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**In and ex situ investigations of the Beta-phase in a Nb and Mo  
containing Gamma-TiAl based alloy**

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4 **In- and ex-situ investigation of the  $\beta$ -phase in a Nb and Mo containing  $\gamma$ -TiAl based alloy**  
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47 *Dedicated to Dr. Arno Bartels on the occasion of his 65th birthday*  
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52 **Abstract**  
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56 In a  $\beta$ -stabilised Ti-43Al-4Nb-1Mo-0.1B alloy (composition in atomic percent) the correlation  
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58 between the occurrence of  $\beta$ -phase and temperature was analyzed experimentally and  
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60 compared to thermodynamic calculations. Results from in-situ high-energy X-ray diffraction,  
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4 texture measurements, heat-treatments, scanning electron microscopy, and temperature-  
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6 dependent flow stress measurements were used to study the evolution of the  $\beta$ -phase with  
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8 temperature. Thermodynamic calculations based on the CALPHAD method were applied to  
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10 correlate the phases developed in the  $\beta$ -solidifying TiAl based alloy under investigation. This  
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12 alloy is characterized by an adjustable  $\beta$ -phase volume fraction at temperatures where hot-  
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14 work processes such as forging and rolling are conducted. Due to a high volume fraction of  $\beta$ -  
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16 phase at elevated temperatures the hot-extruded alloy can be forged under near conventional  
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18 conditions.  
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25 *Key words:* A. titanium aluminides, based on TiAl; B. alloy development; C. phase  
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27 transformation, phase prediction; D. deformation behaviour, conventional forging.  
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## 34 **1. Introduction**

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38 The continuous demand for weight reduction and higher engine efficiencies in automotive,  
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40 aerospace and energy industries pushes the materials applied today towards their limits.  
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42 Therefore, these industries have a strong need for developing novel light-weight materials  
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44 which can withstand temperatures up to 800°C, while maintaining acceptable mechanical  
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46 properties. Intermetallic  $\gamma$ -TiAl based alloys are certainly among the most promising  
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48 candidates to fulfill the required thermal and mechanical specifications [1-3]. Especially, TiAl  
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50 alloys with high Nb-contents, showing a baseline composition of Ti-(42-45)Al-(5-10)Nb  
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52 (at.%), have attracted much attention because of their high creep strength, good ductility at  
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54 room temperature and excellent oxidation resistance [1-6]. Nb reduces the stacking fault  
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56 energy in  $\gamma$ -TiAl, retards diffusion processes and modifies the structure of the oxidation layer  
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4 [2,4,6]. Cast alloys based on Ti-45Al, which solidify via the  $\beta$ -phase, exhibit an isotropic,  
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6 equiaxed and texture-free microstructure with modest micro-segregation, whereas peritectic  
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8 alloys (solidification via the  $\alpha$ -phase) show anisotropic microstructures as well as significant  
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10 texture and segregation [7,8]. Alloy design concepts for  $\gamma$ -TiAl based alloys showing refined  
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12 cast microstructures were recently reported by Imayev et al. [9]. In order to increase the  
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14 economic feasibility of wrought processing for the manufacture of  $\gamma$ -TiAl components, alloys  
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16 are needed which can be processed “near conventionally”, e.g. a conventional forging  
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18 equipment with minor and inexpensive modifications can be used. Thus, a fine-grained  
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20 casting microstructure is favourable to both, ingot breakdown and secondary forming  
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22 operations [2,3]. The alloys should be designed to allow for robust industrial heat-treatments,  
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24 i.e. the alloy must tolerate a specified (and realistic) variation of the constituting elements,  
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26 without pronounced changes of phase transition temperatures and phase volume fractions and  
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28 related variations in mechanical properties. An alloy design strategy to improve the hot-  
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30 workability of TiAl alloys is to exploit a combination of thermo-mechanical processing and  
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32 additional alloying elements to induce the disordered  $\beta$ -phase at elevated temperatures as  
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34 ductile phase [10-13]. The disordered  $\beta$ -phase with bcc lattice provides a sufficient number of  
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36 independent slip systems. Thus, it may improve the deformability at elevated temperature,  
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38 where, for example, processes such as rolling and forging are performed. Several authors  
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40 [10,13-19] have demonstrated that, by stabilizing the  $\beta$ -phase through alloying with Nb, Ta,  
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42 Mo or other elements, an improvement in hot-workability can be achieved and novel types of  
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44 microstructures can be adjusted by exploiting a multitude of solid-state transformations. From  
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46 a processing related point of view the alloy should fulfil the following demands: (i) after  
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48 casting and solidification, the alloy should possess a refined equiaxed microstructure with no  
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50 significant casting texture. (ii) The composition of the alloy must be defined to ensure a  
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52 solidification path according to  $L \rightarrow L + \beta \rightarrow \beta \rightarrow \dots$ , instead of a peritectic solidification  
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54 pathway,  $L \rightarrow L + \beta \rightarrow \alpha \rightarrow \dots$ , which is prone to segregation [7,9]. (iii) During ingot  
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4 breakdown as well as secondary hot-forming operations a significant volume fraction of  
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6 disordered  $\beta$ -phase should be present, which improves the deformability at elevated  
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8 temperature and suppresses grain growth. At service temperature, however, the volume  
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10 fraction of the  $\beta$ -phase, which then shows an ordered B2 structure, should be insignificant in  
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12 order not to deteriorate creep properties [20]. (Note: in the following B2 is referred to as  $\beta$ /B2  
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14 unless stated otherwise). (iv) In order to avoid uncontrollable grain coarsening effects during  
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16 hot-processing as well as during adjustment of the microstructure through subsequent heat-  
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18 treatments the existence of a single phase region at elevated temperatures should be avoided,  
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20 i.e. transitions such as  $\alpha + \gamma \rightarrow \alpha$  must be suppressed. Another possibility is to keep the  $\alpha$ -  
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22 phase region very small. In this case the single  $\alpha$ -field can be passed without significant grain  
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24 coarsening as long as the dissolution kinetics of the  $\beta$ /B2-phase is decelerated by the presence  
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26 of alloying elements with a low diffusibility, e.g. Nb and Mo.  
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## 34 **2. Alloy selection and experimental**

