lamellar microstructure in alloys that contains a significant amount of heavy transition metals. In this talk, such phenomena in cast 4522XD and TNM based alloys will be presented, and the conditions under which they appear will be analyzed.

# High-Temperature Microstructure Evolution of an Advanced Intermetallic Nano-Lamellar γ-TiAl Alloy

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In recent years, engineering intermetallic γ-TiAl based alloys were introduced in the low-pressure turbines of several aeronautic engines and nowadays thousands of blades are flying in commercial aircrafts.

To improve the present performances of these alloys, the creep resistance should be improved to allow an increase of the working service temperature, as well as a longer service life. Recently, a new generation of γ-TiAl, Ti-43.3Al-4.0Nb-1.0Mo-0.3C-0.3Si (at%), called $TNM^+$, was developed to improve the creep resistance by microalloying with C and Si [1] and developing a fully nano-lamellar microstructure. The relaxation processes associated with the diffusion of several atomic species were also studied [2]. The combination of very fine precipitates with the nano-lamellar structure makes this alloy exhibiting a high creep resistance [1]. However, to extend the creep resistance on temperature and on time, the stability of the microstructure should be guaranteed or, as in the case of superalloys, well mastered in what concerns to its evolution.

In the present work, a complete study of the microstructure evolution in this nano-lamellar γ-TiAl alloy was carried out by transmission electron microscopy in a FEI Titan-Cubed G3 300 kV aberration corrected on objective lens and Super-Twin EDX detector. The nucleation and growth of $\beta_o$ and ζ silicide precipitates were studied by TEM, HRTEM and HRSTEM-HAADF and their composition was quantitatively analysed by high-quality EDX maps. In addition, the orientation relationships between them and also with the hosting phases, $\alpha_2$ and γ, were determined. Simultaneously, the slow disintegration of the $\alpha_2$ phase when transforming into γ phase was also studied. Moreover, it was observed that the precipitates nucleate preferentially in the $\alpha_2$ phase slowing down the process of disintegration of $\alpha_2$ phase and its transformation into γ phase. The observed evolution of the microstructure is interpreted through the diffusion processes taking place during the precipitation and $\alpha_2$ disintegration, which were determined through internal friction measurements. Finally, the potential influence of the microstructure evolution on creep resistance is discussed.

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# The effect of zirconium on phase stabilities and microstructure in the Ti-(42-46 at.%)Al alloy system

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In order to seize new application areas for intermetallic γ-TiAl based alloys, an increased service temperature and improved mechanical properties are a necessity. Considering that these alloys consist mainly of γ-TiAl phase, strengthening of this phase is a crucial point to further advance this class of materials. A promising alloying element meeting the requirements for effective solid solution strengthening of the γ phase is Zr. While its beneficial effect on the mechanical properties is already known in the scientific community, a detailed study of its influence on the phase transformations in the relevant Al range is still missing. Consequently, the present work investigates model alloys based on the Ti-(42-46)Al-(2-4)Zr (at.%) alloy system regarding the influence of Zr on the microstructure and phase transitions. Microstructural characterization reveals a stabilization of the γ phase due to Zr in the microstructure, which consists predominately of lamellar $\alpha_2/\gamma$ colonies and a minor amount of globular γ phase after hot-isostatic pressing. The hardness of the material increases due to both solid solution strengthening and refinement of the colonies' lamellar spacing by Zr. The phase transformation behaviour and the related impact of Zr are investigated by differential scanning calorimetry (DSC) as well as high-energy X-ray diffraction (HEXRD) utilizing synchrotron radiation. Especially, two different types of in-situ HEXRD heating experiments are conducted to investigate, on the one hand, the solid-solid transformations below the material's solidus temperature, and, on the other hand, the liquid-solid transformations at higher temperatures. DSC and HEXRD experiments combined with complementary heat treatments clearly show that the Zr additions increase the thermal stability range of the γ phase, e.g. see Fig. 1a. Furthermore, phase transformations involving the liquid phase are drastically shifted towards lower temperatures when alloying with Zr. Thus, the addition of Zr effectively narrows the α phase field region as shown in Fig. 1b. Furthermore, the combination of X-ray diffraction and ab-initio calculations based on density functional theory (DFT) grants valuable insights into the effect of Zr and Al on the lattice parameters of the γ phase. More precisely, Zr is found to cause a strong reduction of the tetragonality of this phase due to an elongation of the shorter unit cell axis *a*, while the longer axis *c* remains mostly unaffected.

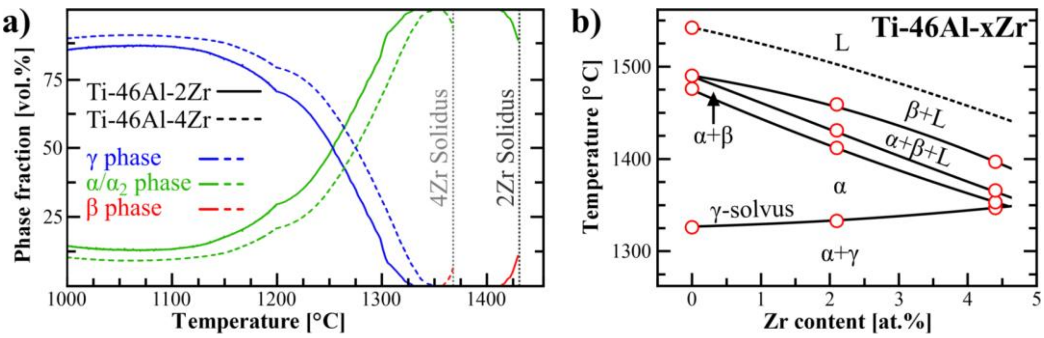


Figure 1. a) Phase fraction evolution of a Ti-46Al-2Zr and a Ti-46Al-4Zr (at.%) alloy; b) Quasi-binary Ti-46Al-xZr phase diagram derived from the results of HEXRD, DSC and heat treatments.

# Additive Manufacturing of Gamma Titanium Aluminides: A Review of Methods, Properties and Challenges

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Additive Manufacturing (AM) of metallic parts has shown a growing interest in the last decade, especially in the aerospace industry for weight saving, functional design, lower buy-to-fly ratio and rapid manufacturing with reduced lead time. Significant AM breakthroughs have already been reported for intermetallic alloys such as gamma titanium aluminides. Currently, γ-TiAl alloys have already been successfully processed via powder bed and powder feed technologies. In particular, electron beam powder bed fusion (EB-PBF) and direct energy deposition (DED) are those reported so far to successfully fabricate crack-free TiAl samples. EB-PBF is probably the most mature technology to produce sound TiAl components with a good geometrical precision for aerospace and energy generation applications. DED coupled with machining steps can become attractive for restoring γ-TiAl alloys parts. Instead of replacing the entire part, repair solutions using DED are more cost effective. Powder feed technology also offers interesting perspectives by enabling the fabrication of functionally graded materials (FGMs) with gradual transition microstructures. In addition, L-PBF as a powder bed technology can be emphasized as a more prospective method for producing crack-free TiAl samples.

These AM technologies are discussed in terms of processing challenges, by highlighting the different methods in reducing and even preventing cracking susceptibility, such as preheating and post-heating stages between layers, process optimisation. In the case of gamma aluminides, because of the large variety of microstructures involved with the rapidly solidified condition, a strong process – microstructure – property relationship is observed. So, the different AM technologies are also compared in terms of optimum process conditions, microstructure characteristics, and related mechanical properties.

Finally, this review addresses novel technology and on-going effort of AM TiAl alloy development. For instance, successful attempts are reported in the literature to combine conventional casting or forging processes with DED by using similar or dissimilar TiAl alloys. Some studies have also emerged on the R&D of *in situ* reactive synthesis from elemental powders by using DED for Ti-Al alloys. Moreover, blending pre-alloyed TiAl powder and $Si_3N_4$ precursor has been used to prepare TiAl metal matrix composites from *in situ* reaction during the AM process. The wire arc additive manufacturing (WAAM) process can also be highlighted to successfully fabricate crack-free TiAl samples by using different elemental wire consumables. Attention is also drawn to the properties and challenges associated with the chemical composition of different AM TiAl alloys.

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# Production of intermetallic, spherical powders

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## Introduction

The development of ultra-high temperature alloys has identified MoSiB, MoTiSi- and VSiB alloys as promising candidates for future applications. They demonstrate high strength, high melting points and intrinsic protection against oxidation [1-4]. Due to the high melting point and high strength, the materials need to be produced through a powder-metallurgical approach and products built through additive manufacturing. The production of spherical MoSiB, MoTiSi and VSiB- Powders, needed for additive manufacturing, will be introduced in this article. The powders have successfully been processed through additive manufacturing [5-7].

## Materials and Methods

To produce the feedstock for the EIGA process, a powder metallurgical route was chosen. Alloying elements were mixed and homogenized, to obtain the target compositions. The powders were compacted and underwent a thermotreatment at a temperature below the exothermal peak, which was determined beforehand through DSC measurements. The dimensions of the EIGA feedstock material were 65mm diameter for all materials, tested.

## Results and Discussion

Stable process conditions could be obtained. The expected phases could be detected in the powder particles, which points to a fully alloyed material. The obtained powders show good flowability for particles 25-80µm, which makes them suitable for additive manufacturing.

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# Development of the TiAl IRIS alloy by Spark Plasma Sintering

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During the last fifteen years, the TiAl-IRIS alloy ($Ti_{49,9}Al_{48}W_2B_{0,1}$) has been developed in consistency with the Spark Plasma Sintering (SPS) for structural applications at temperatures up to 800 °C in aeronautic and automotive engines. SPS is a consolidated powder metallurgy (PM) process where a direct pulsed electric current of high intensity and an uniaxial pressure are simultaneously applied on a graphite assembly containing the powder. This presentation is divided into three parts.

The first part will be devoted to the SPS process. It will be shown how versatile SPS is to control and optimise TiAl microstructures. The most critical issues, as the quality of the powder, the control of the alloy temperature and the sintering of up-scaled billets, will be addressed. The experimental method used to sinter near-net-shape blades in a single SPS step will also be shown.

The aim of the second part is to describe the route to obtain alloys with improved properties, satisfying the industrial requirements. The initial goal was to reach a good compromise between the creep resistance at high temperature and the ductility at room temperature. This was successfully achieved through the addition of boron and tungsten, and through the development of a resistant lamellar microstructure with small colonies. The followed step by step procedure, which is based upon some basic researches on the transformation mechanisms activated along the whole cycle and on the mechanical properties, will be described in details. The mechanism of the microstructural evolution during the SPS cycle will also be presented.

In the last part, the achieved properties of the SPS-IRIS alloy will be emphasised, i.e. ductility, mechanical strength at various temperatures, creep, fatigue, structural stability and oxidation resistance. The alloy behaviour will be correlated to the microstructural characteristics and to the deformation mechanisms which are activated under various solicitations.

In the discussion section, the advantage of the spark plasma sintering process and the interest of tungsten as alloying element will be underlined.

# Development of TiAl turbine blade with metal injection molding process

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Titanium aluminides (TiAl alloy) are attractive materials at intermediate high temperature because of its excellent specific strength. Therefore, TiAl alloy have been replaced Ni base superalloys in the low-pressure turbine (LPT) of the state-of-the-art aircraft engines. The manufacturing process of TiAl turbine blade are mainly casting, forging or additive manufacturing. As more cost effective manufacturing process for large quantity production is required, TiAl turbine blade with metal injection molding process were developed.

The metal injection molding process consists of ingot production, powder production, feedstock production, injection molding, debinding, sintering, and heat treatment. In order to manufacture high quality blade, modeling and simulation of the injection molding and sintering process and optimization of the heat treatment for microstructures were done. The simulation of the injection molding results in control of filling, packing and powder distribution in the binder. The simulation of sintering process using actual alloy data results in control of deformation on the setter during sintering. The microstructure optimization by heat treatment, developed by Takeyama et al, results in $\alpha 2/\gamma$ lamellar microstructure with $\beta/\gamma$ cellular microstructure of high strength and high fracture toughness, regardless of high oxygen content. As a result, high quality, and precise blade of 200mm can be manufactured with metal injection molding.

Fatigue properties of the developed LPT blade (Fig.1) were evaluated. The fatigue properties of the blade are the equivalent to those of small test specimens.

This work was supported by Council for Science, Technology and Innovation (CSTI), Cross-ministerial Strategic Innovation Promotion Program (SIP), “Material Integration” for revolutionary design system of structural materials.

