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Ordering and disordering of β/β$_o$-phase in γ-TiAl based alloys investigated by neutron diffraction

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ABSTRACT

TiAl based alloys are desirable materials for turbine blades in aircraft engines. Depending on alloy composition and temperature additionally disordered β-Ti(Al) (A2 structure) or ordered β$_o$-TiAl (B2 structure) can occur. Unfortunately little is known about the exact order/disorder transformation temperatures of β/β$_o$.

We used the good contrast of neutron diffraction for ordering and disordering of TiAl alloys to determine the order/disorder temperatures, which are not accessible by other methods like DSC measurements. Several binary TiAl alloys as well as alloys with additional alloying elements were used to investigate the influence of different Al concentrations and alloying additions on the occurring ordering/disordering reactions and phase transformations. As a result ordered β$_o$ phase was found only in selected ternary alloys.

INTRODUCTION

High melting point, low density and diffusivity, and good resistance against oxidation and corrosion make TiAl alloys a perfect material to replace Ni-based superalloys in turbine blades for advanced aero engines [1]. Two types of γ-TiAl based alloys are currently already in use in advanced aircraft turbine engines in the last stages of the low pressure turbine (LPT). These are the so called 48-2-2 alloy based on a composition of Ti-48Al-2Nb-2Cr (all compositions are in at.%) and the so called TNM alloy based on a composition of Ti-43.5Al-4Nb-1Mo-0.1B [2].

At room temperature binary γ-TiAl based alloys consist of tetragonal γ-TiAl (P4/mmm, $L1_0$) and hexagonal α$_2$-Ti_3Al ($P6_3/mmc$, $D0_{19}$). At higher temperatures the ordered α$_2$ disorders to hexagonal close packed (hcp) α-Ti(Al) ($P6_3/mmc$, A3). Additional alloying elements like Nb, Mo, Ta, Cr or Fe, can stabilize the disordered β-Ti(Al) (Im-3m, A2). At lower temperatures β can transform to ordered β$_o$-TiAl (Pm-3m, B2) or even to more complex phases.

The ductile body centered cubic (bcc) β phase is of great technological interest because it significantly improves the hot forming behavior of the material. Otherwise its ordered low temperature counterparts like β$_o$ phase are said to embrittle the material at service temperature. Little is known about the exact order/disorder transformation temperatures of β/β$_o$ in several ternary alloy systems. And the influence of β stabilizing elements like Mo, Nb or Ta on the phase diagrams is still under research [3-6]. Additional, even for the binary TiAl phase diagram the

existence of an ordered β_o phase field at high temperatures has yet not been finally proofed or rebutted [7].
Conventional in situ investigation methods like differential scanning calometrie (DSC) reach their limits with the $\beta \leftrightarrow \beta_o$ transformation and ternary TiAl alloys because an unambiguous assignment of a certain peak to the $\beta \leftrightarrow \beta_o$ transformation is not possible. Also in situ X-ray diffraction measurements are not suitable because the superstructure reflections are very weak due to the small electron density differences of the different atom sites in the ordered crystal structures. In situ neutron diffraction (ND) measurements are best suited to study order/disorder transformations in titanium aluminides [8,9]. The neutron scattering lengths of Ti and Al are almost equal in magnitude but of opposite sign. Thus disordered phases, with a Ti:Al ratio close to one, yield only very weak diffraction peaks, because the average scattering length is almost zero. The fundamental reflections in ordered TiAl crystal structures are also very weak because the scattering lengths of Al sites and Ti sites with their opposite sign add up. However the superstructure reflections of these ordered TiAl crystal structures become rather large, because they contain the difference of the scattering lengths of each site.

Therefore we used ND for investigation of the $\beta \leftrightarrow \beta_o$ ordering/disordering transformation. In addition scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) were performed for characterization of microstructure and real chemical composition of the samples under investigation. In order to evaluate the degree of ordering of β_o as well as α_2 phase the fundamental reflections are needed. They were recently measured during Synchrotron experiments at DESY. The results of the synchrotron measurements with a calculation of the degree of ordering will be shown in a forthcoming article.

EXPERIMENT

Three binary TiAl alloys (Ti-xAl with x = 39, 42 and 45) and five alloys with additional alloying elements (Ti-42Al-2y with y = Nb, Mo, Ta, Cr and Fe) were produced by arc melting in a water cooled copper crucible. The melting was done under Ar atmosphere to prevent oxidation and a pure titanium button was used to getter the residual oxygen in the chamber. Pure elements were used and the buttons were remelted 5 times to ensure chemical homogeneity. The buttons have a weight of 30g. In order to homogenize the microstructure after melting, the buttons were heat-treated at 1100°C for 5 days.

The microstructure was analyzed by SEM with a LEO GEMINI 1530 microscope in back scattered electron (BSE) mode.