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39 In order to select an alloy which fulfills the demands as defined in the previous section,  
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41 thermodynamic calculations based on the CALPHAD method were conducted for the  
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43 prediction of the constituent phases and the related transition temperatures. Two different  
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45 software packages - ThermoCalc® and MatCalc - were applied using the same commercial  
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47 TiAl database [21]. In recent publications, however, the thermodynamic database used was  
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49 found to poorly describe the transition temperatures and phase proportions in high Nb bearing  
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51  $\gamma$ -TiAl based alloys as reported in [15,22]. Therefore, it should be pointed out that the  
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53 following calculation was conducted to study alloying trends rather than to give absolute  
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55 values on phase fractions and transition temperatures. Figure 1 shows the calculated phase  
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57 fractions as a function of temperature for the investigated Ti-43Al-4Nb-1Mo-0.1B alloy. Nb  
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4 decreases the stacking fault energy in  $\gamma$ -TiAl, slows down diffusion processes in both,  $\gamma$ -TiAl  
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6 and  $\alpha_2$ -Ti<sub>3</sub>Al and improves the oxidation behaviour [2,4,6]. Like Nb, Mo raises the activation  
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8 energy of diffusion in  $\gamma$  and  $\alpha_2$ , but exhibits a much higher partition coefficient  $k_{\beta\alpha}$  than Nb  
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10 [23]. It must be taken into account that phases, when stabilized by such slow-diffusing  
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12 elements, are expected to exhibit a sluggish dissolution behaviour. A boron content of 0.1 at%  
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14 was selected to ensure a grain refining effect during solidification [24,25]. Boron, which tends  
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16 to form very stable borides is also beneficial in case of heat-treatments conducted at high  
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18 temperatures. Here, the borides retard grain coarsening by pinning of the grain boundaries  
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20 [3,25]. In addition, the borides favour the formation of the lamellar microstructure ( $\alpha \rightarrow \alpha + \gamma$ )  
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22 over the massive transformation ( $\alpha \rightarrow \gamma_M$ ) by heterogeneous nucleation of  $\gamma$ -lamellae [24,25].  
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24 From Figure 1 it is evident that Ti-43Al-4Nb-1Mo-0.1B solidifies entirely via the  $\beta$ -phase.  
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26 Furthermore, the mole fraction of the  $\beta$ -phase shows a minimum around 1250°C. It should be  
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28 noted that below the temperature of the minimum the mole fraction of the  $\beta$ -phase slightly  
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30 increases or shows an approximately unchanging value. Below the eutectoid temperature (~  
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32 1115°C) the  $\beta$ -phase fraction decreases with decreasing temperature and seems to vanish at  
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34 about 600°C. From the phase predictions shown in Figure 1 it is obvious that the chosen alloy  
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36 exhibits no single phase region at temperatures below 1400°C. However, it must be kept in  
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38 mind that Figure 1 predicts phase conditions present under thermodynamic equilibrium and  
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40 that the used database has already shown an inaccuracy in the prediction of phase proportions  
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42 in  $\gamma$ -TiAl based alloys possessing high Nb concentrations [15,22]. However,  $\gamma$ -TiAl based  
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44 alloys, when processed under technical relevant conditions, always show a more or less  
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46 pronounced deviation from phase equilibrium which makes any comparison difficult.  
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49 Ti-43Al-4Nb-1Mo-0.1B ingots with 65 mm (experimental ingots) and with 230 mm (large-  
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51 scale ingots) in diameter were prepared by GfE Metalle und Materialien GmbH, Nuremberg,  
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53 Germany, by means of double vacuum arc melting using commercially pure charge materials  
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4 and master alloys. The total amount of interstitial impurities was well below 750 mass-ppm.  
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6 For details concerning ingot processing the reader is referred to reference [26]. The large-  
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8 scale ingot was protected with a diffusion barrier, canned in steel and hot-extruded below  $T_{\alpha}$   
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10 to a diameter of about 50 mm of TiAl core material. After extrusion a stress-relieve heat-  
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12 treatment was applied (950°C/4hrs/furnace cooling).  
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15 The distribution and constitution of the phases in the as-cast as well as hot-extruded and  
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17 stress-relieved alloy was analyzed using a Zeiss Evo 50 scanning electron microscope (SEM)  
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19 equipped with an Oxford Instruments Inco Crystal 300.  
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22 For texture analysis of the as-cast alloy neutron diffraction was applied which allows  
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24 examination of the whole body of a sample of some cubic centimetres [7]. The measurements  
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26 were carried out at the TEX-2 diffractometer of GKSS Research Centre using cylindrical  
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28 specimens of 10 mm in diameter, which were cut from the centre of an experimental Ti-43Al-  
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30 4Nb-1Mo-0.1B ingot, parallel to its symmetry axis. Quantitative textures were calculated  
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32 using the iterative series expansion method proposed by Dahms and Bunge [27] with a degree  
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34 of series expansion of  $l_{max} = 22$ . The advantage of this method is that fewer pole figures are  
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36 required when compared with standard series expansion methods.  
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40 In-situ high-energy X-ray diffraction (HE-XRD) studies regarding the occurring solid-state  
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42 phase transformations and the temperature dependence of the  $\beta$ -phase were performed at the  
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44 beamline ID15B at the European Synchrotron Radiation Facility (ESRF) in Grenoble, France.  
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46 For the experiment, a custom-made diffraction furnace was used which heated the specimen  
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48 up to 1400°C. Heating was conducted under constant flow of helium to avoid oxidation of the  
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50 sample surface. The furnace had an entrance hole for the primary X-ray beam and an exit  
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52 window for the scattering intensity (Debye-Scherrer rings) with an opening angle of 15°. Both  
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54 X-ray windows were equipped with foils of polyimide (Kapton®, DuPont®, Wilmington, DE,  
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56 USA) in order to hold the controlled atmosphere. The samples were machined to geometries  
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58 of cylinders with a diameter of 4 mm and were mounted on a ceramic sample holder for the  
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4 in-situ diffraction furnace. The specimen were continuously heated to 800°C with a heating  
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6 rate of 5 K min<sup>-1</sup> and further heated to 1400°C with 2 K min<sup>-1</sup>. Monochromatic synchrotron  
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8 radiation with a nominal energy of 89.05 keV and an energy resolution of  $\Delta E/E = 10^{-3}$  was  
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10 used. The cross-section of the primary beam at the sample position was defined as 100 × 100  
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12  $\mu\text{m}^2$ . The intensity of the primary beam was measured by a photo diode. HE-XRD patterns  
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14 were taken continuously during the in-situ heat treatment with a time interval of 2 min  
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16 between two measurements. The specimen was continuously rotated during exposure to avoid  
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18 texture effects and obtain smooth Debye-Scherrer rings. A two-dimensional image plate  
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20 detector (mar345, Marresearch, Norderstedt, Germany) with a resolution of 2300 × 2300  
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22 pixels (pixel size: 150 × 150  $\mu\text{m}^2$ ) was used for detecting the scattered photons. The  
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24 diffraction patterns covered a range of scattering vector of  $2.8 \text{ nm}^{-1} < q < 64 \text{ nm}^{-1}$ , where the  
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26 length of the scattering vector is given by  $|\mathbf{q}| = q = (4\pi / \lambda) \sin \theta$ , with  $2\theta$  being the scattering  
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28 angle and  $\lambda$  the wavelength. An exposure time of 20 s yielded a diffraction pattern with  
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30 excellent counting statistics. The average readout of the detector data took 90 s. The  
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32 diffraction patterns were corrected for background scattering and were normalized to the  
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34 primary intensity of the synchrotron X-ray flux by using the data reduction software program  
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36 FIT2D [28]. The measurements showed isotropic Debye-Scherrer diffraction rings, which  
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38 were azimuthally averaged for equal radial distances from the central X-ray beam.  
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44 In order to study the deformation (plastic flow) behaviour of alloy Ti-43Al-4Nb-1Mo-0.1B,  
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46 cylindrical specimens with dimension Ø 4mm x 10mm were cut from stress-relieved hot-  
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48 extruded material. The experiments were performed in a deformation dilatometer DIL805A/D  
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50 supplied by Bähr-Thermoanalyse GmbH. The samples were heated under helium atmosphere  
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52 up to temperatures in the range of 1240 to 1300°C, held for 10 min and then deformed at rates  
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54 between 0.05 to 0.5 s<sup>-1</sup>.  
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59 Forging tests on an industrial scale were performed on a conventional 10 MN hydraulic press  
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61 without any special isothermal forging equipment. However, due to the high strain rate  
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4 sensitivity of  $\gamma$ -TiAl alloys and thus the low die speed in the forging process, the dies were  
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6 pre-heated prior to forging to avoid excessive cooling during deformation. The die  
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8 temperature during forging was approximately 400 - 800°C below the billet temperature. The  
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10 hydraulic press has been specially automated for low die speeds and to allow an exact control  
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12 of die speed, position and temperature during the whole process. The billets for the forging  
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14 experiments on an industrial scale were produced via the ingot metallurgy route as described  
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16 above. After extrusion the material is cut into cylinders between 40 and 80 mm in length and  
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18 mechanically turned to a diameter of 40 or 50 mm. Prior to forging a heat shielding layer is  
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20 applied to the billet to reduce heat loss during transfer from the furnace to the press as well as  
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22 during the initial forging process. The billet was heated up to a forging temperature above  $T_{\alpha}$   
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24 in an electric furnace under argon atmosphere [18]. After holding on temperature for a defined  
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26 time the billet was manually transferred into the press within 5sec and the forging process is  
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28 started. The total contact time between the billet and the dies can last up to 60 sec depending  
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30 on die speed and total stroke. More information on the industrial forging process is given by  
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32 Kremmer et al. [18].  
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### 41 **3. Results and discussions**