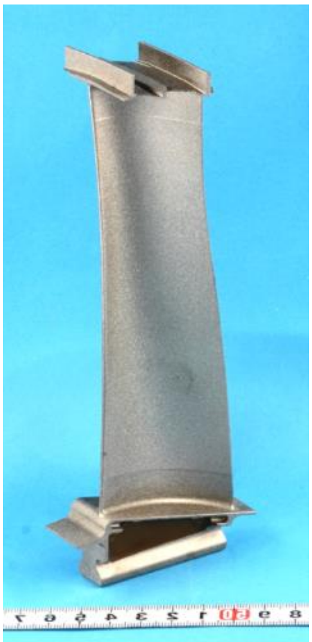

Fig.1 Developed LPT Blade

# FAST/SPS: NEW industrial post-process for full densification of 3D TiAl complex shape from additive manufacturing

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Norimat made an important industrial breakthrough by developing a unique and easy process which enables the consolidation of 3D complex shapes by Spark Plasma Sintering (or Field Assisted Sintering Technique). We have developed the hybridization between sinter based additive manufacturing and SPS to fully densify (porosity <1%) Near Net Shape parts in only one step and less than 1h of thermal treatment.

The conference will focus on the development of fully dense TiAl near net shape parts by FAST/SPS. The recent results on the capability of the process opening the way to large scale production and the improvement of multi-physics modeling to help users at each step (size and scale up) of the FAST/SPS process from conception to production.

Figure 1 : NNS fully densified TiAl blades just after SPS sintering

FAST/SPS is an efficient powder densification technique. It is used to sinter a wide variety of materials (ceramics, metals, alloys, composites, etc.). Compared to conventional processes, it allows to reduce by 10 the production time while enhancing material performances. FAST/SPS is at a turning point in its history, attracting much interest in recent years for multiple reasons:

1. Advanced industry is looking for new process to facilitate innovative material development and manufacture.
2. FAST/SPS is an ecofriendly process without any greenhouse gaz emitted, having zero material losses and low electric consumption.
3. High volume production is now achievable through mastering key process parameters thanks to innovative numerical tools capable of optimizing the process from conception to production. 3D complex shapes are now achievable using FAST/SPS, opening the way to the manufacturing of Near Net Shape parts for all types of material (ceramics, metals, composites, ...)

# Application of MIM process to TiAl Turbine Blade

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Abstract

Titanium aluminides(TiAl) Turbine Blade is now introduced to Low Pressure Turbine(LPT) of Aero Engine as a replacement of conventional Ni based super alloy because of its excellent material properties in high temperature environment with lower density than that of conventional Ni based super alloy. Current TiAl Turbine Blade in production is produced by machining from the raw material of casting or forgings. Recently additive manufacturing(AM) technology is being introduced to produce near net raw material of TiAl Turbine Blade. One of the reasons for this introduction of new manufacturing technology is that there are a few limited raw material suppliers due to its manufacturing difficulties in casting or forging process related to chemical reaction characteristics or low ductility. As one of other solutions to produce TiAl Turbine Blade, Metal Injection Molding (MIM) process might be a potential option to produce near net shape raw material because of its high flexibility in shape, material and high productivity for high volume production.

This article describes overview of the past experimental trials in application of MIM process to TiAl LPT Blade such as sintering process, material tests, component tests for the confirmation of microstructure and material capability in strength, creep and fatigue using small scale TiAl Turbine Blade and future challenges of large scale TiAl Turbine Blade produced by MIM process.


Fig.1 Small scale Turbine Blae

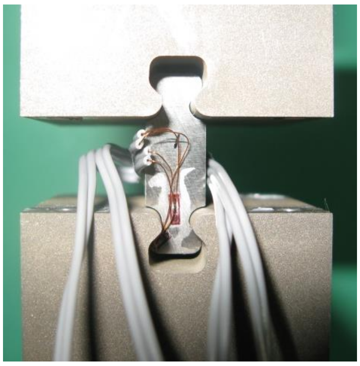
Fig.2 Dovetail Test

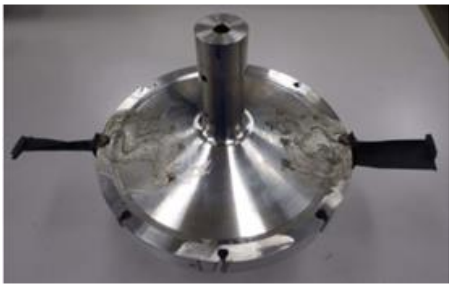
Fig.3 Spin Test

# Microstructural Heterogeneity and Post Processing Effects on Mechanical Properties of Ti-48Al-2Cr-2Nb Additively Manufactured by Electron Beam Melting (EBM)

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**Abstract:**

Both cast and wrought titanium aluminide alloys have been studied for more than two decades because of attractive properties that include low density, high specific strength, high specific stiffness and oxidation resistance up to about 700°C. Electron beam melting provides another processing approach to produce net shape components, although little work has been conducted to examine processing-microstructure-property relationships. This work examines as-deposited γ-TiAl (Ti-48Al-2Cr-2Nb) specimens made by Arcam AB and post-processed materials. Mechanical behavior studies on as-deposited, HIPed, HIPed + HT conditions included Vickers micro-hardness, compression, fracture toughness and fatigue crack growth testing. In addition, microstructural details were investigated over a range of scales using various microscopy tools. The presentation will summarize this evolving work on the characterization of AM γ TiAl and provide some comparison to other conventional (e.g. as-cast, wrought) γ-TiAl alloys.

# Influence of some promising alloying elements on the mechanical properties and deformation mechanisms of TiAl based alloys

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The aerospace sector faces a significant challenge in reducing CO2 emissions, which calls for the creation of novel functional materials that can withstand high temperatures. TiAl intermetallic alloys make suitable choices because of their low density and their good mechanical resistance at high temperature. These alloys exhibit a maximum working temperature around 700°C and are used in low-pressure parts of turbine blades. One of the major issues consists in improve their function temperatures of at least 800°C to be incorporated in higher pressure parts. Three alloys which exhibit high potential for industrial applications at high temperatures have been developed with the addition of alloying elements: IRIS alloy (Ti49.92-Al48-W2-B0.08), developed by CEMES and ONERA and TNM (Ti51.4-Al43.5-Nb4-Mo1-Bo0.1) / TNM+ (Ti50.7-Al44-Nb3.6-Mo1-C0.3-Si0.3-Bo0.1) alloys, developed by Leoben University. These developments reveal that the incorporation of alloying elements such as tungsten, molybdenum, carbon, and silicon, in the chemical composition of the TiAl alloys is an interesting way to improve their resistance at higher temperature. However, these enhanced properties are not yet well understood.

In this context a fundamental study has been carried out to better understand the effect of each element on the high temperature properties of TiAl with a particular interest in the role of tungsten. Thus, five model alloys with specific chemical composition (Ti48.4Al / Ti-48.7Al-2.1Mo / Ti-46.3Al-2.2W / Ti-46.3Al-2.2W-0.2C / Ti-46.5Al-1.9W-0.3Si) were developed to isolate the specific effect of each element of the study. Elaboration was done by spark plasma sintering from powders obtained by EIGA process. Tensile and creep tests were performed on the different alloys with a lamellar microstructure to determine which alloy exhibits the best potential for future industrial applications. Specimens of gamma microstructure of Ti48.4Al and Ti-46.3Al-2.2W composition were deformed in creep at 800°C, on which *post mortem* and *in situ* TEM observations have been carried out. These observations allowed to determine the active deformation mechanisms, as well as their kinetics. In addition to these observations, atomic probe experiments were conducted on the Ti-46.3Al-2.2W alloy to investigate the role of tungsten on the properties of TiAl at the atomic scale.

# High Temperature Oxidation of γ-TiAl Alloys: Effect of Zr

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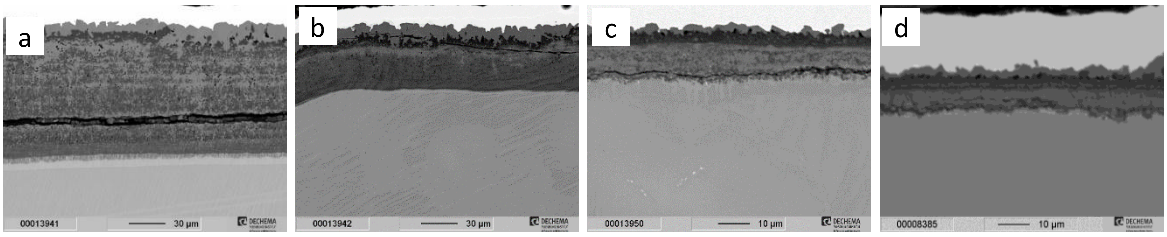
Intermetallic titanium aluminides based on the γ-TiAl phase (TiAl alloys) are candidates for several high temperature applications due to their high specific strength at elevated temperatures among other properties. TiAl alloys are already used as turbine blades in the last stages of the low-pressure turbine of jet engines. Their maximum operating temperature does not exceed about 700°C for longer service times due to an insufficient oxidation resistance and oxygen induced embrittlement. Alloying is one possibility to improve the oxidation resistance of TiAl alloys. The study of alloying additions, e.g., with Nb is a major topic in research on TiAl alloys since many years. Several technical alloys possess a certain amount of the β-stabilizing element Nb, e.g., the alloy Ti-48Al-2Nb-2Cr (TiAl 4822) used in the GEnX engine. New developments led to the addition of Zr to increase the mechanical properties. Yet it is unknown how Zr affects the oxidation behavior of γ-TiAl based alloys. Zr is known to improve the oxidation behavior of alumina forming Fe- or Ni-based materials by the so-called reactive element effect. Hence, different amounts of Zr (2, 4 and 8, all in at. %) were alloyed to a binary alloy with the base composition Ti-46.5Al. The Al content was kept constant. For a fourth alloy also Nb was added next to Zr (3 at.% Zr + 1 at.% Nb) and the effect of these additions on the oxidation behavior at 900°C in air were studied. Isothermal and thermocyclic exposure tests were performed and the results are discussed also in comparison to technical alloys, e.g., the TNM and the TiAl 4822 alloy. All alloys form mixed oxide scales which show spallation, but the tendency of spallation differs. Figures 1a-d show SEM images of the three Zr containing specimens and a TiAl 4822 sample after thermocyclic exposure (25 h-cycle) at 900°C for 120 h in air. Alloying with 2 at. % Zr reduces the oxide scale thickness (Fig. 1b) compared to the binary alloy (Fig. 1a). The additional alloying with Nb further improves the oxidation behavior without impeding mixed scale formation (Fig. 1c) which comes close to the thickness of the scale formed on the technical alloy TiAl 4822 already in service (Fig. 1d).




Figures 1: SEM images taken in BSE contrast of the mixed scales formed on the surface of the investigated alloys: (a) Ti-46.5Al, (b) Ti-46.5Al-2Zr, (c) Ti-46.5Al-3Zr-1Nb, and (d) TiAl 4822 after 5 cycles of oxidation (= 120 h) at 900°C in air. Note that the scale bar in Figs. (a) and (b) is 30 µm, while in Figs. (c) and (d) it is 10 µm.

# Effects of Various Factors on the Impact Resistance of TiAl Alloys

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Despite the widespread use of TiAl alloys, their poor impact resistance has recently emerged again as a major issue. Therefore, in this study, the effects of various factors on the impact resistance of TiAl alloys were investigated. Cast and forged materials were used as test samples. The investigated alloy composition were binary alloys, ternary alloys with varying amounts of additive elements, and various practical TiAl alloys including 4822 and TNM. Additionally, the microstructure was changed by heat treatment. The evaluation method of impact resistance used was the Charpy impact test with a small-capacity hammer using a non-notched test specimen, and the test temperature ranged from room temperature to 1000 °C. Consequently, the following findings were clarified:

- All TiAl alloys underwent brittle fracturing, even at high temperatures. Figure 1 shows a secondary electron image of the fractured surface of 4822 as a representative example.
- The Charpy absorbed energy of practical TiAl alloys was less than 1/20 at room temperature and less than 1/8 at elevated temperatures compared to that of Inconel713C.

When comparing between TiAl alloys:

- The absorbed energy increased at Al concentrations of 46–47at%.
- As for the microstructure, fully lamellar was good, and the $\gamma$ single phase and $\gamma + \beta$, which do not contain the lamellar structure, exhibited significantly low impact resistance. Moreover, the $\beta$ phase reduced impact resistance at low and medium temperatures. Therefore, cast alloys have better impact resistance than forged alloys.
- Additive elements that improved impact resistance were only Cr and V with appropriate amount, and all the other elements reduced impact resistance compared to the binary alloy.
- Particularly, Nb, C, and Si, which are usually added to improve high-temperature properties, significantly reduced impact resistance.
- The impact resistance of all practical TiAl alloys was significantly lower than that of the binary alloys.

Based on the above-mentioned findings, it is questionable to add Nb to the TiAl alloys for low-temperature applications, and considering the material cost together, a binary alloy or a ternary alloy with Cr/V addition is sufficient.