The in situ ND measurements were performed in the materials science diffractometer STRESS-SPEC at FRM II in Garching near Munich (Germany). The experimental setup is shown in figure 1. Cylindrical samples with 5 mm diameter and 20 mm length were spark eroded from the center of heat treated buttons. A gauge volume of 5x5x20 mm^3 was used. ND images were recorded by a MWPC (multi wire proportional chamber) plate-detector, with an active area of 250x250 mm^2, and readout channels of 256x256 pixel. Nominal position resolution is 1.3 mm in x and y directions. A Ge (311)-monochromator gave a wavelength of 2.1 Å was selected. The sample detector distance and the beam center were estimated using a standardized Si powder sample. The detector allows to record a 2θ-range of 15°. In order to see superstructure reflections of all three phases γ, β_o and α_2 a 2θ range from 32.5° - 45.5° was chosen. The diffractograms were calculated with the program SteCa [10] and they were analyzed with the Rietveld program MAUD [11].

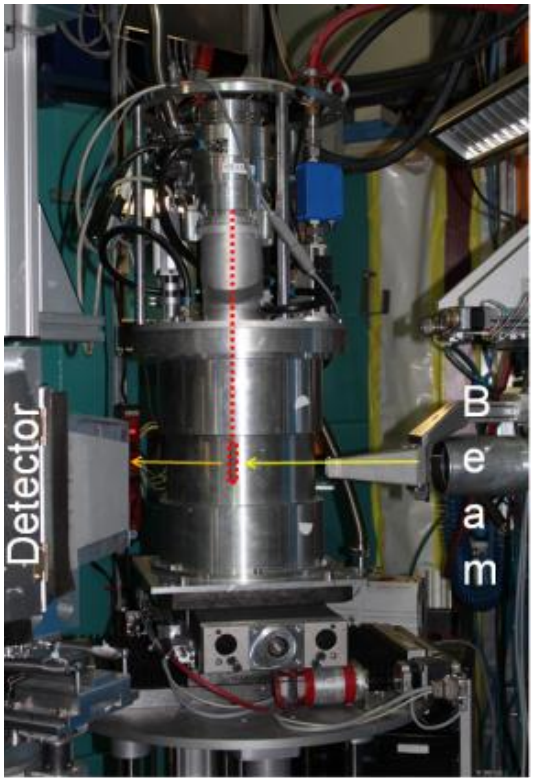

Figure 1. Experimental setup. The vacuum high temperature furnace in the neutron beamline STRESS-SPEC.

For the heat treatments a vacuum high temperature furnace was used. The samples were put into Vanadium containers and were stepwise heated in a temperature range from 1100°C up to 1440°C. Temperature steps were normally 50 °C and changed to 10 °C in temperature ranges of special interest. Depending on the chemical composition of the samples and thus from the amount of the phases present, the exposure time was varied from 10 minutes to 1 hour in order to have a good peak to background ratio. A heating ramp of 20 °C/min was used between the steps. The temperature was kept constant at every step.

RESULTS AND DISCUSSION

Microstructure

Due to the fast cooling after arc-melting the microstructures mainly consisted of large lamellar ($\alpha_2+\gamma$) colonies. Thus the alloys were subsequently heat-treated in order to refine and homogenize the microstructure. Figures 2 a-c show typical microstructures of the heat-treated alloys. Due to their different brightness in the BSE mode, different phases can be distinguished in the micrographs. The γ phase appears dark grey whereas the α_2 phase appears light grey and the β_o phase is almost white.

The alloy Ti-42Al in figure 2a shows the typical microstructure of a binary alloy, consisting of lamellar ($\alpha_2+\gamma$) colonies. During the heat treatment the lamellae were coarsened and some

colonies started to spheroidize at the colony boundaries. In the ternary alloy Ti-42Al-2Fe the lamellar colonies are almost completely vanished (fig. 2b). The microstructure consists of globular γ and α_2 grains and platelets with grain sizes up to 40 µm. Between these grains, smaller drawn-out β_o grains are visible. All other alloys show mixed microstructures, like Ti-42Al-2Cr in figure 2c, with some remaining lamellar colonies surrounded by more globular parts. Three ternary alloys, namely the alloys with Fe, Cr and Mo, show additional β_o phase in the SEM images whereas all other alloys consist only of γ and α_2 phase. Using a program for image analysis the amount of β_o phase was estimated to about 10, 5 and 10 wt.% correspondingly.