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45 Figure 2a shows the microstructure of alloy Ti-43Al-4Nb-1Mo-0.1B in as-cast condition. The  
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47 microstructure can be explained by a complete solidification via the  $\beta$ -phase [7,9,17] as  
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49 predicted in Figure 1. It consists of equiaxed lamellar ( $\gamma + \alpha_2$ )-colonies with a colony diameter  
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51 of about 100  $\mu\text{m}$ . The  $\beta$ /B2-phase is mainly located along colony boundaries and only a small  
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53 volume fraction is present within the colonies. In addition, the existence of rod- shaped Ti-  
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55 borides, enriched in Nb and Mo, was detected [16,17]. EDX analysis has provided evidence  
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57 that the  $\beta$ -phase is more enriched in Mo than in Nb, which confirms that Mo exhibits a higher  
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4 partition coefficient  $k_{\beta\alpha}$  than Nb [23]. From this observation it is assumed that both  $\beta$ -  
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6 stabilizing elements have segregated to  $\beta/\alpha$ -interface boundaries in the course of the  $\beta \rightarrow \alpha$   
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8 transformation. However, the  $\beta/\text{B2}$ -phase does not form a complete layer around the lamellar  
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10 colonies, but is intersected by small  $\gamma$ -grains. According to Zhang et al. [24] the mixture of  
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12  $\beta/\text{B2}$  and  $\gamma$  grains as shown in the inset of Figure 2a results from the cellular (discontinuous)  
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14 reaction  $\beta \rightarrow \beta + \gamma$ . At room temperature the as-cast material shows a high amount of  $\beta/\text{B2}$ -  
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16 phase which is inconsistent with the thermodynamic calculation shown in Figure 1. This  
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18 behaviour might be attributed to both, the high solidification and cooling rates. This finding  
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20 also implies that a considerable amount of  $\beta/\text{B2}$  must exist in a metastable state. For the sake  
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22 of completeness it should be mentioned that the as-cast microstructure (Figure 2a) can further  
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24 be refined by subsequent heat-treatments. Annealing in the temperature range of 1250 -  
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26 1400°C followed by air cooling leads to colony sizes well below 50  $\mu\text{m}$  as reported by H.  
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28 Chladil et al. [17]. Recently, for a comparable alloy system similar results have been reported  
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30 by Imayev et al. [9].  
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37 Figure 2b shows alloy Ti-43Al-4Nb-1Mo-0.1B after hot-extrusion below the  $\alpha$ -transus  
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39 temperature ( $T_\alpha \sim 1260^\circ\text{C}$ , as determined by DSC measurements [16]). During extrusion a  
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41 refinement of the ingot microstructure took place and the  $\beta/\text{B2}$ -phase has been aligned in  
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43 extrusion direction which also was observed for the borides [16]. After extrusion a stress-  
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45 relieve heat-treatment at 950°C was applied [17].  
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Here, a pronounced preferential orientation of the  $\gamma$ -grains is evident. As the pole figure