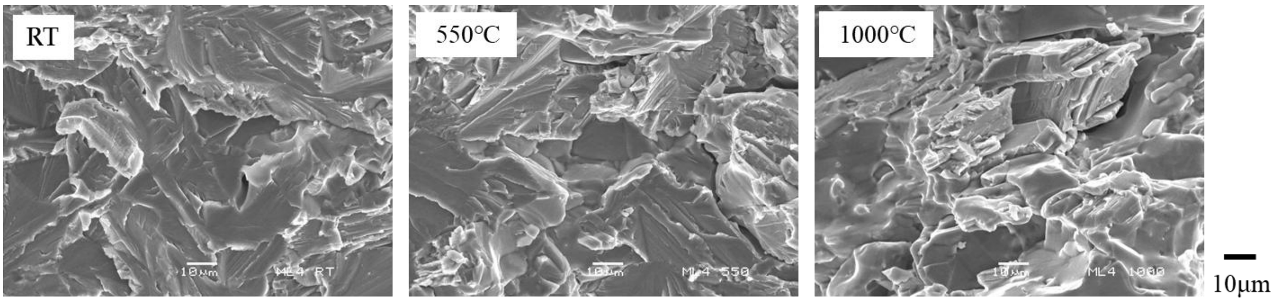


**Figure 1.** Secondary electron images showing the fractured surfaces of 4822 tested by Charpy impact tests at RT, 550 and 1000 °C.

# Deformation twins in $\alpha_2$-$Ti_3Al$ phase

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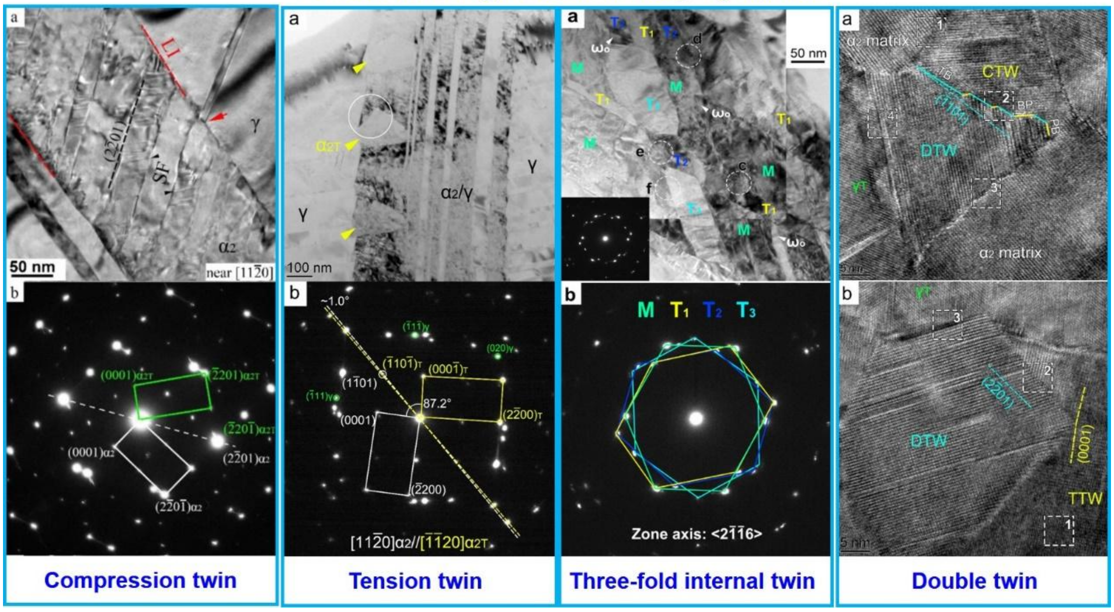
**Abstract**

Compared to the disordered HCP metals, the $\alpha_2$-$Ti_3Al$ phase is reluctant to twin due to its $D0_{19}$ ordered structure. However, in a high Nb-containing TiAl alloy under high temperature compression, abundant deformation twins were observed in the $\alpha_2$ phase. The twinning modes include $\{20\bar{2}1\}<\bar{1}014>$ compression twin, $\{1\bar{1}01\}<\bar{1}102>$ tension twin, three-fold internal twins and double twins in various modes. Given the large shufflings and the atomic site interchanges required for the operation of this twinning system, it is concluded that twinning of the $\alpha_2$ phase is a diffusive-displacive process. The details of the atomic processes of each type of twin are elucidated by electron microscopy. In the compression twins, $\omega_o$ phase nucleation was observed at the twin interface. In the tension twins, the structure of interface is similar to those reported in the disordered $\alpha$ phase, which is formed by prismatic (P) and (0001) basal (B) planes. The shear mechanisms of the twins were discussed based on the observed morphology. The factors that support the deformation twins in the $\alpha_2$ phase were summarized. Our results indicate that in modern TiAl alloys deformation twins in the $\alpha_2$ phase could be an important mechanism at service temperatures.

**Various twinning modes in the $\alpha_2$-$Ti_3Al$ phase**



# An Overview on "Materials Integration" for Revolutionary Design System of Structural Materials in SIP in Japan

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Cross-ministerial Strategic Innovation Promotion Program (SIP) is a national program led by the Council for Science, Technology and Innovation（CSTI）of the Government of Japan, in order to realize scientific and technological innovation to solve the most socially and economically important national issues. 'Materials Integration for Revolutionary Design System of Structural Materials', one of the 12 subjects in the 2nd term of SIP, was conducted for 5 years from FY2018 till FY2022.

To strengthen the global competitiveness of materials science and engineering of Japan, materials integration (MI) systems have been developed which combine materials engineering methods, experiments, theoretical computations, and data science to connect processing, structure, property, and performance on computers to accelerate R&D. In 5 years, 10.7 billion JPY was invested, and around 800 researchers from 49 organizations (19 in industry, 25 in academia and 5 in government) tackled the subject. As main R&D accomplishments, MI systems called 'MInt' for structural metallic materials and 'CoSMIC' for carbon fiber reinforced plastics (CFRP), and MI systems for new materials such as intermetallic compounds and ceramic matrix composites have been developed.

These systems contain inverse design functionality, which can form the conditions of materials and manufacturing processes necessary to realize required performance. In the final fiscal year, computational modules and databases were installed in these MI systems, verified and validated. Regarding practical application, the final goal is to have our MI system be broadly utilized by industry, academia and government to support their materials R&D.

One of the most remarkable accomplishments in this program is TiAl LPT blade manufactured by MIM and AM processes. The newly designed blades are developed through a uniquely developed inverse MI system, and they are verified to meet the required performance. You will know how good they are in this workshop. We always welcome any of you to work together on R&D of materials in the future.

# Lamella orientation control of β-Solidifying TNM Alloys via High-Temperature Compression

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The third generation β-solidifying TNM alloys with (α2+γ) lamellar microstructures have been considered excellent candidates for modern turbine blades due to their low density, high specific strength and stiffness, excellent creep resistance, and good corrosion resistance. It has been found that orienting the γ lamellae to the direction of the load can significantly increase the mechanical properties of the alloys, making lamella orientation control (texturization) an interesting topic for property optimization [1].

In this study, high-temperature compression (with a dilatometer) was first achieved to texturize the alpha phase through optimization of compressive speed and strain. An optimum fiber texture for the alpha phase has been identified by combining EBSD analysis with an in-situ XRD synchrotron.

Moreover, experiments were performed to observe the effect of the strain rates, taking a high strain rate of 1 $s^{-1}$ and low strain rate of $10^{-2}$ $s^{-1}$ while keeping the other parameters constant (cooling rates, externally applied load during cooling, total deformation, and temperature). A difference in behavior for the true stress-strain curve has been highlighted corresponding to a different type of mechanisms of dynamic recrystallization.

Our results show that the microstructure and texture of TiAl alloys can be effectively controlled, and it seems that having a high deformation and a low strain rate should promote the uniform fiber texture.

Further analyses are needed to understand the mechanisms behind the observed texture evolution. These findings have potential implications for optimizing the processing and performance of TiAl alloys.

[1] « Polysynthetic twinned TiAl single crystals for high-temperature applications » Chen et al. Nature Materials vol.15 August 2016

# Effect of Microstructure on Creep Properties of TiAl4822 Built by Selective Laser Melting

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## Introduction

In this study, selective laser melting (SLM) with a new heating system and an inert gas control unit was applied to Ti-48Al-2Cr-2Nb and investigated the effect of addition of yttrium and heat treatment on creep properties at high temperature.

## Experiments

An M290 (EOS GmbH Electro Optical Systems) with a high-temperature modification unit that features a cooling of the process chamber and an inert gas purification system was used to build the sample. Microstructure was analyzed by scanning electron microscope (SEM). The creep test was carried out with the condition of 750℃/200 MPa in air. Addition of 0.21 wt.% of yttrium and heat treatment at 1400℃ for 2 hours were applied to the TiAl4822 to control the microstructure.

## Results and Discussions

Fig. 1(a) shows the microstructure of the as-built specimen. The microstructure of the SLM specimens with yttrium was shown in Fig. 1 (b). It was found that the addition of yttrium effectively contributed to the suppression of the growth of the grain size. Fig. 1 (c) is the microstructure with both heat treatment and addition of yttrium. By applying both heat treatment and addition of yttrium, small grains of lamellae were obtained. The pinning effect on the grain growth contributed to the suppression of the movement of the grain boundary. Fig. 2 shows the results of creep tests. Adoption of both heat treatment and yttrium was the most effective among all the conditions to improve the creep rupture life. This is mainly because the appropriate grain size of lamellar was formed by a combination of the addition of the yttrium and the heat treatment.

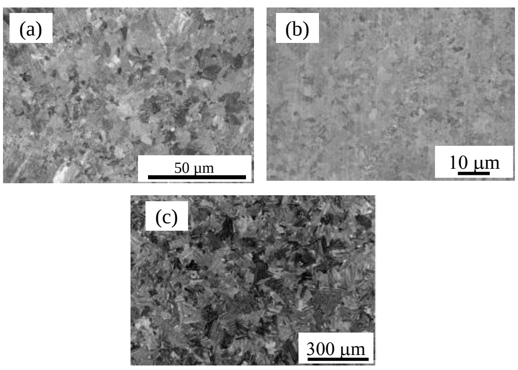


Fig. 1 SEM images of the microstructure of SLM specimens. (a) As-built SLM specimen. (b) Specimen with 0.21 wt.% of yttrium. (c) Specimen with both the heat treatment and the addition of yttrium.

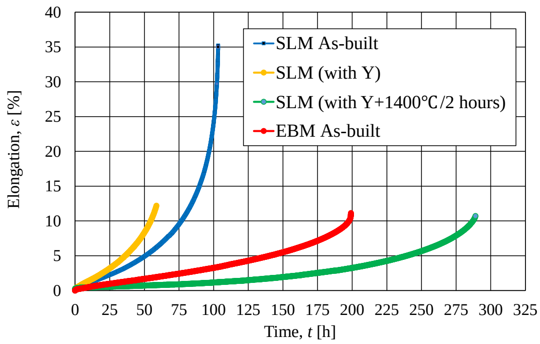


Fig. 2. Comparison of the creep curves. Creep rupture life of the SLM specimen with both addition of yttrium and the heat treatment was improved by more than 200 % and was even longer than the conventional electron beam melting (EBM) specimen.

# Fatigue cracking in additively manufactured gamma-TiAl

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As the aeroengine industry is constantly increasing its interest in exploiting the advantages offered by lightweight intermetallic alloys based on titanium aluminides, improved materials properties satisfying high demanding design requirements are needed, so that novel designs with enhanced levels of structural integrity would allow to further decrease the consumption of fossil fuels and reduce the carbon footprint of long-haul flight.

In the last few years, additive manufacturing technologies have been used to effectively produce gamma-TiAl alloys suitable for structural components.

In this work, the fatigue properties of different variants of γ-TiAl intermetallics, with slightly different chemical compositions and microstructures, produced by (selective) Electron Beam Melting (EBM) are presented. The fatigue properties of an enhanced Ti-48Al-2Nb-2Cr alloy, a high Nb containing and a Mo-containing TiAl alloys are compared with already established alloys, all produced by additive manufacturing by selective Electron Beam Melting (EBM), with the aim of highlighting, when possible, the effect of the microstructure on the fatigue properties. High-cycle fatigue experiments have been conducted in the fatigue crack growth threshold region for examining how the local microstructure influences local damage accumulation processes.

Additionally, specific monotonic and cyclic loading experiments with sub-size samples have been recently conducted for investigating the influence of the microstructure on the fatigue damage accumulation process. This analysis provides insightful information on the role of the intermetallic phases on the fatigue behavior of gamma-TiAl alloys and, by allowing a comparison between different heat treatments and resulting lamellar colonies size, permits to highlight the influence of the position of grain boundaries and the orientation of the lamellae for the onset of fatigue cracking. Finally, a handful of fatigue crack growth experiments with sub-size specimens have been also conducted with the aim of highlighting the behavior of fatigue cracks in the threshold region, with special focus to the contribution of shielding mechanisms in the near threshold regime. The analysis and comparisons of different microstructures and alloy formulations presented here is aimed at providing information for the selection of the microstructures suitable for designing against fatigue with gamma-TiAl intermetallics.

