



	<b>Experiment title: <i>In-situ</i> high-temperature characterization of phases, phase transformations and microstructural evolution in novel <math>\beta</math>-solidified <math>\gamma</math>-TiAl alloys</b>	<b>Experiment number:</b> MA-490
<b>Beamline:</b> ID15B	<b>Date of experiment:</b> from: 11/07/2008 to: 15/07/2008	<b>Date of report:</b> 11/02/2009
<b>Shifts:</b> 12	<b>Local contact(s):</b> Thomas Buslaps	<i>Received at ESRF:</i>
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## Introduction

Intermetallic  $\gamma$ -TiAl based alloys are increasingly important for high-temperature applications in aerospace and automotive industries [1]. The advantages of these materials are mainly low density, high specific yield strength, stiffness, and good creep properties as well as favorable oxidation resistance. Knowledge of the constituent phases and their solid-state transformation temperatures is the basis for customized heat treatments, which are conducted to optimize the mechanical properties. Thus, for the development of advanced alloys the influence of alloying elements, heat treatments and processing parameters on volume fraction and thermodynamic stability of the phases has to be entirely understood.

For about one decade, high niobium containing  $\gamma$ -TiAl based alloys with a chemical composition of Ti-(42-45at.%)Al-(5-10at.%)Nb have attracted scientific and technological attention. Conventional high-niobium bearing alloys show a relatively strong tendency to form segregations due to the peritectic solidification via the  $\alpha$ -phase. This effect leads to anisotropic microstructures with local heterogeneities as well as significant textures and segregations, which aggravates hot-workability and processing and results in non-reproducible mechanical properties of these materials. New alloy design strategies are aimed to overcome these deficiencies by developing alloys which solidify via the  $\beta$ -phase. These  $\beta$ -solidifying alloys show a more homogeneous and finer microstructure in the cast and extruded state. A possible alloy design strategy to improve the hot-workability of  $\gamma$ -TiAl alloys is to exploit a combination of thermo-mechanical processing and additional alloying to induce the formation of ductile  $\beta$ -phase at elevated temperatures.

The aim of the present study is to gain fundamental knowledge on the prevailing phases and phase transformations in novel  $\gamma$ -TiAl based alloys. As in previous studies, high-energy X-ray diffraction was used as a powerful experimental tool [2-4].