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4 shows that a preferential alignment of  $\langle 110 \rangle$  directions perpendicular to the symmetry axis  
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6 occurred. This texture corresponds to that usually observed for cast  $\gamma$ -TiAl alloys [29,30] and  
7  
8 can be explained by a [0001] dendrite orientation of the hexagonal  $\alpha$ -phase with respect to the  
9  
10 heat flow direction. In conclusion, the microstructural observation of equiaxed solidification  
11  
12 of the  $\beta$ -stabilized Ti-43Al-4Nb-1Mo-0.1B alloy agrees with the results of texture analysis.  
13  
14 The absence of a casting texture is beneficial as far as subsequent hot-forming operations are  
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16 concerned.  
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19  
20 Figure 4 shows four azimuthally averaged HE-XRD patterns (Debye-Scherrer rings) of the  
21  
22 alloy Ti-43Al-4Nb-1Mo-0.1B for 21°C (Figure 4a), 1179°C (Figure 4b), 1259°C (Figure 4c),  
23  
24 and 1320°C (Figure 4d). In the initial state (Figure 4a) the diffraction pattern solely contains  
25  
26 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  $\gamma$ -  
27  
28 111 are completely overlapping and appear as one single peak at  $q = 27.20 \text{ nm}^{-1}$ . The  
29  
30 diffraction peaks of  $\gamma$ -002 and  $\gamma$ -200 at  $q = 31.15 \text{ nm}^{-1}$  and  $q = 31.55 \text{ nm}^{-1}$ , respectively, are  
31  
32 clearly separated due to slight differences in the lattice spacing along the  $a$  and  $c$  axis of the  
33  
34 tetragonal  $\gamma$ -TiAl crystal unit cell (L1<sub>0</sub> structure). With increasing temperature, the diffraction  
35  
36 peaks are shifted towards lower  $q$  values because of thermal expansion of the crystal lattice.  
37  
38 The transformation  $\alpha_2 \rightarrow \alpha$  takes place at the eutectoid temperature, which is indicated by the  
39  
40 appearance of the disordered  $\alpha$ -Ti phase. The order-disorder transition is accompanied by a  
41  
42 reduction of the size of crystal unit cell with the reciprocal lattice correlation  $\alpha_2$ -200  $\equiv$   $\alpha$ -100  
43  
44 at  $q \approx 25.2 \text{ nm}^{-1}$  and  $\alpha_2$ -002  $\equiv$   $\alpha$ -002 at  $q \approx 27.0 \text{ nm}^{-1}$ . Therefore, the superstructure diffraction  
45  
46 peak  $\alpha_2$ -101 at  $q = 18.6 \text{ nm}^{-1}$  disappears. The eutectoid temperature was determined to be  
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48 about 1165°C, which matches well with the result obtained from differential scanning  
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50 calorimetry (DSC) measurements [16,17], but is about 50°C above that predicted by the phase  
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52 fraction calculations (Figure 1). Figure 4b illustrates the diffraction pattern in the phase field  $\alpha$   
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54 +  $\beta$ /B2 +  $\gamma$  at 1179°C above the eutectoid temperature.  
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4 The thermodynamic equilibrium calculations shown in Figure 1 indicate a minimum of the  
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6  $\beta$ /B2-phase fraction at about 1260°C, which is confirmed by the diffraction pattern at 1259°C  
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8 (Figure 4c), where the intensities of the B2-100 and B2-110 diffraction peaks at  $q = 19.20$   
9  $\text{nm}^{-1}$  and  $q = 27.35 \text{ nm}^{-1}$ , respectively, show a minimum. As visible in Figure 4d, the  
10  
11 diffraction pattern significantly changes at temperatures above  $\alpha$ -transus, where the phase  $\gamma$ -  
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13 TiAl disappears and the alloy only contains  $\beta$ /B2-phase and  $\alpha$ -phase. Figure 4d shows the  
14  
15 presence of  $\alpha$ ,  $\beta$  and B2 phases above the  $\alpha$ -transus temperature, which indicates that  
16  
17 disordered  $\beta$  and ordered B2 coexist up to high temperatures, presumably forming a domain  
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19 structure. This finding is in agreement with thermodynamic calculations (Figure 1), where the  
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21 presence of B2 is predicted up to about 1410°C. However, the  $\beta$ /B2 coexistence is in partial  
22  
23 contradiction with the demand of a disordered bcc high-temperature phase (see Introduction).  
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25 Nevertheless, one can speculate about the difference in the deformation behaviour of  $\beta$  and  
26  
27 B2 at such high temperatures. This question, however, will be topic of further studies. The  
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29 HE-XRD measurements provide no evidence for passing through a single  $\alpha$  phase field, which  
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31 is in agreement with the thermodynamic equilibrium calculation (Figure 1). However,  
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33 previous studies have shown that the thermodynamic databank used was found to poorly  
34  
35 describe the transition temperatures and phase proportions in high-alloyed  $\gamma$ -TiAl based alloys  
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37 [15,16,22]. In addition, the heating rate of  $2 \text{ K min}^{-1}$  might be too fast for reaching the  
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39 thermodynamic equilibrium if the single  $\alpha$  phase field is rather small and/or the  $\beta$ /B2-phase  
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41 shows a slow dissolution kinetic. In the temperature range above  $\alpha$ -transus, the two-  
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43 dimensional diffraction patterns (area detector images) indicate a evolution from sharp and  
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45 well defined Debye-Scherrer rings to more or less regular diffraction spots relating to local  
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47 reciprocal lattice maps from a few single crystallites (see reference [31]). This effect is due to  
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49 significant grain growth above the  $\alpha$ -transus temperature and the remaining low number of  
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4 illuminated crystallites. Table 1 summarizes the transformation temperatures of the alloy Ti-  
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6 43Al-4Nb-1Mo-0.1B determined by in-situ HE-XRD.

