

# The order/disorder transformation of $\beta$ phase in binary and ternary $\gamma$ TiAl based alloys studied by synchrotron and neutron diffraction

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## Introduction

Due to their high melting point, low density, and good oxidation resistance,  $\gamma$ -TiAl based alloys have recently started to replace Ni-based superalloys as a material for turbine blades in aircraft engines [1]. Conventional  $\gamma$ -TiAl alloys usually contain the ordered phases  $\gamma$ -TiAl and  $\alpha_2$ -Ti<sub>3</sub>Al at lower temperatures and disordered  $\alpha$ -Ti(Al) phase above 1120°C. Additional alloying elements like Nb, Mo, Ta, Cr or Fe, can stabilize the disordered  $\beta$ -Ti(Al) phase (A2 structure), which could stay at lower temperatures in its ordered  $\beta_0$ -TiAl (B2 structure) state or sometimes transforms to more complex phases [2].

The ductile body centered cubic (bcc)  $\beta$  phase is important for processing because it significantly improves the hot forming behaviour of the material. Otherwise the ordered low temperature  $\beta_0$  phase is said to embrittle the material at service temperature. Unfortunately little is known about the exact order/disorder transformation temperatures of  $\beta/\beta_0$  in ternary alloy systems and the influence of  $\beta$  stabilizing element content is still under research. Additionally, even for the binary TiAl phase diagram the existence of an ordered  $\beta_0$  phase field at high temperatures has yet not been finally proofed or rebutted [3].

In situ HEXRD could be used for determination of the  $\beta_0 \leftrightarrow \beta$  phase transformation, if the superstructure reflex of the  $\beta_0$  phase is not too weak. Neutron diffraction (ND) is best suited to study order/disorder transformations in titanium aluminides [4, 5]. The fundamental reflections in ordered and unordered beta phase are very weak because of the opposite sign of the scattering lengths of Al sites and Ti sites which sum up to the overall signal. Otherwise, the superstructure reflections of the ordered TiAl crystal structures including the ordered  $\beta_0$ -phase become rather large, because they scale with the difference of the scattering lengths of each site. Thus, the application of in situ neutron diffraction in situ is expected to be a powerful tool to determine the temperature of  $\beta_0 \leftrightarrow \beta$  order-disorder transformation. Nevertheless, it is found that a combination of in situ neutron and synchrotron diffraction is even more powerful as also the fundamental peaks can be monitored yielding additional information about the disordered phases. If the order-disorder transformation temperatures in thermodynamic equilibrium are the aim of the investigation it is obligatory to take the heating ramp into account. The heating velocity determines the measured phase transformation temperatures and could yield to different results of neutron and synchrotron measurements. Application of the same heating ramp, when possible, with the same furnace would give the best correspondence between results.

## Materials and Methods

We studied three binary TiAl alloys (Ti-xAl with x = 39, 42 and 45), two semi-binary (with boron addition for finer grain size Ti-xAl-0.2B with x = 39 and 42) and fifteen alloys with additional alloying elements (Ti-42Al- $a_y$  with  $a_y$  = 2; 8.5; 10 at.% of Nb, 2; 4; 6 at.% of Mo, 2; 8.5; 10 at.% of Ta, 2; 3; 4 at.% of Cr and 1; 2; 3 at.% of Fe). All alloys except Ti-42Al-8.5Nb were produced by arc melting under Ar atmosphere. The melt buttons were remelted 5 times to ensure chemical homogeneity and subsequently heat-treated at 1100°C for 5 and 7 days in order to homogenize the microstructure. Samples have fine and medium grain size up to 200  $\mu$ m with duplex microstructure. The sample of Ti-42Al-8.5Nb has been produced by Markus Rackel and production conditions are described in [6].

In situ ND measurements were performed in the materials science diffractometer STRESS-SPEC (FRM II in Garching near Munich, Germany) twice. Two different furnaces have been applied for heating. A standard high temperature furnace (HTF) and a dilatometer, which differ from each other by heating method and applied sample size. The gauge volume for the first experiment was 5 mm $\times$ 5 mm $\times$ 20 mm=500 mm<sup>3</sup> and for the second 5 mm $\times$ 5 mm $\times$ 10 mm=250 mm<sup>3</sup>. We used a wavelength of 2.1 Å. By a 3He-PSD, 25 x 25 cm<sup>2</sup> neutron detector an angular range of 15° has been covered. The detector covered the q-range of 1.7-2.3 2 $\pi$ Å<sup>-1</sup> which enables to monitor superstructure reflections of all three ordered phases simultaneously, namely  $\alpha_2$  101,  $\beta_0$  100 and  $\gamma$  110. Because of longer exposure time needed for better statistics the samples were stepwise heated between 1100°C and 1440°C with a minimum step of 10°C in ranges of special interest. The exposure time was varied from 20 minutes to 1 hour in order to have a better peak to background ratio. Some problems with the dilatometer cooling system appeared during the second neutron experiment. Therefore measurements have been performed between 1100 and 1250°C. Neutron measurements with HTF have been performed up

to 1450°C.

Complementary in situ synchrotron XRD measurements were performed in the High Energy Material Science (HEMS) beamline at DESY in Hamburg, Germany. Because of very good detector time resolution, one picture has been recorded every 0.5 sec and continuous heating was applied. High-energy X-rays with a photon energy of 100 and 87.1 keV, corresponding to a wave length of 0.124 Å and 0.14235 Å were used. The gauge volume was 1 mm×1 mm×5 mm=5 mm<sup>3</sup>. The heat treatments were performed in a DIL805A/D dilatometer with a heating rate of 5 K/min from 1000-1250 °C and 20 K/min from 1250-1450 °C. The high intensity of the synchrotron beam and corresponding short measurement times allowed to cover also the RT –1000°C temperature region. Complete Debye-Scherrer diffraction rings up to a q-value of 5.5 2πÅ<sup>-1</sup> were continuously recorded on a PerkinElmer XRD 1621 flat panel detector with a frame rate of 0.15 Hz and an exposure time of 3 s.

### Results and Discussion

Data analysis showed some differences between neutron and synchrotron results. When measuring samples of identical composition different temperatures of the β<sub>0</sub>↔β phase transformation were detected. Such differences could be explained by the application of different heating ramps. Based on our literature knowleges and experiment results during the in situ ND measurements the superstructure reflection β<sub>0</sub>-100 was never observed in the binary TiAl alloys [7]. However the in situ high-energy XRD experiments clearly show the formation of disordered β phase at about 1360 °C and 1400 °C for Ti-42Al and Ti-45Al respectively. These results proof the direct transformation of disordered α to disordered β in the binary Ti-Al system without the formation of a high temperature ordered β<sub>0</sub>-TiAl phase. The grain size of Ti-39Al is too big and therefore due to bad grain statistic no reliable measurements could be made.

In ternary alloys namely with 1; 2; 3 at. % of Fe; 2; 6 at. % of Mo; 2; 4 at. % of Cr; 8.5; 10 at. % of Nb; 8,5 at. % of Ta the superstructure β<sub>0</sub> reflection was observed by ND]. The synchrotron experiments show that after the β<sub>0</sub> 100 disappeared β is stable up to the highest measured temperatures. The ternary alloys with 2 at.% of Nb and Ta show no ordered β<sub>0</sub> phase. No superstructure β<sub>0</sub> reflection was observed by ND. Samples with 3 at. % of Cr; 10 at. % of Ta, as well as the semi-binary Ti-39/42Al-0.2B alloys were not measured by ND, but this is planned if the project will be continued.

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