# Plasticity and brittleness of the ordered $\beta_o$ phase in a TNM-TiAl alloy

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In order to identify the reasons for the brittleness of a TNM-TiAl alloy ($Ti_{51.05}Al_{43.9}Nb_4Mo_{0.95}B_{0.1}$ in at.%), the plastic deformation behaviour of the ordered $\beta_o$ phase is investigated. The corresponding powder was densified by Spark Plasma Sintering to produce a near lamellar microstructure made of $\gamma/\alpha_2$ lamellar colonies surrounded by $\gamma$ and $\beta_o$ grains. Then, the room temperature tensile behaviour is studied by carrying out tensile tests and additional in-situ straining experiments in a transmission electron microscope (TEM). The TNM alloy exhibits a limited ductility, with the fracture surface of test specimens showing cleavage facets along lamellar interfaces. In addition, the $\beta_o$ grains contain nano-precipitates of the $\omega_o$ phase and deform plastically by <111> superdislocations dissociated into two superpartial dislocations separated by an antiphase boundary. These dislocations glide in {011} planes and are observed to be localized into pile-ups, which is related to the $\omega_o$ strengthening precipitation within the $\beta_o$ grains. This localised hardening behaviour leads to stress concentration in grain boundaries which is assumed to be responsible for delamination along lamellar interfaces in neighbouring lamellar colonies.

Corresponding paper:

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# Embrittlement after high temperature exposure: overview of the literature in view of new findings

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It is well known that TiAl alloys are embrittled on room temperature tensile testing after being exposed to elevated temperatures, however the reason behind the phenomena is little understood. This contribution will present new results from an investigation that has been performed on exposed tensile test samples that were tested under liquid nitrogen. The findings present a new light on the embrittlement phenomena and will be discussed in relation to the various TiAl embrittlement theories that have been presented in the literature.

# Study of fatigue mechanisms at the microstructure level in TiAl alloys

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With a view to lightening aeronautical structures and using aircraft engines at increasingly high temperatures, research into titanium aluminide intermetallic alloys has increased in recent years, leading to their introduction in the most recent civil jet engines.

These alloys are highy dependent on the thermal history due to the processing route of the alloy, meaning that for the same chemical composition, different microstructures can be obtained, resulting in a dispersion of mechanical properties. In order to be able to take this specificity into account, studies have been carried out to propose a finite element analysis methodology that is able to take into account the effect of the microstructure of TiAl parts subjected to mechanical loads.

This study is part of the development of a multi-scale fatigue life assessment tool for TiAl intermetallics and more specifically for the TiAl IRIS grade. The aim of this work is to understand the link between TiAl alloys microstructures and their fatigue strengths.

The fatigue analysis at the microstructure scale is performed using Fatigue indicators (FIP) that give robust measures of the driving force for fatigue crack formation. Two types of potential cracks relevant to the mechanisms observed in the material are considered here, volume fatigue transgranular and surface crack formation. The criteria are applied in a post-processing step after a set number of cycles of a semi-periodic synthetic crystalline aggregate. The simulations can be made very efficient by taking advantage of the concept of structural zoom, where sub-domains of a larger model can be studied with increased detail (taking into account the lamellar structure of individual grains for example). Pertinent FIP for transgranular volumetric crack propagation are those that associate the crack direction with the plastic slip directions, for example a modified Fatemi-Socie criterion. The surface rugosity that develops on a free surface during cyclic loading can be estimated using the results from the crystal plasticity simulation. A criterion based on the slip activity on each individual slip system is proposed that provides both the amplitude of the intrusions and extrusions as well as their direction. The results of these simulations are compared with experimental characterisations of these intrusion/extrusion phenomena, obtained in particular from SEM observations and quantified by means of AFM measurements (Figure 1).

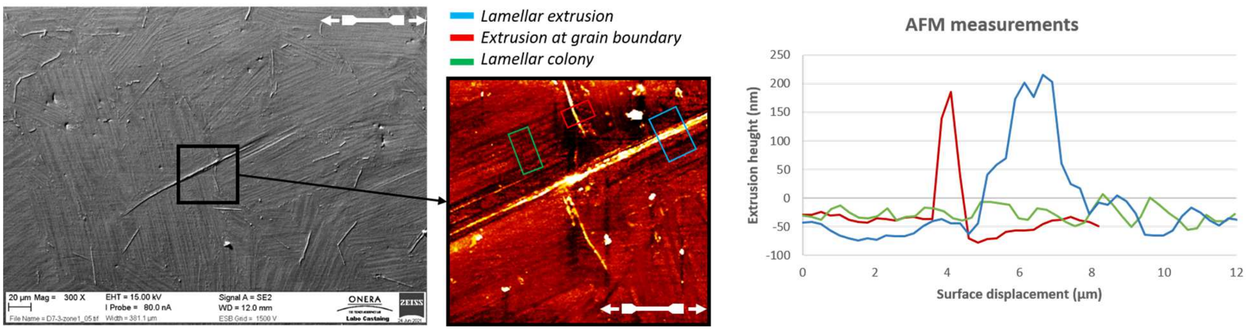


Figure 1. SEM intrusion and extrusion on a TiAl IRIS specimen subjected to cyclic loading at 750°C. Focus on AFM measurement on a lamellar extrusion, on an extrusion at grain boundary and on a lamellar colony.

# Microstructure Control of TiAl Alloys by Additive Manufacturing

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Cylindrical rods of Ti-48Al-2Cr-2Nb alloys were additively manufactured by electron beam powder bed fusion (EB-PBF), so that the angle (θ) between the rods and the building direction is 0, 45 and 90 deg. Microstructure and mechanical properties of the alloys depend strongly on the building condition and θ. For instance, high energy densities lead to the formation of $\alpha_2/\gamma$ lamellar structure while near-γ structure is formed at low energy densities. On the other hand, at middle energy densities, unique layered structure composed of duplex-like region and equiaxed γ grains (γ band) is formed. Temperature distribution near the melt pool and the layer-on-layer process during EB-PBF are responsible for the formation of the layered structure. If tensile load is applied parallel to the longitudinal direction of the cylindrical rods at room temperature, large elongation above 2% can be obtained at θ = 45 deg, while tensile elongation at θ = 0 deg is less than 1%. This is because shear deformation preferentially takes place parallel to the soft γ bands. Moreover, tensile elongation at room temperature is dependent on the thickness of the γ bands at θ = 45 deg. On the other hand, the plastic anisotropy depending on θ becomes insignificant above 973 K. In addition, fatigue and creep properties of the additively manufactured alloys at 1023 K are comparable to that of the cast alloys. Hot isostatic pressing (HIP) is favorable to eliminate pores, while the layered structure is stable even after HIP at and below 1463 K. When large TiAl turbine blades are fabricated by EB-PBF at constant process parameters, temperature distribution during EB-PBF depends on the size and shape of the blade, which results in inhomogenous microstructure. A scan strategy considering heat transfer around an electron beam enables us to fabricate TiAl turbine blades with homogenous layered structure. Additive manufacturing of TiAl alloys containing the β phase was also examined. This study was supported by the Cross-Ministerial Strategic Innovation Promotion Program (SIP) from the Japan Science and Technology Agency (JST).

# A simulation-based approach for EBM additive manufacturing of γ-TiAl

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Electron Beam Melting (EBM) is a metal powder bed additive manufacturing technology that has a proven capability of producing advanced components in several "hard to weld" alloys, Avio Aero - a GE Aerospace business uses EBM technology for production of γ-TiAl blades for the low-pressure turbine of GE's GE9X engine. The key benefits of using EBM are the vacuum environment, the high process temperature, and the high-speed beam movements.

By combining these EBM unique benefits with recent developments in mesoscale melt pool modelling and thermal simulations on the macroscale, new melting and heating strategies have been developed. The melt pool modeling results provide details of the melt pool dynamics and the solidification rates of the material, leading to new melt strategies. The macroscale thermal simulations are used for controlling the overall energy input and the temperature distribution on the part level. Together, these new developments open up EBM for enhanced process control during production, as well as detailed microstructure tailoring at the part level in the future

# Microstructure and Mechanical Properties of Additively Manufactured β–containing TiAl Alloys

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Recently, electron beam powder bed fusion (EB-PBF) has attracted much attention to fabricate difficult-to-process materials such as TiAl alloys since the process can build 3D objects with complex shape. In this study, rectangular rods of β-containing TiAl alloys were prepared by EB-PBF with various process conditions. The microstructure and mechanical properties of these rods were investigated focusing on input energy density. We found that it is necessary to lower the energy density to improve the dimensional accuracy of the rods. However, excessively low energy density causes formation of defects due to a lack of fusion. This means that suitable process conditions should be selected for fabricating defect free samples with excellent dimensional accuracy. We also found that the microstructure of the alloy fabricated by the EB-PBF process varies drastically depending on the energy density. A uniform $\alpha_2/\beta/\gamma$ mixed structure containing an $\alpha_2/\gamma$ lamellar and a $\beta/\gamma$ dual-phase regions is formed at high energy density conditions. On the other hand, a lower energy density leads to the formation of ultrafine $\alpha_2/\gamma$ lamellar grains with $\beta/\gamma$ cells discontinuously precipitated at the grain boundary. The ultrafine $\alpha_2/\gamma$ lamellar grains are formed via massive $\alpha$ phase transformation induced by rapid cooling under low energy density conditions. It is important to note that the ultrafine $\alpha_2/\gamma$ lamellar grains and the $\beta/\gamma$ cells are effective in increasing strength and ductility of the alloys, respectively. The alloys with optimum microstructure exhibit higher strength and larger ductility at 1023 K, compared with the cast alloys.

This study was supported by the Council for Science, Technology and Innovation(CSTI), Cross-ministerial Strategic Innovation Promotion Program (SIP), “Materials integration for revolutionary design system of structural materials”, Japan.

# Solid solution Strengthening of TiAl alloys with Zirconium, mechanical properties

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**Introduction**

The objective of the work is to improve the high temperature mechanical properties of titanium aluminide alloys by using Zirconium for solid solution strengthening. Zr is a rarely used alloying element in titanium aluminides but has a high potential of influencing the high temperature properties positively due to its large atomic radius compared to Ti and/or Al.

**Materials and Methods**

Materials manufacturing is based on single Vacuum Arc Remelting (VAR) using compacted consumable electrodes. Resulting VAR ingot are being remelted and homogenized in an Induction Skull Melter and centrifugally cast in steel molds [1]. Two different ternary alloys containing 2 and 4 at% Zr and 46,5 at% Al each have been manufactured. Cast parts were subject to HIP for closing remaining casting porosity. Different heat treatments (HT) for creating appropriate microstructures have been applied. Chemical analyses have been carried out. The microstructural analysis was done by metallography and SEM. The tensile tests were done under standard test conditions at room temperature, 300°C, 700 °C and 850°C. The creep tests were performed with 150 MPa at 750°C and 850°C.

**Results and Discussion**

DSC measurements show that the content of Zr within the alloy has a strong influence on the phase transition temperatures as well as on the solidification path [2]. Adjusted heat treatments had to be developed for each alloy for generating comparable lamellar microstructures.

The increase of Zr content improves the yield strength in all temperature ranges. The decrease of strength at high temperature above 700°C was determined to be significantly lower compared to State-of-the- industrial TiAl alloys such as TNM and TiAl48-2-2 indicating a very effective solid solution strengthening capability of Zr in TiAl alloys.

The creep resistance could be improved at 750°C and 850°C compared to common TiAl alloys like TNM alloys.

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# Development of Rapid Determination Technique of Molten TiAl Alloys using X-ray Fluorescence

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## 1.Introduction

Since TiAl alloy is an active metal, melting techniques such as Cold Crucible Induction Melting (CCIM) have been developed, in which a solidifying shell is formed where the water-cooled copper crucible and molten metal come into contact, thereby preventing oxygen and impurities from entering the molten metal. On the other hand, changes in the solidifying shell may cause fluctuations in molten metal concentration. In the melting process of a new concentration system, it is difficult to accurately ascertain changes in the solidifying shell, which may lead to concentration deviations.

In order to meet the growing demand for TiAl alloys used in aircraft engine components, high-quality TiAl ingots must be produced at low cost. Therefore, if ingot scrapping can be reduced by controlling the concentration narrowing during melting, it will lead to the production of high-quality ingots at low cost. The author's group has been developing a rapid determination technique using X-ray fluorescence [1], but there were issues with the small number of N (the analysis variation in the same Al concentration was unknown) and the accuracy of certified reference materials and analytical curves. Therefore, the purpose of this study was to improve the accuracy of rapid determination technology and to verify the accuracy (N increase) through the preparation of high-precision certified reference materials and analytical curves.

## 2.Experiment

The target concentrations were Ti-(27.3, 29.3, 31.3, 33.3)Al-4.8Nb-2.55Cr (mass%). 16 samples were used for the four Al concentrations. 20 kg of material was melted at CCIM and the molten metal was collected (S-1). The Al concentration in the sample was then analyzed by X-ray fluorescence analysis (XRF). The material was added to fill the difference between the analysis result and the target concentration, and the sample was taken again (S-2). The concentration of the collected sample was then analyzed. As described above, loading and sampling were repeated until the target concentration was controlled.