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8 In order to check the appearance of the minimum of the  $\beta$ /B2-phase as predicted by phase  
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10 calculations (Figure 1), an annealing treatment has been conducted. Figure 5 shows the  
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12 microstructure of a sample with an actual Al content of 43.8 at% which was annealed at  
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14 1270°C for 3 hrs followed by air cooling. The microstructure of the starting material is  
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16 depicted in Figure 2b. From Figure 5 it is evident that during annealing the  $\alpha$ -grains have  
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18 grown and the  $\beta$ /B2-phase almost disappeared. However, a small volume fraction of  $\beta$ /B2-  
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20 phase was still present (along with the borides) preventing catastrophic grain growth. Many of  
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22 the  $\beta$ -particles are situated along former  $\alpha$ -grain boundaries and triple points contributing to  
23  
24 the so-called “Zener-drag” mechanism [33]. Again, the question if the predicted minimum of  
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26 the  $\beta$ /B2-phase is real or if a small single  $\alpha$ -phase field still exists can not be answered  
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28 unambiguously. Although a holding time of 3 hrs was used in this experiment the existence of  
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30 a single  $\alpha$ -phase field can be masked if the (metastable)  $\beta$ /B2-phase exhibits a rather slow  
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32 dissolution behaviour. In order to answer this question further experiments are in progress and  
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34 the results will be reported in a forthcoming paper.

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41 Figure 6 depicts schematically the dependence of the flow stress on temperature and strain  
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43 rate. The flow stress shows a distinct strain rate dependency, which is a specific behaviour of  
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45  $\gamma$ -TiAl based alloys [34]. Of particular interest, however, is the pronounced flow stress peak.  
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47 The temperature of the peak maximum corresponds with that temperature where CALPHAD  
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49 predicts the minimum fraction of  $\beta$ /B2-phase (Figure 1). For a deformation temperature below  
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51 the peak maximum the flow stress decreases as it decreases for temperatures above the flow  
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53 stress maximum. The behaviour of the flow stress correlates directly with the volume  
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55 fractions of the  $\beta$ /B2-phase at the respective temperatures. Therefore, it is tempting to  
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57 speculate that the  $\beta$ /B2-phase with bcc lattice provides a sufficient number of independent slip  
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4 systems. Thus, it improves the deformability at elevated temperature, where, for example,  
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6 processes such as rolling and forging are carried out.

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8 The hot-workability of alloy Ti-43Al-4Nb-1Mo-0.1B was evaluated by performing various  
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10 forging experiments using a parameter matrix of different billet dimensions, billet  
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12 temperatures, die temperatures, and die speeds. In Figure 7a a representative result of a  
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14 forging experiment is shown. The pancake was forged to an overall strain of 1.3. The  
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16 microstructure after forging is displayed in Figure 7b. At room temperature the microstructure  
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18 consists of lamellar ( $\alpha_2 + \gamma$ )-colonies with  $\beta$ -phase situated on colony boundaries and triple  
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20 points. Analysing all forging experiments, it was found that the forging window of alloy Ti-  
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22 43Al-4Nb-1Mo-0.1B is significantly extended when compared to other high Nb bearing  $\gamma$ -  
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24 TiAl based alloys [18]. The observed widening can mainly be attributed to the presence of the  
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26 bcc  $\beta$ -phase at forging temperature and its influence on the deformation behaviour (Figure 6).  
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28 Finally, Figure 7c shows a blade pre-form made of alloy Ti-43Al-4Nb-1Mo-0.1B. More  
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30 information on the blade forging process is reported in refs. [18].  
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#### 38 **4. Summary**

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42 Thermodynamic calculations based on the CALPHAD method were used to design a  $\beta$ -  
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44 solidifying TiAl based alloy which exhibits an adjustable volume fraction of  $\beta$ -phase. In the  
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46 as-cast state the selected alloy Ti-43Al-4Nb-1Mo-0.1B shows a microstructure consisting of  
47  
48 equiaxed lamellar ( $\gamma + \alpha_2$ )-colonies with a colony diameter of about 100  $\mu\text{m}$ . The  $\beta/\text{B2}$ -phase  
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50 is mainly located along colony boundaries. Texture analysis using neutron diffraction has  
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52 shown no occurrence of a casting texture. A subsequent hot extrusion process leads to a  
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54 pronounced refinement of the microstructure. At temperatures where hot-deformation  
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56 processes, such as forging and rolling are conducted, the amount of  $\beta/\text{B2}$ -phase is  
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4 considerably high in order to facilitate plastic deformation. However, the fraction of the  $\beta$ /B2-  
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6 phase exhibits a minimum around the  $\alpha$ -transus temperature which is reflected in a maximum  
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8 of the flow stress. At service temperature ( $T < 800^\circ\text{C}$ ), however, the volume fraction of the  
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10  $\beta$ /B2-phase is designed to be small in order not to deteriorate mechanical properties, e.g.  
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12 creep strength. The evolution of the  $\beta$ /B2-phase with temperature was investigated in-situ by  
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14 means of high-energy XRD as well as conventional ex-situ characterization methods. In  
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16 principle, the results obtained experimentally confirm the temperature dependence of the  
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18  $\beta$ /B2-phase as predicted by thermodynamic calculations, although the question concerning the  
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20 existence of a single  $\alpha$  phase field could not be answered. Due to a high volume fraction of  
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22  $\beta$ /B2-phase at elevated temperatures the alloy can be forged under near conventional  
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24 conditions. Alloy Ti-43Al-4Nb-1Mo-0.1B shows a large forging window with regard to billet  
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26 temperature, die temperature, and die speed, providing the basis for a robust industrial forging  
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28 process.  
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## Figure Captions

**Figure 1:** Calculated phase fractions as a function of temperature for the investigated alloy Ti-43Al-4Nb-1Mo-0.1B including 450 mass-ppm oxygen. Note the temperature dependence of the  $\beta$ -phase. The phase fraction of  $\beta$ -phase decreases from the eutectoid temperature (here at  $\sim 1115^\circ\text{C}$ ) towards the  $\alpha$ -transus temperature ( $T_\alpha \sim 1265^\circ\text{C}$ ), exhibits a minimum at  $T_\alpha$  and increases again for temperatures above  $T_\alpha$ .