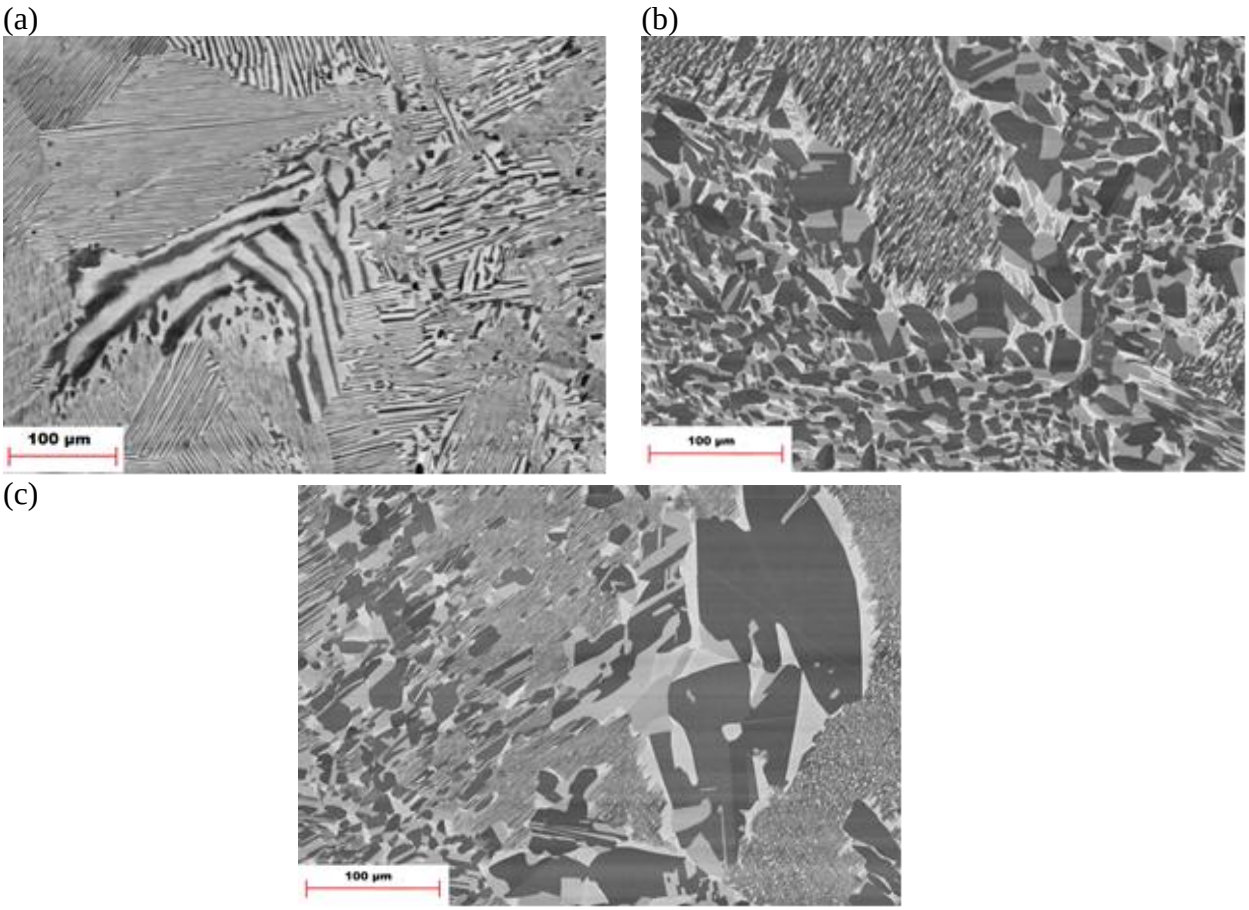

Figure 2. SEM images in BSE mode of (a) Ti-42Al, (b) Ti-42Al-2Fe and (c) Ti-42Al-2Cr. γ phase appears dark grey, α_2 phase light grey and β_o phase is almost white.

In situ heating neutron diffraction experiments

Figure 3 shows two typical neutron diffraction patterns taken at 1100 °C. A 2θ range was chosen in which superlattice peaks of β_o and α_2 and additionally one γ peak were visible, namely γ-110, β_o-100, α_2-101 and α_2-110. It should be noted that one neutron diffraction pattern of Ti-42Al-2Cr needed 1 hour recording time due to small content of β_o phase. The phase fractions of all alloys at 1100°C estimated by Rietveld refinement are given in table 1. Three alloys with β-stabilizing elements, namely with Fe, Mo and Cr, exhibit β_o reflections.

During heating the superlattice peaks decrease and finally vanish indicating the order/disorder transformation temperatures for $\alpha_2 \leftrightarrow \alpha$ and $\beta_o \leftrightarrow \beta$ and also the γ solvus

temperature. No evidence for the presence of ordered β_o was found in the binary Ti-Al alloys at all temperatures investigated. Figure 4 shows for example the evolution of the ND pattern of Ti-42Al-2Fe during step-wise heating from 1100°C to 1250°C. The β_o-100 disappears between 1160°C and 1170°C whereas weak α_2 reflections are still visible up to 1250°C. The transformation temperatures estimated from the in situ heating experiments for all alloys are given in table 1. The addition of Mo shifts the $\beta_o \leftrightarrow \beta$ transformation temperature to higher values compared to Ti-42Al-2Cr and -2Fe. The $\alpha_2 \leftrightarrow \alpha$ transformation temperatures and the γ solvus temperatures for the binary alloys are about 30-40°C higher than values published [1,7]. This might be due to the position of the thermocouple in the furnace and might consecutively shift all other transformation temperatures measured. The values will be checked by in situ heating experiments using synchrotron high-energy X-ray diffraction which are planned to determine the transformation temperatures of the disordered phases and will be published soon.