## 3.Result and discussion

The analysis results of Al concentration were shown in Figure 1. The horizontal axis was the target Al concentration and the vertical axis was the analysis value of XRF. The upper and lower dotted lines indicated the target Al concentration range of ±0.2mass%, which shows that it was possible to control all 16 samples to within ±0.15mass% of the target Al concentration, indicating that the Al concentration could be controlled with high accuracy in the Al concentration range of 27mass% to 34mass%.

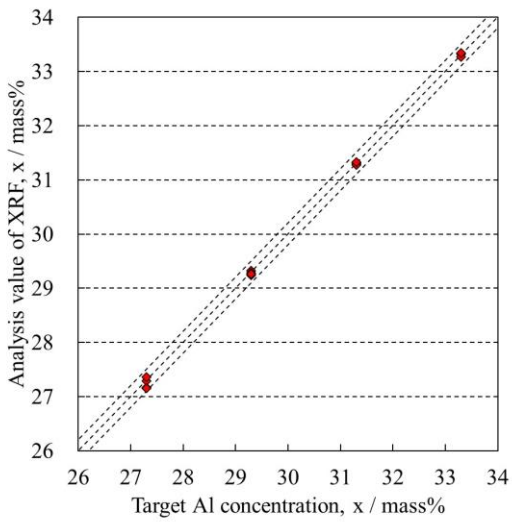


Fig.1 Result of Al concentration using rapid determination.

Acknowledgements

This study was supported by the Council for Science, Technology and Innovation (CSTI), Cross-ministerial Strategic Innovation Promotion Program (SIP), "Materials integration for revolutionary design system of structural materials", Japan.

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# Progress on Titanium Aluminides within the NEWTEAM EU project

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In order to follow the today's aero-engine market needs, the exploitation of high-performance materials is required. Introducing advanced alloys capable of withstanding higher operating temperatures and/or exhibiting higher performances at a given temperature is a never-ending focus of materials research. Additionally, coupling the new materials with advanced manufacturing systems, such as Additive Manufacturing (AM), will allow to produce complex geometry components and reduce their overall buy-to-fly ratio.

Among AM processes, the Electron Beam Powder Bed Fusion (EB-PBF) has already proved to be promising for processing intermetallic TiAl based alloys. However, considerable efforts are necessary to understand the role of the process parameters to precisely control the process in terms of residual porosity, Al loss and homogeneity of the microstructure, thus improving the material performances. Moreover, in the intermetallic TiAl based alloys produced by EB-PBF, the state-of-art-alloy for the industrial application of components, such as low-pressure turbine blades, is the Ti-48Al-2Cr-2Nb alloy.

The NEWTEAM project evaluated a modification of the chemical composition of the Ti-48Al-2Cr-2Nb reference alloy to increase the room temperature ductility of the alloy and limit the penalty in creep performances as much as possible. The post-processing, involving a Hot Isostatic Pressing (HIP) and a subsequent Heat Treatment (HT), was tailored for these new chemical compositions, thus optimising the material’s microstructure. One of the two investigated chemical compositions was of particular interest to the progress of the project.

Theoretical and experimental analysis has been combined with simulation tools to optimise the process parameters for EB-PBF production. Such a combined approach was conducted regarding the influence of Al loss during the EB-PBF process on phase fraction and phase transition temperatures.

The present work summarises the results obtained in the NEWTEAM project "Next gEneration loW pressure TurbinE Airfoils by aM" funded by the EU's Horizon 2020 programme in the framework of Clean Sky 2.

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# Data to aid materials design and process optimization of Titanium Aluminides

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Alloys are complex systems where microstructure and properties depend on both processing conditions and chemical composition. Such variations are not typically reflected in handbook data and repositories that tend to be limited in the scope of materials covered (their compositions) or the temperature ranges (processing conditions) or lack of time dependence. As such, the engineering simulations which depend on these data are limited, especially for cases involving novel materials or new processes and often it becomes necessary to go and measure the needed data or live with the uncertainty.

The CALPHAD approach captures the composition and temperature dependence of properties, as well as their temporal evolution, for industrial multicomponent alloys. As a result, data can be calculated for materials or conditions where there are gaps in the measured data. Additionally, location specific properties can be predicted and optimized for a part, which means that manufacturers will no longer be restricted to design minimums.

CALPHAD simulations can be used to complement compilations or repositories of measured data, improve machine learning models, and can also be used as input into engineering codes that require more reliable materials property data.

In this presentation we introduce the CALPHAD approach and exemplify how CALPHAD based tools can be efficiently used to aid materials design and process optimization of Titanium Aluminides.

# Cost-effective Vacuum induction melting of gamma TiAl and NiTi alloy

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Due to the nature of TiAl and NiTi as the intermetallic compound, the requests on materials composition and the materials soundness are outstanding high which is a very big challenge for manufacturing such attractive materials. As the first procedure of material process, melting directly determines the main element chemical composition precision and impurity level, these really affect

such intermetallic compound properties. Basically, the melting technology for TiAl or NiTi mostly are confined to vacuum arc remelting (VAR), plasma arc melting (PAM) as applied in the titanium industry. But such melting process still exists their own shortcomings, as well as the cost challenges.

The present research was mainly focused on the vacuum induction melting (VIM) technique development to explore the possibility that whether such cost-effective process can be applied for melting the more reactive TiAl and NiTi alloys. The thermodynamic stable CaO refractory was selected as the melting crucible. Firstly, the study on thermodynamics of calcium and oxygen in molten TiAl and NiTi alloys was performed to clarify the equilibrium properties by melting material in CaO crucible. Secondly, these two kinds of alloys were practically melted in CaO crucible as the functions of superheating temperature and holding time. The results show that the contamination can be minimized when the thermodynamic stable CaO crucible and proper melting technique were adopted. The oxygen content of actual melting TiAl ingot can be controlled below 700 ppm, and that of NiTi is below 250 ppm. The main element concentration deviation can be successfully controlled within 0.2 wt% from different melting heats. We expect this work can provide a strong basic data support for cost-effectively melting high performance homogenized gamma TiAl and NiTi shape memory alloys.

**Keywords:** TiAl; NiTi; melting; vacuum induction melting (VIM); CaO crucible.

# Control of lamellar colony size in sintering process for injection-molded TiAl alloys

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Metal injection molding (MIM) is a manufacturing method that combines injection molding and sintering. It enables the near-net-shape mass production of metal parts with complex geometries. Metal injection molded parts are densified by sintering, and the density of the sintered parts significantly affects the mechanical properties. A high density of the sintered parts can be obtained by increasing the sintering temperature. However, in the sintering of titanium aluminide (TiAl) based alloy, the lamellar colony becomes coarse when the sintering temperature is increased. If the lamellar colony size is large, a high creep strength can be obtained, although the fatigue strength is low. In order to obtain the desired mechanical properties, it is important to control the lamellar colony size. In the present study, sintering experiments were conducted to obtain the desired density while suppressing coarsening of the lamellar colonies.

TiAl based alloy powder was mixed with a certain amount binder to obtain a feedstock. The feedstock was injection molded into tensile specimens. After debinding by heating under a reduced pressure condition, the specimens were sintered under various conditions. The obtained specimens were evaluated through density measurements, microstructure observation and energy dispersive spectroscopy (EDS).

It was revealed that the desired density of the sintered compacts can be obtained while suppressing the coarsening of the lamellar colonies. It was also revealed that there is a sufficient possibility that the lamellar colony size of the sintered parts can be controlled through the sintering conditions.

# How elaborate *in situ* experiments guide modern alloy development


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Intermetallic titanium aluminide alloys based on the ordered γ-TiAl phase are promising materials for lightweight high-temperature applications. In addition to their low density of roughly 4 $g{\cdot}cm^{-3}$, their high specific Young's modulus and strength even at elevated temperatures, and their good oxidation and burn resistance, especially their excellent creep properties make these alloys a material of choice for challenging structural applications. Following intensive research and development activities, γ-TiAl based alloys have recently entered service in the automotive and aircraft engine industries, e.g. as low-pressure turbine blades in environment-friendly jet engines, as engine valves in sports and racing cars, or as turbocharger turbine wheels. In the course of the past decades, the development of these complex multi-phase alloys has benefited greatly from the application of *in situ* synchrotron X-ray techniques. Diffraction and scattering techniques (Fig. 1), in particular, have offered access to the atomic structure of the material and provided insights into a variety of microstructural parameters. Advanced experimental setups, which are steadily refined, have even allowed the exploration of elaborate manufacturing processes and yielded insights that have so far been inaccessible by means of conventional methods. Here, a practical introduction and overview of recent progress in this field of research are provided. Current prospects at modern synchrotron radiation sources will be illustrated by means of selected case studies pertaining to different stages in the development of modern γ-TiAl based alloys (*i.e.*, fundamental research, manufacturing, and fine-tuning of properties for application). In this context, available setups for *in situ* high-energy X-ray diffraction and small-angle X-ray scattering experiments will be discussed in terms of their advantages as well as their limitations.

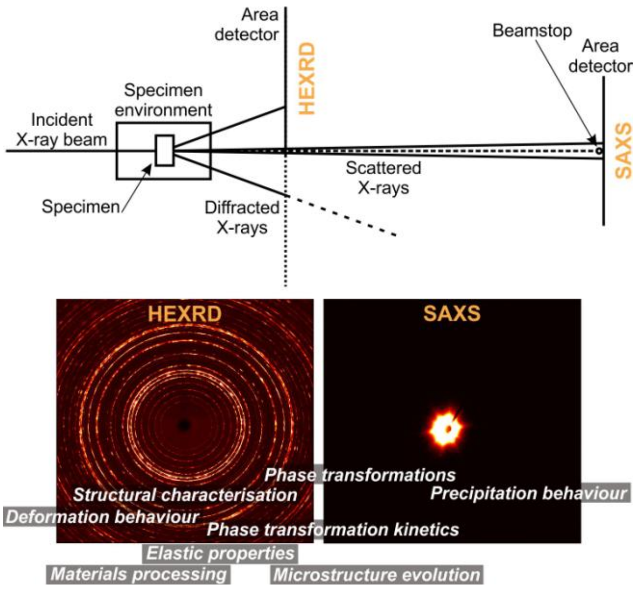


**Figure 1:** Schematic setup of a (combined) high-energy X-ray diffraction (HEXRD) and small-angle X-ray scattering (SAXS) experiment, exemplified patterns as recorded by means of the area detectors, and typical application areas for HEXRD and SAXS in the field of alloy development.

# Relaxation Mechanisms and Diffusion Processes in γ-TiAl Intermetallics

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In the last decades there has been a growing interest in developing new intermetallic families, which would we able to improve the specific performances of superalloys at high temperature. The TNB (Ti-Al-Nb) and the TNM (Ti-Al-Nb-Mo) families were developed to fulfil the required performances. In particular, the creep resistance should be improved and consequently the study of the diffusion mechanisms and the associated relaxation processes becomes very useful to get a deep understanding of the physics involved during creep. Mechanical spectroscopy, including the measurement of the internal friction spectra and the dynamic modulus curves as a function of temperature between 600 K and 1400 K allowed approaching the study of such processes. Previous works on several γ-TiAl alloys show several relaxation processes associated with the Ti diffusion in the $\alpha_2$ phase [1-3] and with the Al diffusion in the γ phase [4], as well as an internal friction background at high temperature, which is associated with the creep behaviour [2]. At present, new generation of γ-TiAl, called $TNM^+$, is being developed to improve the creep resistance by microalloying with C and Si [5]. However, Nb and C atoms in solid solution could have a secondary effect on creep resistance by slowing down the diffusion of Ti in the constitutive phases.

The aim of the present work is to overview the relaxation processes associated with Ti diffusion in $\alpha_2$ phase, which have been observed in several alloys with different amounts of Nb and C in solid solution, in order to evaluate their potential influence on such relaxation processes; the influence on Al diffusion will be also analysed. In addition, the relaxation processes observed by internal friction are related, through the activation parameters, to the diffusion coefficients of the different atomic species and compared with those from the literature. This comparison allows to obtain a plot of the diffusion coefficients of Ti and Al in γ-TiAl, expanded towards the temperature range close to the temperatures used in service conditions.

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# Microstructure and elastic properties in Ti-42Al-8.5Nb after long-term annealing at 550°C

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Titanium aluminides are often alloyed with beta stabilizing elements for enhanced creep strength and corrosion resistance. As a side effect, the addition of a third element to a binary alloy entails the formation of a new phase in its microstructure.

In the alloy Ti-42Al-8.5Nb, the orthorhombic O-phase forms besides the $\alpha_2$ phase and $\gamma$ phase that exist in binary Ti-42Al alloys. The O-phase forms from the $\alpha_2$ parent phase in the temperature range between 450 and 650°C and it is stable between room temperature and 700°C [1]. During annealing, the shape, composition and relative volume fractions of the $\alpha_2$-phase and O-phase continuously change over time.