**Figure 2:** (a) Microstructure of alloy Ti-43Al-4Nb-1Mo-0.1B in as-cast condition. Inset: detail in larger magnification. (b) Microstructure of the same alloy after hot-extrusion below the  $\alpha$ -transus temperature. The extrusion direction is vertical; extrusion ratio:  $\sim 7:1$ . Arrows: borides aligned in extrusion direction. SEM images taken in back-scattered electron (BSE) mode, i.e.  $\gamma$ -TiAl appears grey to dark,  $\alpha_2$ -Ti<sub>3</sub>Al light grey and  $\beta$  exhibits the brightest contrast.

**Figure 3:** Recalculated 110 - pole figure of the  $\gamma$ -TiAl phase in alloy Ti-43Al-4Nb-1Mo-0.1B after casting and solidification. A maximum pole density of 1.8 multiple random distribution (MRD) suggests no statistically significant texture. Inset: 110 - pole figure of an arc-melted Ti-48Al button (2.64 MRD). A well developed casting texture is present (see text and ref. [7]).

**Figure 4:** A series of HE-XRD patterns (azimuthally averaged scattering intensity versus |scattering vector|  $q$ ) during continuous heating of the alloy Ti-43Al-4Nb-1Mo-0.1B at (a)

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4 21°C, (b) 1179°C, (c) 1259°C and (d) 1320°C. The diffraction peaks are indexed according to  
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6 the present phases: (a)  $\gamma$ ,  $\alpha_2$  and B2, (b)  $\gamma$ ,  $\alpha$  and B2, (c)  $\gamma$ ,  $\alpha$  and B2, (d)  $\alpha$  and  $\beta$ /B2.  
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10 **Figure 5:** Microstructure of a Ti-43Al-4Nb-1Mo-0.1B sample after annealing within the  
11 single  $\alpha$ -phase region at 1270°C for 3 hrs. The starting microstructure is shown in Figure 2b.  
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13 During annealing the  $\alpha$ -grains have grown to an average diameter of about 200  $\mu\text{m}$ . The  
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15 remaining  $\beta$ -phase (bright contrast) is partly situated along former  $\alpha$ -grain boundaries and  
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17 triple points. During subsequent air cooling the  $\alpha$ -grains transformed to lamellar ( $\alpha_2 + \gamma$ )-  
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19 colonies. SEM image taken in BSE mode.  
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30 **Figure 6:** Dependence of the flow stress of hot-extruded alloy Ti-43.8Al-4Nb-1Mo-0.1B on  
31 temperature and strain rate (here 0.05 and 0.5  $\text{s}^{-1}$ ). Because of confidentiality reasons the  
32 numerical values of the flow stresses have been omitted. The temperature ( $\sim T_\alpha$ ) where the  
33 maximum of the flow stress occurs corresponds with that temperature where CHAPHAD  
34 predicts the minimum fraction of  $\beta$ -phase (Figure 1).  
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46 **Figure 7:** (a) Pancake of Ti-43Al-4Nb-1Mo-0.1B. The billet was forged to an overall strain  
47 of 1.3. (b) Microstructure of the forged pancake. The microstructure consists of lamellar ( $\alpha_2$   
48 +  $\gamma$ )-colonies with  $\beta$ -phase (bright contrast) situated on colony boundaries and triple points.  
49 SEM image taken in BSE mode. (c) Ground blade pre-form manufactured by upsetting both  
50 sides of a cylindrical bar and subsequent side pressing to the final shape.  
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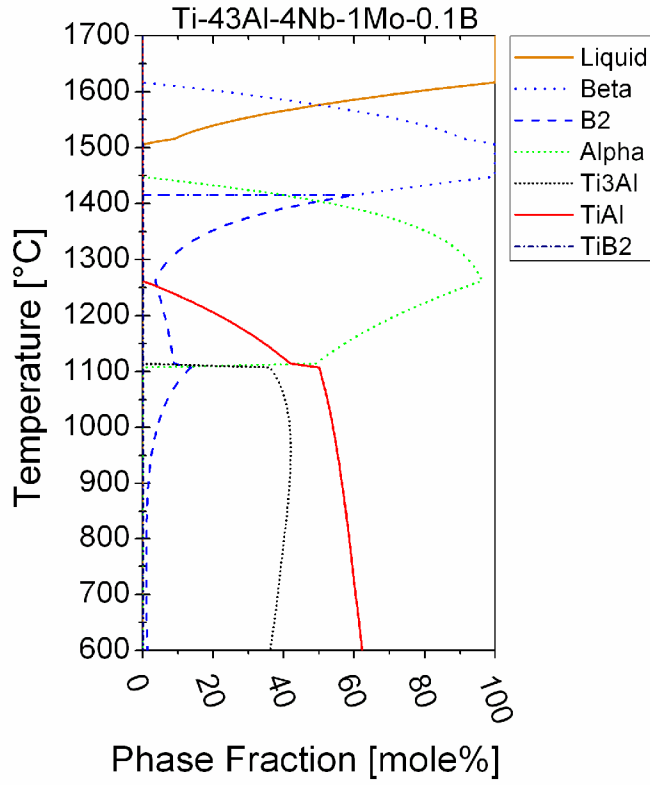