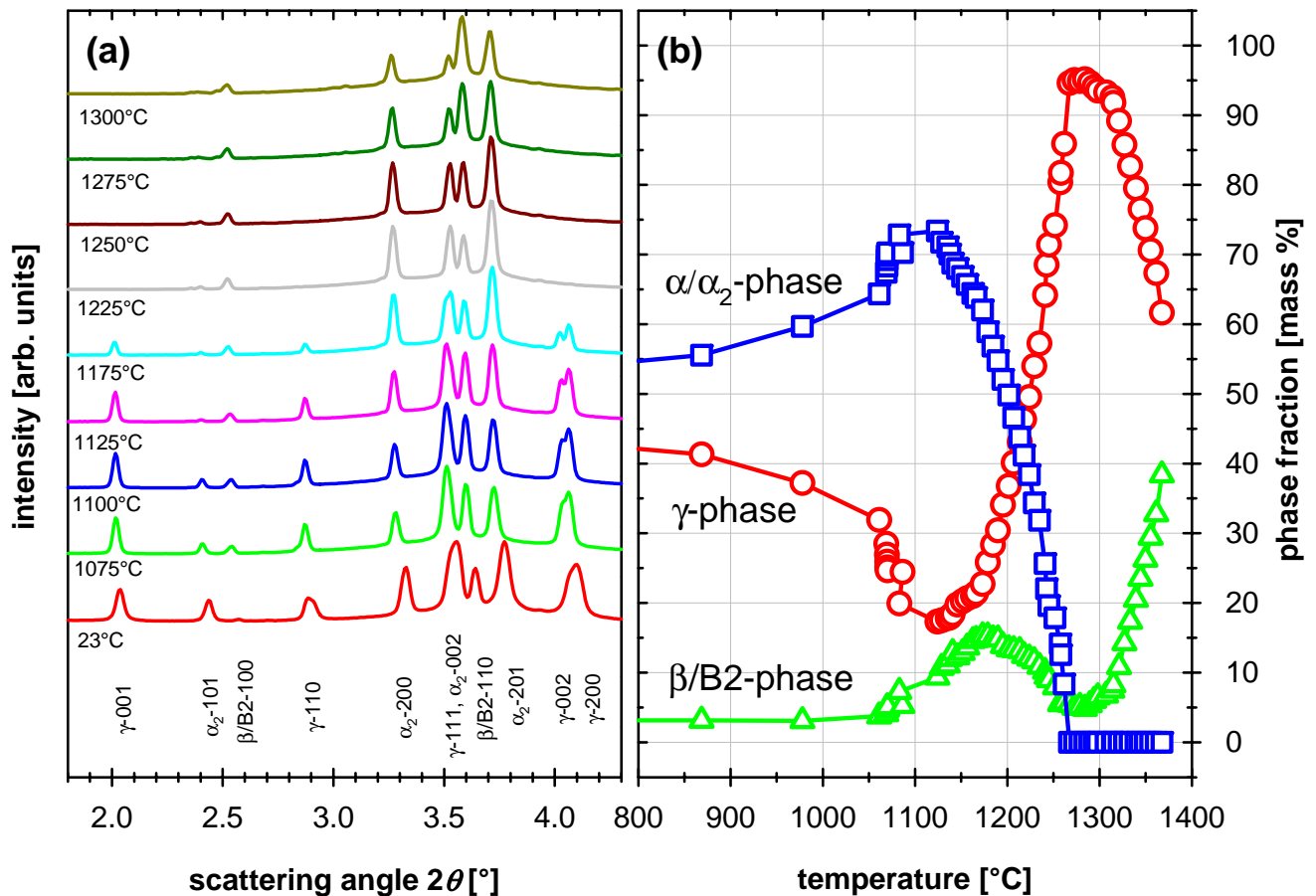
## Experimental method

The *in-situ* high-energy X-ray diffraction studies regarding the solid-state phase transformations of the investigated alloy system and the temperature dependence of the individual phases were performed at the beamline ID15B at the ESRF. For the experiment, a custom-made diffraction furnace was supplied by the ESRF. Heating was performed by high-frequency induction, and the temperature was controlled by two pyrometers. The *in-situ* heat treatments were conducted under constant flow of argon to avoid oxidation of the sample surface. The furnace had an entrance hole for the primary beam X-ray beam and an exit window for the scattered photons equipped with thin foils of polyimide (Kapton) in order to hold the controlled atmosphere. The specimen was continuously rotated during exposure to avoid texture effects and obtain smooth Debye-Scherrer rings. For further details of the furnace see reference [5]. The samples were cylinders with a diameter of 4 mm, which were mounted on a ceramic sample holder for the *in-situ* diffraction furnace. Monochromatic synchrotron radiation with a nominal energy of 86.78 keV and an energy resolution of  $\Delta E/E = 10^{-3}$  was used. The diffraction patterns were recorded continuously during the *in-situ* heat treatment. A two-dimensional area detector Pixium Thales with a resolution of  $2640 \times 1920$  pixels and a pixel size of  $154 \times 154 \mu\text{m}^2$  detected the scattered photons. To avoid overexposure while maintaining acceptable counting statistics, four single images were taken with an exposure time of 4 s and subsequently averaged. A cumulative exposure time of 12 s yielded a diffraction pattern with excellent counting statistics. The measurements showed isotropic Debye-Scherrer diffraction rings, which were azimuthally averaged for equal radial distances from the central X-ray beam.

## Results and discussion

Figure 1a shows azimuthally averaged X-ray diffraction patterns of the alloy Ti-43Al-4Nb-1Mo-0.1B (at.%) for various temperatures from ambient conditions up to 1300°C. In the initial state the diffraction pattern solely contains peaks from the phases  $\alpha_2$ -Ti<sub>3</sub>Al,  $\gamma$ -TiAl and  $\beta$ /B2-Ti. The diffraction peaks of  $\alpha_2$ -002 and  $\gamma$ -111 are overlapping and appear as one single peak. The diffraction peaks of  $\gamma$ -002 and  $\gamma$ -200 are clearly separated due to slight differences in the lattice spacing along the *a* and *c* axes of the tetragonal  $\gamma$ -TiAl crystal unit cell (L1<sub>0</sub> structure). With increasing temperature, the diffraction peaks are shifting towards lower scattering angles due to the thermal expansion of the crystal lattices. The transformation  $\alpha_2 \rightarrow \alpha$  takes place at the eutectoid temperature, which is indicated by the appearance of the disordered  $\alpha$ -Ti phase. The order-disorder transition is accompanied by a reduction of the crystal lattice parameters.

For quantitative evaluation of the prevailing phases, Rietveld analysis of the diffraction patterns was conducted. To this end, the commercially available Topas Software from Bruker AXS was used. Figure 1b shows the phase fraction of the individual phases as function of temperature.



**Figure 1:** Quantitative Rietveld evaluation yielding phase fractions for alloy Ti-43Al-4Nb-1Mo-0.1B (at.%). At 1170°C, the  $\beta$ -phase fraction exhibits a local maximum which indicates the eutectoid temperature.

From the results of these experiments a phase diagram for the alloy system investigated was established. With the information contained in the phase diagram, the heat treatments used for adjusting the mechanical properties were optimized.

### References:

- [1] H. Kestler, H. Clemens, in *Titanium and Titanium Alloys* (Eds: M. Peters, M. Leyens), Wiley-VCH, Weinheim **2003**, 351.
- [2] K.-D. Liss, A. Bartels, H. Clemens, S. Bystrzanowski, A. Stark, T. Buslaps, F.-P. Schimansky, R. Gerling, C. Scheu, A. Schreyer, *Acta Mater.* **2006**, *54*, 3721.
- [3] H. F. Chladil, H. Clemens, G. A. Zickler, M. Takeyama, E. Kozeschnik, A. Bartels, T. Buslaps, R. Gerling, S. Kremmer, L. Yeoh, K.-D. Liss, *Int. J. Mater. Res.* **2007**, *98*, 1131.
- [4] H. Clemens, H. F. Chladil, W. Wallgram, G. A. Zickler, R. Gerling, K.-D. Liss, S. Kremmer, V. Güther, W. Smarsly, *Intermetallics* **2008**, *16*, 827.
- [5] S. G. Eeckhout, B. Gorges, L. Barthe, O. Pelosi, O. Safonovaa, G. Giulic, *J. Synchrotron Rad.* **2008**, *15*, 489.