In this presentation, we investigate how the ongoing microstructural changes affect the mechanical properties of the alloy. Using Transmission Electron Microscopy we monitor the evolution of the microstructure within $\alpha_2$ lamellae of lamellar ($\alpha_2$ +$\gamma$) colonies during annealing at 550°C for up to 5000 h. This is complemented by in-situ Resonant-Ultrasound-Spectroscopy measurements of the elastic moduli during annealing of separate samples at 550°C for 672 h.

The results show a continuous increase of the alloy's overall elastic moduli. Thereby, a displacive transformation at the beginning of the annealing experiment has a much stronger impact on elastic moduli than the subsequent diffusion assisted phase separation into $\alpha_2$ and O-phase during the remaining annealing time [2].

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# The Role that Hot Deformation Plays in Determining the Static and Dynamic Mechanical Behavior of a High Niobium Containing γ-TiAl Alloy at Elevated Temperature

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The elevated temperature fatigue behavior of a high Nb containing γ-TiAl alloy is related to the degree of metal working the billet receives during extrusion. In this study, we conducted high cycle fatigue tests on a γ-TiAl alloy containing 6 at% Nb at 450°C and 815°C. Prior to fatigue testing, the Ti-Al ingots were hot extruded at various extrusion ratios. We found the fatigue life was noticeably improved as we increased the extrusion ratio from 10:1 to 25:1. As-extruded billets featured a fully lamellar microstructure. Metallographic analysis showed no discernible difference in lamellae spacing and colony size. Fracture surface of the fatigue samples was inspected to help understand the fracture behavior of the highly strained γ-TiAl alloy. The improvement of fatigue life at elevated temperature is likely attributed to more intensive metal work as extrusion ratio was increased. Fatigue life improvement is found to be more profound at 450°C than 815°C. This is evidence showing the improved fatigue strength is likely a result of work hardening. As fatigue test temperature increases, the material recovers from work hardening. Another possible reason for improved fatigue life is the complete closure of cast voids at high extrusion ratio. Tensile tests were also conducted at 450°C and 815°C at various extrusion ratios. We found both the yield strength and ultimate tensile stress were improved as we increased the extrusion ratio from 10:1 to 25:1. In conclusion, increasing extrusion ratio is proved to be an effective way to improve the fatigue and tensile properties of the high Nb containing γ-TiAl alloy.

# Thermal stability in microstructure of Mn containing $\beta$-solidifying $\gamma$-TiAl alloy

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Advanced intermetallic $\beta$-solidifying $\gamma$-TiAl based alloys with low density, good hot workability and high specific strength/modulus, are known as promising candidates for aerospace and automobile industries. However, the existence of large amount $\beta$ stabilizing elements, such as Mo, Cr, Nb Mn, in these alloys will make the ordered $\beta$ phase (B2/$\beta_o$) retain in the room temperature with the volume fraction even above 20 vol.%. The retained B2 phase is generally quite detrimental to the ambient or elevated mechanical properties due to its brittlement characteristic at room temperature and easily decomposition tendency into some harmful precipitates at service temperature(700~900°C). Meanwhile, the $\alpha_2/\gamma$ lamellar structure is also proved to be thermodynamically unstable due to the metastable $\alpha_2$ lamellae and the large thermal expansion difference of α2 and γ, and the type of lamellar decomposition reaction will largely depend on the alloy composition. In this study, we choose a typical low-cost $\beta$-solidifying $\gamma$-TiAl based alloy with the composition of Ti-42Al-5Mn (at.%) whose volume fraction of retained B2 phase is about 20 vol.% for the as-casted microstructure. The phase evolution behaviors of the retained B2 at the temperature range of 650-950°C were systematically investigated. The transformation types and the composition and structure were clarified with X-ray diffraction (XRD), electron probe micro analyzer (EPMA), advanced electron backscatter diffraction (EBSD), transmission electron microscope (TEM). The effects and mechanisms of Fe, Zr, Nb, W, Mo additions on the thermal stability and tailored of the retained B2 in the Ti-42Al-5Mn alloy were further studied. And on this basis, we invented a novel alloy with the nominal composition of Ti-44Al-3Mn-0.8Mo-0.1B-0.1C (named TMM), which can be forged or rolled directly from the ingot without the pre-forging and near-isothermal canned conditions and whose B2 phase is thermodynamically stable even exposure at 800°C for 3000h. Finally, the lamellar decomposition reactions and the effects of Ta, Hf additions on its the thermal stability of TMM alloy were investigated during exposure at 750~800°C. We expect this work will provide a strong technical support to optimize the microstructure of the low cost containing $\beta$-solidifying $\gamma$-TiAl alloy, and to provide fundamental data, knowledge and information (DKI) for the advanced intermetallic $\beta$-solidifying $\gamma$-TiAl based alloys development.

**Key words:** Titanium aluminides; $\beta$-solidifying; B2/$\beta_o$; lamellar structure; phase transformation; microstructural evolution.

# Oxidation Behavior of TiAl Alloys, its Influence on the Mechanical Properties and Mitigation Strategies via Coatings

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Two representatives of the intermetallic γ–based TiAl alloys, namely the GE 4822 as well as the TNM-B1 alloy, have already found application in the LPT section of today´s aero engines. Their current service temperature is limited to 700-750°C, above that temperature range an unprotective mixed oxide scale grows too fast, that consists of $TiO_2$ and $Al_2O_3$. However, oxidation below 700°C can already affect the mechanical properties of TiAl negatively although mass gains are very low in this temperature range. Since most existing studies investigate the oxidation behavior of TiAl at temperatures well above the current service temperature, comparative isothermal (600-900°C, dry air) and cyclic exposures (700°C, dry/wet air) of the GE 4822 and TNM-B1 alloys have been conducted for up to 1000 h. Four-point-bending tests at room-temperature revealed a successive decrease in the fracture strain values with increasing temperature and duration of exposure for both alloys. Emphasis was placed on the different oxide scale morphologies as well as the microstructural degradation of the subsurface zone which already appeared at 700°C (Fig. 1). Not only the existence of an oxygen-enriched Al-depleted layer at the interface between the oxide scale and the substrate seems to affect the mechanical properties of the alloys, but also the increase of the $\alpha_2$ phase at the expense of the β–phase that decomposes in the subsurface zone. Finally coating strategies are compared and discussed to suppress or delay the detrimental subsurface degradation.

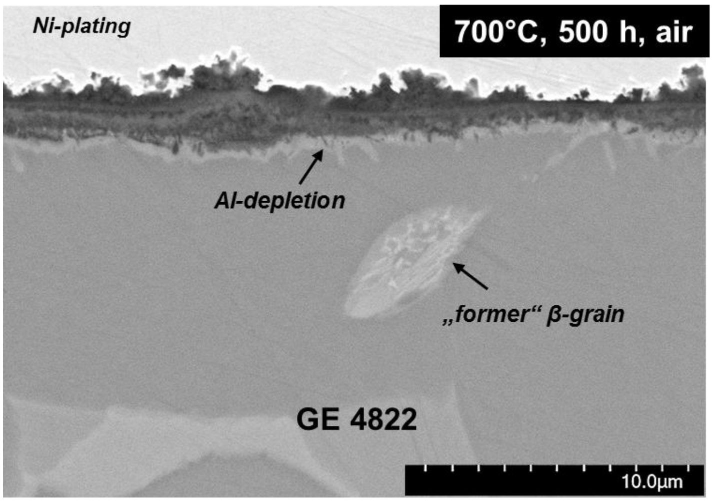


**Fig. 1:** BSE image of the GE 4822 alloy (HIPed condition) after isothermal oxidation for 500 h at 700°C in dry air.

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# Deposition of $Ti_2AlC$ MAX-phase based coating on Titaniumaluminides to improve the oxidation resistance

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Currently, the application of intermetallic γ-TiAl alloys is limited by their deterioration in strength and creep resistance at elevated temperatures as well as by reduced oxidation resistance above 800 °C. The deposition of protective coatings is a promising opportunity to enhance the oxidation resistance by several orders of magnitude. Moreover, additive manufacturing processes have enabled the production of components with more complex geometries. Thus, a design of turbine blades made of γ-TiAl with internal cooling will be feasible in the near future. In this context, the deposition of thermal barrier coatings and, therefore, protective bond coatings become important.
MAX-phases are of increasing interest as material for high temperature applications due to their unique combination of metallic and ceramic properties. Especially the alumina forming MAX phases $Cr_2AlC$, $Ti_2AlC$ or $Ti_2AlN$ are promising as oxidation resistant coatings. Unfortunately, degradation of MAX phases is observed when applied on various Ti- or Ni-based alloys due to interdiffusion processes between coating and alloy resulting in Al-depletion of the coating. This degradation is not present when MAX-phases are applied on the Al-rich γ-TiAl based alloys, which leads to a diffusion of Al from the substrate alloy into the coating and thus to a stabilization of the thermally grown alumina layer. Moreover, MAX-phases are known as a ductile material and could therefore prevent the deterioration of the mechanical properties especially the fatigue behavior of such coated components in contrast to the common intermetallic, protective but brittle coatings on γ-TiAl alloys.
In the present work, a $Ti_2AlC$ MAX-phase based coating was deposited by DC magnetron sputtering. Using three pure elemental target materials of Ti, Al and C and a two-fold sample rotation a homogenous all-around coating with a coating thickness of 10 µm. was applied on the γ-TiAl alloy TiAl48-2-2. After the deposition process, the stoichiometric $Ti_2AlC$ coating was X-ray amorphous. Therefore, a post-heat treatment at 800°C for 1 h was performed to achieve the desired hexagonal MAX-Phase. Finally, the MAX-phase coated TiAl48-2-2 alloy was subjected to cyclic oxidation tests at 850°C in laboratory air for up to 100 1 h-cycles.
The $Ti_2AlC$ MAX-phase coated TiAl48-2-2 alloy exhibits an excellent oxidation behavior at 850°C, due to the formation of a thermally grown alumina top layer. The phase formations during the heat treatment were analysed by HT-XRD measurements indicating a homogenous coating microstructure of the hexagonal $Ti_2AlC$ phase below an $\alpha$-$Al_2O_3$ oxide layer. The interface reaction zones between the coating and the TGO, as well as between the coating and the TiAl48-2-2 substrate alloy were analyzed by SEM with EDS and especially by high-resolution STEM.

# Influence of oxidation protective coatings on the high temperature fatigue behaviour of Ti-48Al-2Cr-2Nb

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With their excellent weight-specific properties and good oxidation resistance, intermetallic Titanium Aluminides are suitable candidates to replace heavier nickel based super alloys in high temperature applications. Ti-48Al-2Cr-2Nb is one promising example, which is already in use for low-pressure turbine blades. While its duplex microstructure is stable up to 850 °C, the oxidation resistance drastically decreases between 650 °C and 750 °C. Hence, oxidation protective coatings must be applied to further increase the application temperature. However, these coatings are mostly brittle, which may promote fatigue crack initiation and therefore reduce the fatigue lifetime. To investigate the influence of oxidation protective coatings on the high temperature fatigue behavior of 48-2-2, uncoated, pre-oxidized and coated plus pre-oxidized samples were push-pull cycled under stress control at 750 °C. A TiCrAlY and a TiCrAlYSi coating were applied via Magnetron Sputtering and Closed Hollow Cathod - Physical Vapour Desposition respective. The pre-oxidation was conducted in air at 850 °C for 300 h. The results show that at higher stress amplitudes, pre-oxidized as well as coated and pre-oxidized samples fail early. With decreasing stress amplitude, in a narrow transition of about 15 MPa, the lifetime of coated samples increases drastically. When the stress amplitude is in the range of the fatigue strength, both coatings as well as pre-oxidizing had only a minor influence on the fatigue lifetime. The fatigue behavior of the different sample types is discussed based on damage observations and the implications for service application of the coatings are analyzed.

# Development of Microstructure Factor-based Mechanical-property Prediction Module for TiAl Alloys

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A mechanical properties prediction module (MPM) for TiAl alloys has been developed as a part of an integrated computational inverse problem approach to accelerate the development of TiAl low pressure turbine blade in jet engines and reduce the extensive experimental efforts. The input into this module is specific values of the properties required by industries, say, tensile strength (UTS) and fracture toughness ($K_{IC}$). Then the output is a concrete value, such as volume fraction of the microstructural constituent governing the property. In order to get great performance out of this module, the relationship database (DB) between microstructure and mechanical properties and calculation model that describe mechanical properties as a function of microstructure factor are needed. Therefore, this presentation exhibit how these DB and model have been constructed.

The construction of DB was carried out using a model alloy that is uniquely-designed ourselves. Heat treatments were conducted for this alloy at given temperatures and time based on our sophisticated phase diagram knowledge for microstructure control. Here, there are two important microstructural constituents in the alloys: one is the lamellar microstructure consisting of $\alpha_2$-$Ti_3Al$and $\gamma$-TiAl phases and the other the cellular microstructure consisting of $\beta$-Ti and $\gamma$ phases, since we revealed the introduction of the cellular microstructure is effective in increasing the mechanical properties. Then, volume fraction of this $\beta/\gamma$ cellular microstructure ($V_c$) was quantitatively analyzed from back-scattered electron images took from a scanning electron microscope by an image analysis software. Mechanical properties were evaluated by conventional tensile and three-point bending test using round-bar shape and chevron-notched bar specimens, respectively.