Figure 1

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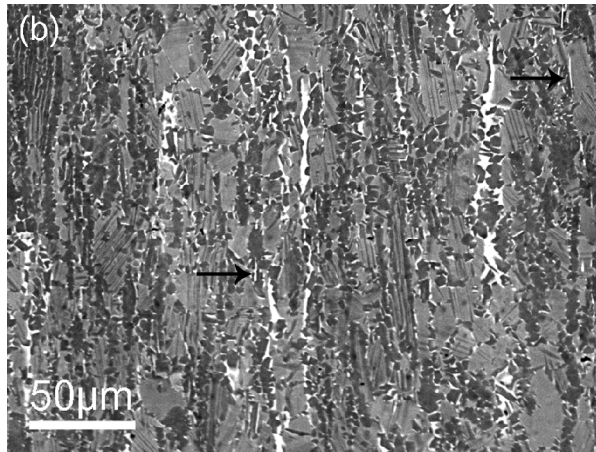
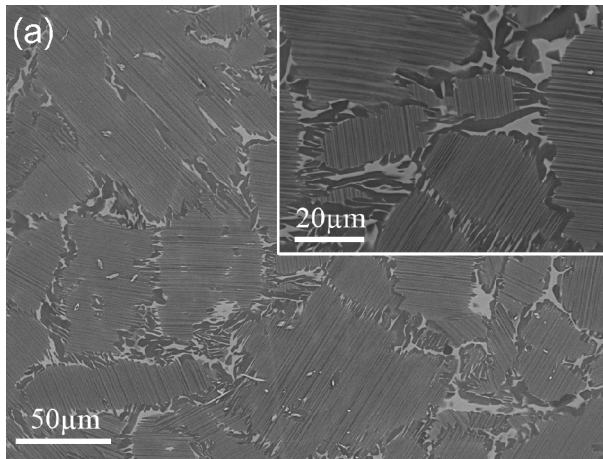


Figure 2

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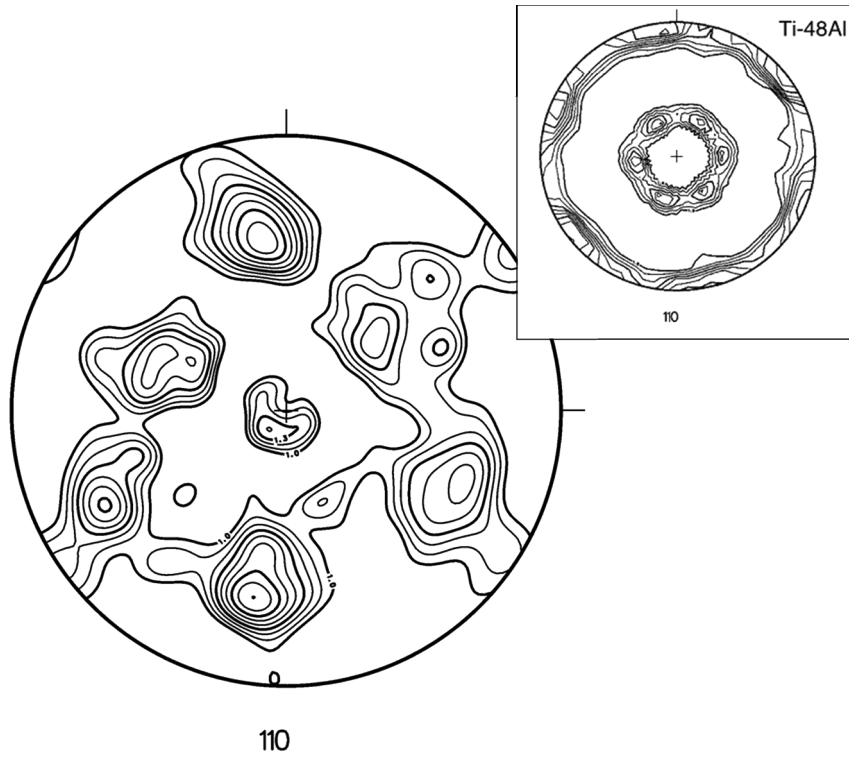


Figure 3

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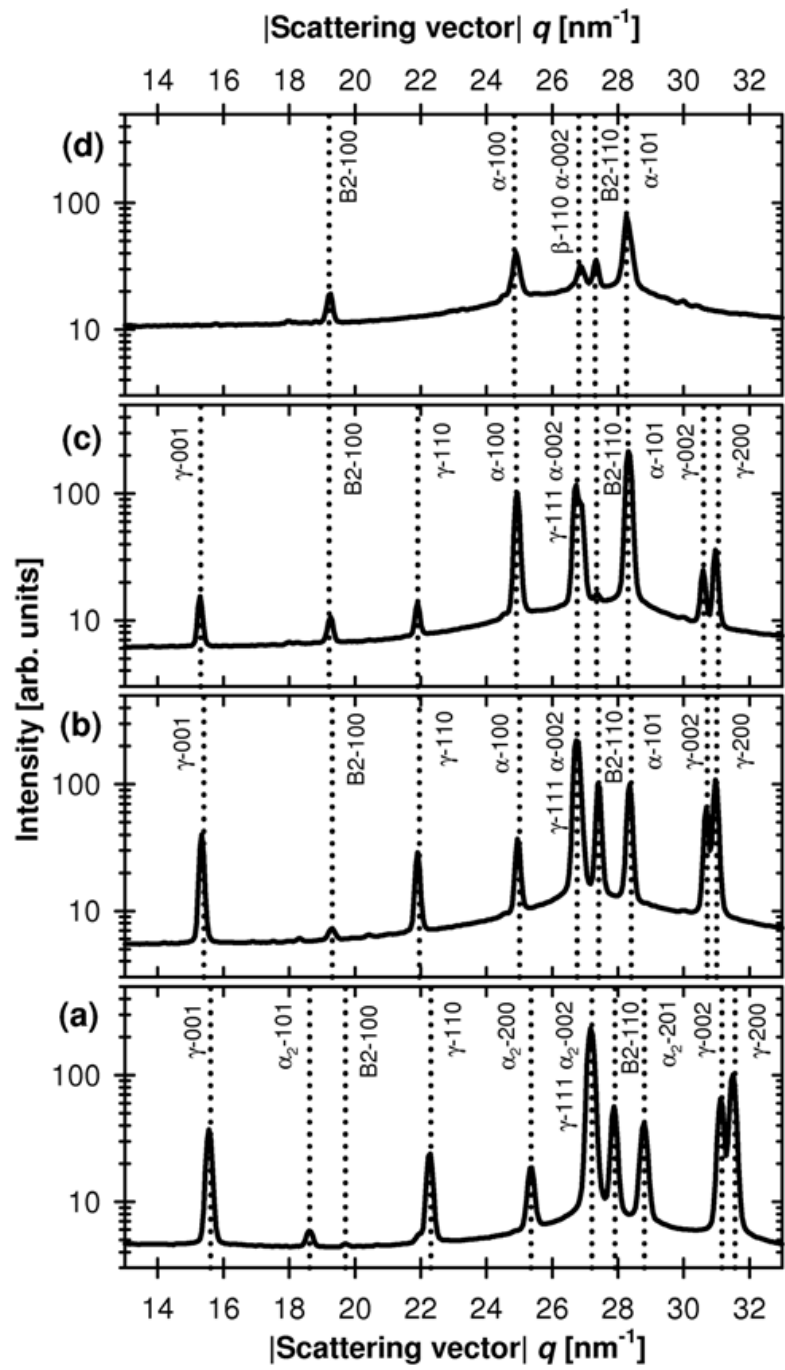