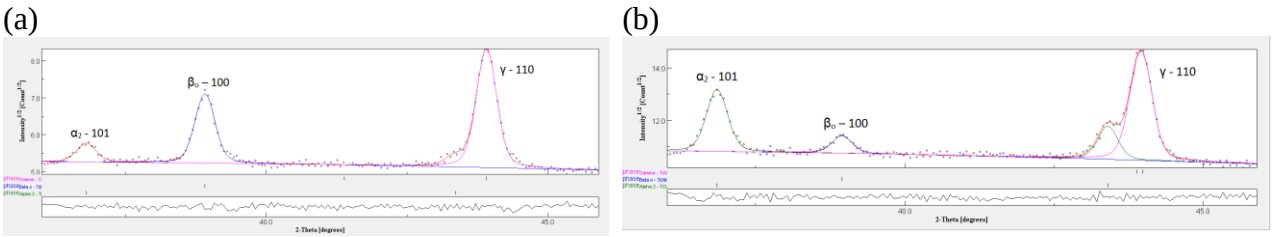

Figure 3. ND spectrum of (a) Ti-42Al-2Mo and (b) Ti-42Al-2Cr at 1100°C. Dots: ND measurement; continous lines: Rietveld calculation with MAUD.

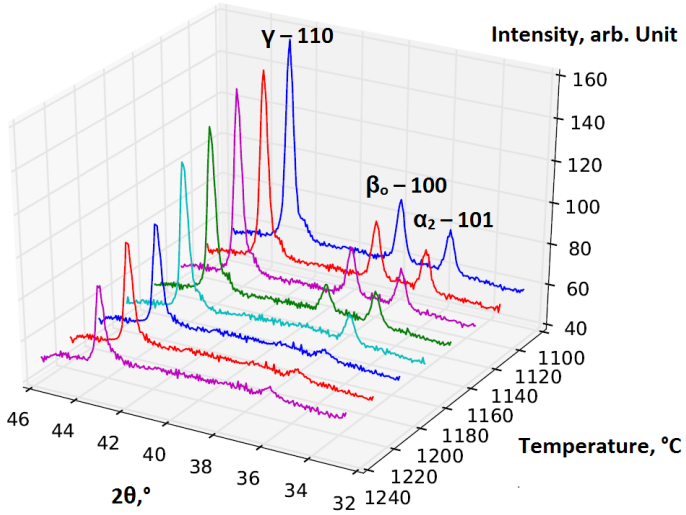

Figure 4. Evolution of the ND pattern of Ti-42Al-2Fe during step-wise heating

Table 1. Phase fractions at 1100°C calculated from the ND spectrums in mass % and transformation temperatures estimated from the in situ heating experiments in °C.
* No exact determination was possible.

Alloy composition	α_2	β_o	γ	$\alpha_2 \leftrightarrow \alpha$	$\beta_o \leftrightarrow \beta$	γ solvus
Ti-42Al-2Mo	12	18	70	1180-1190	1280-1290	1260-1270

Ti-42Al-2Fe	22	12	66	>1250	1160-1170	>1250
Ti-42Al	44.5		55.5	1150-1160		1240-1250
Ti-42Al-2Cr	58.5	5.5	40	1150-1160	1165-1180	1190-1200
Ti-39Al	94		6	1150-1160		*
Ti-45Al	40		60	1160-1170		1325-1350
Ti-42Al-2Ta	38		62	1160-1170		1240-1250
Ti-42Al-2Nb	42		58	1160-1170		1240-1250

CONCLUSIONS

Neutrons are an ideal tool to study the ordering/disordering phase transformations in γ-TiAl based alloys. This transformation temperature of β/β_o of 3 binary and 5 ternary TiAl alloys has been successfully obtained using in situ ND in current study.

Three samples, with 2 at.% of Fe, Mo and Cr, show a superlattice β_o-100 peak in the three phase field $\beta/\beta_o+\alpha/\alpha_2+\gamma$. Five samples including all binary Ti-Al based compositions show no β_o phase at all. At temperatures above 1300 °C no ordered β_o phase appeared even in the high temperature two phase region $\beta+\alpha$. Mo additions shift the $\beta_o \leftrightarrow \beta$ transformation to higher temperatures.

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