The calculation models were developed as follows:

$$\sigma_{UTS}=\sigma_{UTS}^{\alpha_2/\gamma}+\Delta\sigma_{UTS}=\sigma_{UTS}^{\alpha_2/\gamma}+(\Delta\sigma_{0.2}\ _{V_c}+\left(\frac{d\sigma}{d\varepsilon}\ _{V_c}\cdot(\varepsilon)_{V_c}\right.$$

$$K_{IC}=K_{IC}^{\alpha_2/\gamma}+\Delta K_{IC}=K_{IC}^{\alpha_2/\gamma}+\alpha(\Delta E_i\ _{V_c}$$

These models are characterized by these can be described using the terms of the properties of $\alpha_2/\gamma$ lamellar microstructure and deviation from it, that is the effect of introduction of $\beta/\gamma$ cellular microstructure. By using these models, UTS and $K_{IC}$. can be predicted have a margin of error of ±5% in UTS and ±20% in $K_{IC}$. Therefore, the volume fraction of the microstructure constituent to meet the required properties can be calculated as an output value of this module, then, it will become an input value into microstructure design module for calculation of the alloy composition and heat treatment conditions for meet satisfy the required properties.

This work was supported by Council for Science, Technology and Innovation (CSTI), Cross-ministerial Strategic Innovation Promotion Program (SIP), “Material Integration” for revolutionary design system of structural materials.

# Abstracts

## Posters

## (*in alphabetical order of the person presenting it*)

# Morphology and formation mechanism of titanium boride in Ti-45Al-2Mn-2Nb-*x*B

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**Abstract**

γ-TiAl alloys have attracted much attention for their high specific strength and good mechanical properties at elevated temperature. Boron is added to refine the lamellar grain size, which is believed to improve the poor room temperature ductility of TiAl alloys. However, it may degenerate the mechanical properties due to the formation of borides with curvy morphology and large size, which are severely influenced by the alloy composition and processing parameters. The morphology and crystal structure of borides in Ti-45Al-2Mn-2Nb-*x*B (at.%) alloys containing different amount of B have been investigated to reveal the relationship between B content and boride formation. In alloys containing 0.25 at.% and 0.5 at.% B, boride particles are curved and flake-like, and are distributed at β dendrite boundaries, resulting from late nucleation and constrained by matrix during growth. Curved, ribbon-like boride particles are observed to randomly distribute in alloy containing 0.75 at.% B, and insufficient behind-time supply of B atoms to the growth front is believed to be responsible for their irregularity and large curvature bending and branching. In alloys containing 1.0 at.% or higher B, straight needle/rod-like boride particles dominate, and a small number of blocky particles appear in alloys containing 1.25 at.% and 1.5 at.% B. Most boride particles are of C32-$TiB_2$ and have their major axes along [0001] or [1$\bar{2}$10] in all alloys studied in this work, and $B_f$-TiB and $D7_b$-$Ti_3B_4$ randomly appear in conjunction with C32-$TiB_2$ to form intergrowths, while B27-TiB particles are free standing and only appear in alloys containing 1 at.% or higher B.

***Keywords*:** γ-TiAl, borides, solidification, formation mechanism, morphology

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# On the phase transformation sequence during rapid solidification and cooling in the Ti-48Al-2Cr-2Nb alloy

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In recent years, additive manufacturing has emerged as a useful alternative to conventional manufacturing techniques due to its numerous benefits, e.g. the generation of near-net-shape parts or the implementation of complex structures such as cooling channels [1,2]. Consequently, the process is considered for the production of turbine blades made of intermetallic γ-TiAl based alloys. At the moment, research is focused on two major methods – electron beam melting and laser powder bed fusion [1,2]. Using these techniques, high cooling rates are applied, leading to out-of-equilibrium phase transformations upon solidification and cooling. Due to the inherent heat treatment during additive manufacturing, the original solidification microstructure is, however, oftentimes not apparent after the process and thus not well studied yet. The focus of this work is, hence, to explore the solidification pathway and the phase transitions that occur upon cooling in the Ti-48Al-2Cr-2Nb (at.%) alloy via *in situ* high-energy X-ray diffraction (HEXRD) at a synchrotron facility. For the experiment a small experimental powder bed system was employed [3]. However, in this case, the powder bed was replaced by a thin platelet, which was single-track fused along the upper edge with a laser [3,4]. In this way, the disturbing influence of solid-phase signal generated by non-fused powder particles on the diffraction patterns was avoided. An exemplified result of such an experiment conducted with a detector frame rate of 250 Hz is given in Fig. 1. From the stacking of the azimuthally integrated diffraction patterns along the time axis of the experiment, the primary solidification phase could in this case be clearly identified as β, see Fig. 1. This experiment was corroborated by microstructural studies as well as finite element analysis.

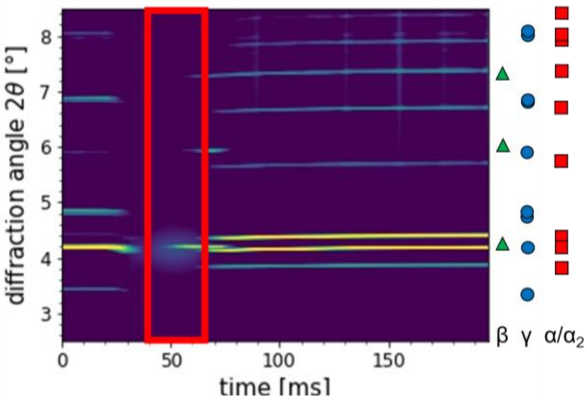


Figure 1: Phase evolution during laser fusion experiment determined by means of *in situ* HEXRD (β: β-Ti(Al); γ: γ-TiAl; $\alpha/\alpha_2$: α-Ti(Al)/$\alpha$-$Ti_3Al$).

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# Fatigue Crack Growth Behavior of TiAl Based Alloys based on Compact Tension Test at Elevated Temperatures

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Our previous study on fatigue crack growth (FCG) behavior of wrought TiAl alloys using compact tension (CT) test at room temperature (RT) revealed that the introduction of bcc β-Ti phase along the $\alpha_2/\gamma$ lamellar colony grain boundaries toughens the materials, that is, increasing the stress intensity factor threshold ($\Delta K_{th}$) and reducing Paris slope $m$ (crack growth rate: $da/dN$), superior to those of the conventional alloys (L. Signori et. al, Intermetallics, vol. 100 (2018), pp. 77-87). In this study, thus, the FCG behavior at elevated temperatures was examined in air and Ar atmosphere, in order to understand the underlying principle of crack initiation/propagation mechanisms of the alloys. The microstructure of the specimens is similar to the previous ones with β phase along the lamellar boundaries. The β phase was introduced using the cellular reaction ($\alpha_2+\gamma\rightarrow\beta+\gamma$) through multi-step heat treatments. A pre-crack was first introduced to each CT specimen at RT, and FCG tests were done at RT, 873 K and 1073 K under 20 Hz with a tension/tension load ratio $R$=0.1. The elevated-temperature tests were conducted in a uniquely designed environment-controllable chamber with heating elements. In case of the tests in Ar, the chamber was evacuated and backfilled with Ar flowing. Crack length was measured by DCPD (direct current potential drop) method.

The $\Delta K_{th}$ obviously decreases at 873 K in both atmospheres. However, it goes up to the same value or even better at 1073 K in air whereas it stays low in case of Ar atmosphere. The $m$ value is nearly the same, regardless of the test conditions, but the $da/dN$ value becomes apparently low by an order of magnitude at the same $\Delta K$ value. We confirmed that almost no oxide was formed in the cracks whereas thick oxide layer was formed in the sample tested in air. From these results, it should be suggested that the FCG behavior obtained in Ar atmosphere is the nature of the alloys, and the behavior is highly affected by the oxide formation. The underlying mechanism of FCG will be discussed, in conjunction with the microstructure analyses of the crack tips.

A part of this study was carried under the research of SIP in JST (Japan Science and Technology Agency).

# Influence of grinding depth on the surface integrity and fatigue properties of γ-TiAl alloys

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Due to their low ductility and fracture toughness at room temperature, γ-TiAl alloys are very difficult to machine, and their mechanical properties are very sensitive to the surface conditions, so it is necessary to study the surface integrity and relate it to fatigue properties of TiAl alloys after surface grinding of cast components. In this study, cast plates of Ti-45Al-2Nb-2Mn-1B alloys were ground with different depth, their surface integrity, such as surface roughness, microstructure, microhardness, and corresponding fatigue properties were compared. Cracks were detected in samples ground with 0.5 mm and 1 mm depth, but not in samples ground with 0.2 mm depth or below. With increasing grinding depth, the number and depth of groove increased, and surface roughness parameters Ra and Rz increased, while Rsk decreased. The $\gamma+\alpha_2$ lamellae bent in the surface layer, and the bent layer thickness increased with increasing grinding depth. The microhardness decreased first but increased from the surface to the interior. The rotating bending fatigue life at 650 °C under a load of 440 MPa decreased with increasing grinding depth: larger than $10^6$ cycles when the grinding depth was 0.05 mm, but dropped to about $10^4$ cycles when the grinding depth was 0.2 mm. Fracture surface analysis on the latter showed that the cracks mainly nucleated at the grooves on the surface, resulting from the stress concentration induced by the grooves. The fatigue life decreased with increasing Rz, and it kept above $10^6$ cycles when Rz was smaller than 4 μm. A nonlinear relationship between fatigue life and Rz was revealed.

# Forging process of a large TiAl alloy ingot without envelope

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TiAl alloys have low thermo-plasticity, high deformation resistance, and a very narrow window of thermal deformation parameters. It is necessary to realize thermal deformation under near-isothermal conditions under complicated metal envelope isolation. How to improve the yield of products at an acceptable cost is the focus of today's technology developers. This study aims to provide a convenient TiAl alloy forging method, by using glass lubrication powder high temperature adhesive quick fit insulation fiber cotton to replace envelope. The design of metal insulation envelope was thus removed, improving the dimensional accuracy and preparation efficiency of the product. Large TiAl alloy ingots with uniform deformation and fine grain size were obtained by isothermal and low rate deformation.

# Embrittlement of γ-TiAl by oxygen diffusion

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TiAl alloys are sensitive to embrittlement by exposure in air at temperatures around 700°C and for durations as low as one hour. Though this effect is important for the development of the TiAl alloys in hot parts of automotive and aeronautical engines, it remains poorly understood. Thus, we address the questions of the penetration kinetics of oxygen in a Ti-48Al-2Cr-2Nb alloy with a near γ microstructure, and its impact on tensile properties at room temperature. For this purpose, tensile specimens have been exposed in a 20 % $O_2$ + 80 %Ar atmosphere, for temperatures ranging from 400°C to 700°C. The tensile tests show that the embrittlement phenomenon is characterized, in addition to the well-known ductility loss effect, by a significant increase in yield stress. The origin of this increase has been investigated in details looking at microscopic deformation mechanisms, by transmission electron microscopy. In particular, the dislocation morphologies have been determined in samples of different oxygen concentrations. The diffusion coefficient of oxygen in the γ phase has been calculated theoretically by DFT to quantify the oxygen penetration kinetics. In parallel, experimental values have been obtained for the first time, using $^{18}O$ isotope and SIMS concentration profile analyzes. Surprisingly, the experimental diffusion kinetics were significantly lower than those theoretically predicted. This discrepancy is discussed in terms of potential effects, based on DFT calculations, of the Nb and Cr substitutional solutes on the interstitial diffusion mechanisms of oxygen. This study will therefore present key results enabling to better understand the impact of oxygen dissolution and diffusion in the γ phase on the origin of the increase in yield stress, and its correlation with the embrittlement phenomenon.

# Joining of Ti-42Al-5Mn and S65007 by resistance brazing

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In the practical application of TiAl to automobile industries, the joining of TiAl and structural steels with excellent wear resistance and toughness are required. Ti-42Al-5Mn and S65007 were joined by resistance brazing using the active silver-based filler metal of AgCuInTi and AgCuTi filler alloys, respectively. Brazing was performed in the temperature range of 800 to 900ºC for 30 s by using a Gleeble-3500 thermal simulation testing machine. The tensile strength of the joints was determined and the microstructure and the chemical composition of the interfaces were studied by scanning electron microscopy (SEM) and by Electron probe microanalysis (EMPA), respectively.

**Key words:** TiAl alloy; S65007; resistance brazing; silver-based filler; tensile strength;

# Influence of Ni Powder Addition in Sintering Behavior of TiAl Alloy Powder

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Recently, the development of jet engine parts made of TiAl by Additive Manufacturing (AM) using alloy powder has been promoted, and Metal Injection Molding (MIM) is also attracting attention because of its freedom of design. In the MIM method, the sinterability of the powder is most important. Therefore, in this study, the effect of Ni addition on the sinterability of TiAl alloy powders was investigated by adding Ni powder, which has the effect of lowering the melting point of Ti, and the mechanism of enhancing the sinterability was investigated.