Figure 4



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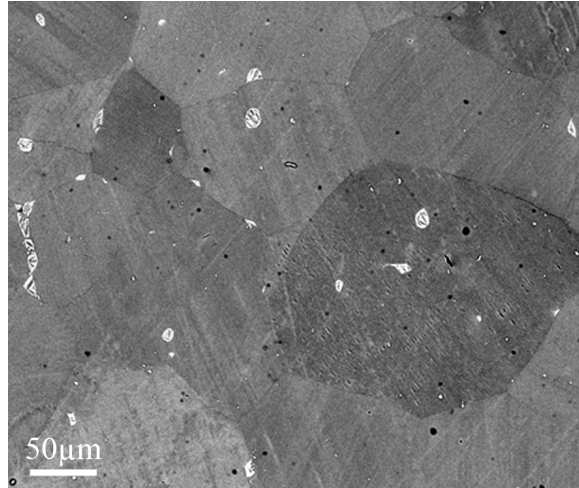


Figure 5

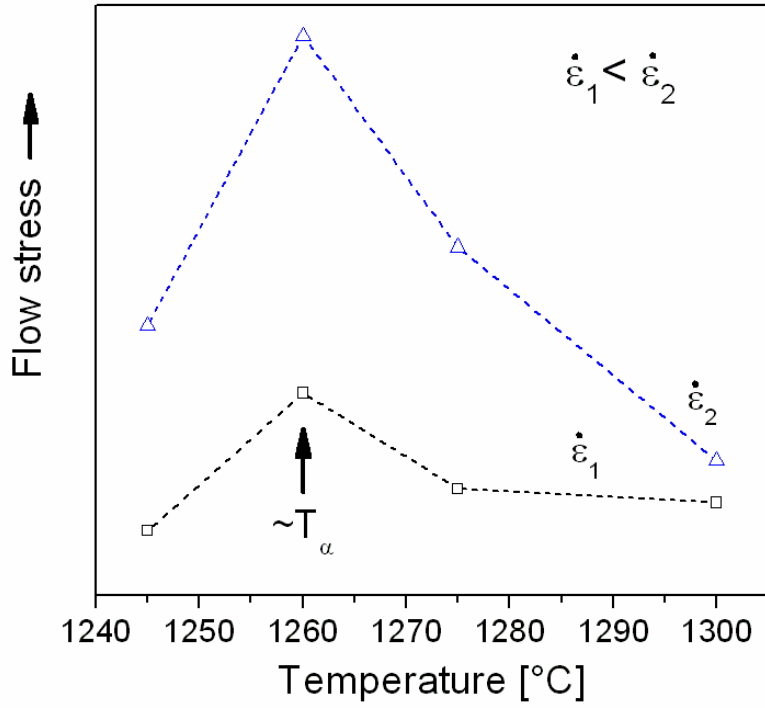


Figure 6

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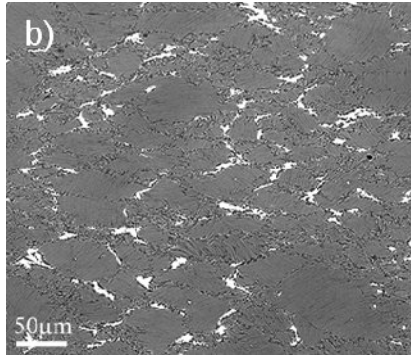
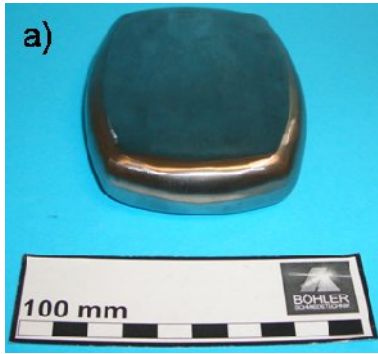


Figure 7 a) – c)

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## Table

**Table 1:** Transformation temperatures and corresponding phase fields of alloy Ti-43Al-4Nb-1Mo-0.1B as determined by in-situ high-energy X-ray diffraction. The determined temperatures fit well to the results obtained from DSC measurements and the evaluation of static heat-treatments [17,32].

<i>phase field</i>	$\alpha, \beta/B2$
$\alpha$ -transus temperature	$1285^{\circ}\text{C}\pm 5^{\circ}\text{C}^*$
<i>phase field</i>	$\alpha, B2, \gamma$
eutectoid temperature	$1165^{\circ}\text{C}\pm 5^{\circ}\text{C}$
<i>phase field</i>	$\alpha_2, B2, \gamma$

\*The discrepancy between the  $\alpha$  transus temperature determined by HE-XRD and DSC measurements is most likely due to the finite heating rate of the in-situ XRD experiment. This divergence is not observed at the eutectoid temperature, because the ordering reaction from  $\alpha_2$  to  $\alpha$  is rather fast compared to the  $\alpha$  transus reaction.