The specimen was compacted TiAl alloy powder of ϕ28 mm by t5 mm, which was prepared by EIGA (powder particle size d < 45 μm) and mixed with various amounts of Ni powder in a resin binder. They were subjected to various sintering heat treatments under Ar atmosphere, and density measurements and structural observations were carried out. Some samples were subjected to DTA to investigate their behavior during sintering and tensile tests to evaluate the integrity of the material after sintering.

Fig. 1 shows the effect of Ni addition on the relative density of sintered materials. The relative density increases sharply with the addition of about 0.5 wt% of Ni, and this tendency appears on the lower Ni side as the sintering temperature increases. According to the results of the post-sintering structural analysis, in the case of a small amount of Ni addition (0.1 wt% Ni), there are many voids of about 10 μm at the lamellar colony grain boundaries, but when the amount of Ni addition is 0.6 wt% or more, the voids decrease significantly, resulting in a larger lamellar grain size (Fig. 2). The less voids, the higher the tensile strength. Therefore, the addition of Ni has a significant effect on improving the sinterability. From the DTA analysis, the phase diagram of the alloy system and the diffusion coefficients in each phase, the improvement of sinterability by the addition of Ni is not due to the formation of the liquid phase but to the enhancement of the formation of the beta-Ti phase with a large diffusion coefficient. This study was supported by the Cross-Ministerial Strategic Innovation Promotion Program (SIP) "Structural Materials for Innovation" from the Japan Science and Technology Agency (JST).

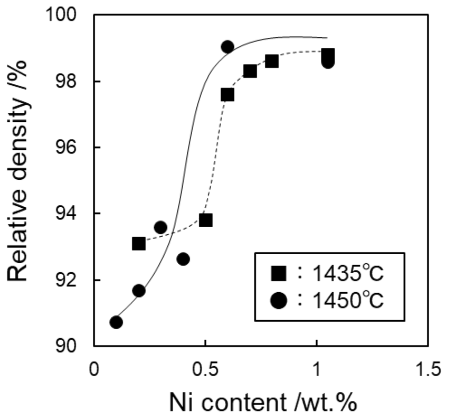


Fig.1 Effect of Ni addition on relative density of specimens sintered at 1435°C and 1450°C.

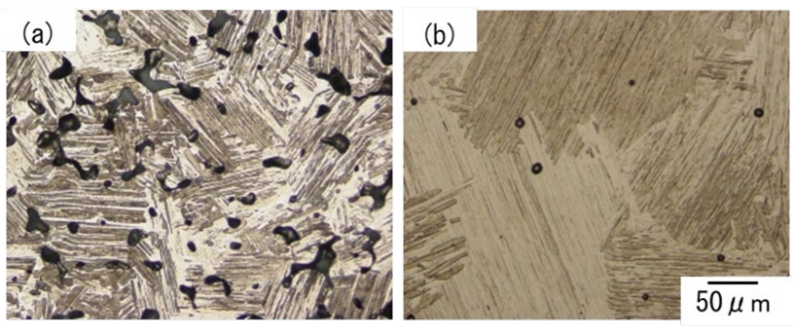


Fig.2 Microstructures sintered at 1450°C: (a) 0.1Ni, (b) 0.6Ni.

# Phase Equilibria Between $\alpha$-Ti/ $\alpha_2$-$Ti_3Al$ Phases near 1473 K in TiAl Alloys Containing Oxygen

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TiAl alloys are characterized by the $\alpha_2/\gamma$ lamellar structure formed by the decompositions of the high-temperature $\alpha$ (hcp) phase ($\alpha \rightarrow \alpha_2$ ($D0_{19}$)+$\gamma$-TiAl ($L1_0$)). Microstructure design using this phase transformation greatly controls the mechanical properties. Especially in the powder process, contamination with oxygen is unavoidable. Thus, understanding of the phase equilibria among TiAl-based alloys containing oxygen is very important to control the microstructure.

In the previous study, we clarified the effects of oxygen on the phase equilibrium between $\alpha/\gamma$ phases at 1473 K by analyzing the oxygen in each phase by EPMA equipped with a soft X-ray emission spectroscopy. The phase was uncertain and it is necessary to figure out whether $\alpha$ or $\alpha_2$ phase is equilibrated with $\gamma$ phase in this study because even a very low content of oxygen may affect the stability of $\alpha$ phase and $\alpha_2$ phase. In the Ti-Al binary phase diagram, the phase transition temperature of $\alpha/\alpha_2$ is 1448 K, which is only 25 K lower than 1473 K. If oxygen is $\alpha_2$ phase stabilizing element, $\alpha_2$ exists at 1473 K. If oxygen is $\alpha$ phase stabilizing element, then $\alpha$ phase exists at 1473 K.

In this study, we explored the presence of $\alpha/\alpha_2$ phases at 1473 K by using TEM to observe the presence of APD. Samples with oxygen content at 0.2 and 1.0 at.% were observed and found that APD with a size of approximately 100 nm is present in the 0.2O sample but not in the 1.0O sample. That means the 0.2O sample has the ordering process from the $\alpha$ phase to the $\alpha_2$ phase during water cooling from 1473 K, while the 1.0O sample already has the $\alpha_2$ phase in 1473 K. Therefore, oxygen has the effect of stabilizing the $\alpha_2$ phases.

Phase diagram calculation software named Pandat was used to calculate and the data of the Ti-Al-O interaction factor (${}^{0}L_{\mathrm{Al:Ti:O,\ Va}}$) and Al-Al-O interaction factor (${}^{0}L_{\mathrm{Al:Al:O,\ Va}}$) of $\alpha_2$ phase were changed on the basis of the original database (DB), and a ternary phase diagram that can reproduce the experimental results to a certain extent was successfully obtained. Although there is still room for improvement, the $\alpha_2$+$\alpha$+$\gamma$ three-phase region in our calculation results reflects the experimental results better than that from the previous DB.

# Effect of Microstructure on Oxidation Behavior of TiAl Alloys at 1023 K

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Titanium-aluminide (TiAl) alloys are one of candidate materials for low pressure turbine (LPT) blades of jet engines. Microstructure of TiAl alloys consists of various phases such as $\alpha_2$, $\gamma$ and $\beta$ and it can be controlled by alloying elements and heat treatments. Optimization of microstructure is very important to obtain further excellent mechanical properties. There is a useful design concept for obtaining suitable microstructure. However, there are few attempts to clarify the effect of microstructure on oxidation behavior. This study tries to make clear the relationship between microstructure and oxidation behavior of TiAl alloys at lower temperatures. Various TiAl alloys with chromium (Cr) and/or niobium (Nb) and/or oxygen (O) were oxidized at 1023 K in ambient air for 360 ks (100 h) to evaluate oxidation resistance and to clarify the effect of microstructure on oxidation behavior. TiAl alloys with both Cr and Nb show good oxidation resistance in this experimental condition and Nb addition into the alloys is beneficial to obtain better oxidation resistance compared with the addition of Cr. Oxidation resistant is also slightly improved by O addition into the alloys. In this study, the effect of microstructure on oxidation behavior of these alloys will be discussed based on various parameters, such as volume fraction, grain size and lamellar distance, which are obtained from microstructure observation.

# Toughness bottleneck of γ-TiAl alloy via integrated computation

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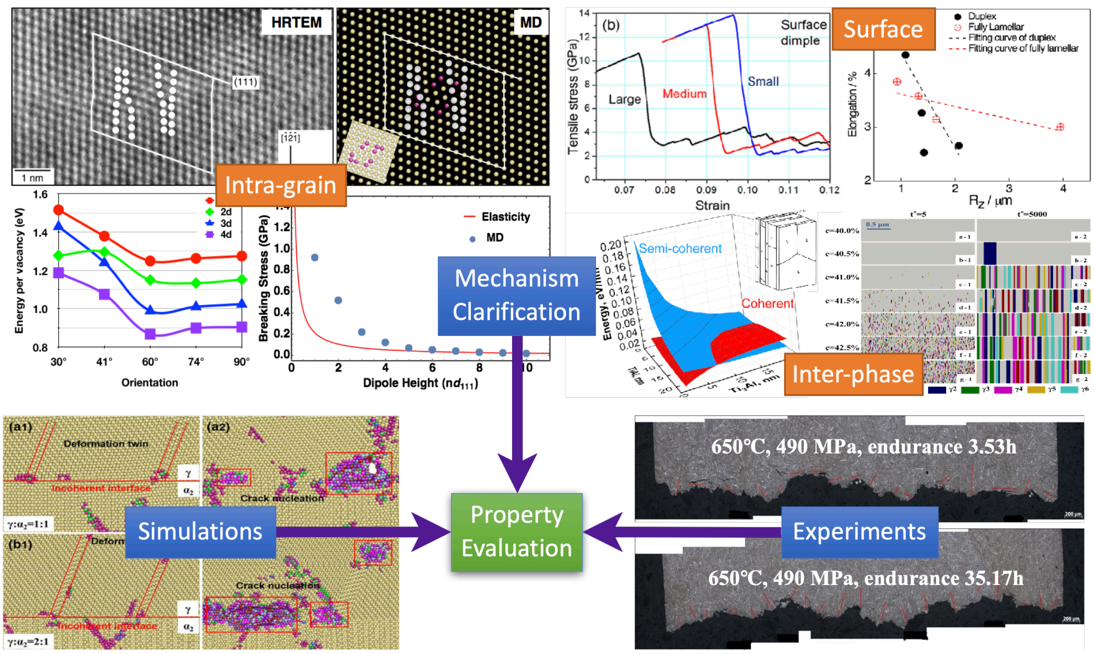
The present work employs integrated computation on various scales to achieve a comprehensive and quantitative understanding of the toughness bottleneck of γ-TiAl alloy, aiming at clarifying the atomic-scale origin and providing potential solutions for the improvement of the overall performance.

1) Intra-grain cracks are not likely to occur through dislocation interactions. The critical annihilation distance of dipolar dislocations significantly differs from traditional estimation and faulted dipoles can be stable over experimental timescales, in full agreement with high-resolution observations.

2) The ductility and fatigue toughness are sensitive to surface defects. The defects on surfaces and edges cause weakening with various effects depending on defect type, size, position and orientation, while the edge dimples are the most influential. The effects of surface scratches are orientation and shape sensitive.

3) The fracture toughness is sensitive to the $\gamma/\alpha_2$ interfaces. The coherency of $\gamma/\alpha_2$ interfaces depend on the thickness ratio of γ lamellae to $\alpha_2$ lamellae, and there exists a critical lamella thickness, below/above which the interface is coherent/semi-coherent. Both interfaces exhibit low fracture toughness against perpendicular loading.

*Keywords*: TiAl; Dislocation dipole; Phase interface; Surface; Mechanical behavior



# Deep understanding of phase equilibria and CALPHAD modelling in Ti-Al-X (X=Nb, Mo, W, Si, Zr, O, B, C) systems

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Design of new TiAl alloys for turbine blades is crucial in order to meet the goals to reduce $CO_2$ and $NO_x$ emissions from turbine jet engines while increasing their efficiency. Many investigations have been carried out by a variety of experimental techniques in Ti-Al-X systems and it has been reported that adding Nb, Mo, W, Si, Zr, B, C is beneficial to improve the ductility, fracture toughness and oxidation resistance in TiAl alloys. In view of alloy development to optimize the microstructures in these alloys, quantitative data on phase equilibria involving $\beta/\beta_o$ (Bcc_A2 or Bcc_B2), $\gamma$ (TiAl: $L1_0$), $\alpha_2$ ($Ti_3Al$: $D0_{19}$) and $\alpha$ (Hcp_A3) would be essential.

Due to the inevitable experimental challenges (unquenchable phase transformations, impurity contamination, *etc.*), available data on phase equilibria in the ternary systems Ti-Al-X (X=Nb, Mo, W, Si, Zr, O, B, C) are often incomplete or contradictory. In order to resolve existing experimental controversies and to develop a more accurate thermodynamic database, a detailed understanding of phase equilibria and CALPHAD modelling in these Ti-Al-X systems have been achieved in the ADVANCE project [1]. The sophisticated experiments performed during the project resulted in strengthened thermodynamic descriptions for the titanium aluminide systems. The significantly improved thermodynamic descriptions have been integrated into the TCTI [2] database so that the calculated results can be readily applied to support the design of new, lightweight, high-temperature structural alloys. This presentation will show the major achievements from the ADVANCE project connected to the development of a state-of-the-art CALPHAD database for TiAl alloys.

[1] ADVANCE: Sophisticated experiments and optimization to advance an existing CALPHAD database for next generation TiAl alloys. This project has received funding from the European Union's Horizon 2020 research and innovation Program under grant agreement No. 820647.

< https://thermocalc.com/about-us/advance/>

[2] Thermo-Calc Software, Titanium and TiAl-based Alloys database, version 5.

< https://thermocalc.com/products/databases/titanium-and-titanium-aluminide-based-alloys/>

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