



Nucleation and thermal stability of carbide precipitates in high Nb containing TiAl alloys



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ABSTRACT

This work monitors the nucleation, growth and coarsening of carbides in powder metallurgically processed, high Nb containing TiAl alloys. The effects of carbon content, annealing conditions and internal defects on the precipitation and stability of carbides were systematically investigated by high energy X-ray diffraction and transmission electron microscopy. In general, at 800 °C the carbide microstructure can still change significantly up to 1000 h of annealing. It is found that a higher carbon concentration promotes the carbide precipitation process and increases the thermal stability of carbides. Internal interfaces and other crystallographic defects act not only as heterogeneous nucleation sites for perovskite Ti_3AlC carbides but also as carbon sinks. This retards the carbide nucleation in the interior of γ -TiAl grains. By homogenising the carbon distribution through solution heat treatment the nucleation of carbides in the γ -TiAl matrix is significantly accelerated as an effect of higher matrix carbon content.

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1. Introduction

γ -TiAl alloys were recently introduced as low pressure turbine blades with about 700 °C service temperature in civil aero engines, this being the first industrial use of this material besides niche applications [1]. Nevertheless, for other applications, namely turbochargers for gasoline engines and turbine blades at higher operating temperatures, an increased temperature capability of TiAl alloys is desirable [2,3]. Precipitation hardening by carbides is a possible means to achieve this [4,5]. By addition of 0.1–1 at% carbon carbides could form in the material and significantly improve the high temperature strength [6–8]. Especially a fine population of perovskite so called P-type Ti_3AlC carbides, as encountered in alloys with lower carbon contents (0.1–0.5 at%), proofed to be efficient to harden the material [7–9]. At higher carbon contents a more carbon rich hexagonal H-type Ti_2AlC carbide variant is formed which exists as coarser particles at grain boundaries [10] and is not as efficient in hardening the material compared to P-type carbides [4]. Up to now most of the available studies were performed on alloys with rather high aluminium content and limited additions of

ternary or quaternary alloying elements (2 at% or below) [7–9,11,12]. From a technological point of view up-coming alloys differ strongly from these, being lower in aluminium and having ternary or quaternary alloying additions of up to 8 at% niobium and other β -stabilizing elements. This new generation of alloys could also be fundamentally different with respect to carbide precipitation as niobium slows down diffusion and leads to a refinement of the microstructure [4,13]. Also there are hints in literature, that the presence of niobium alters the binding conditions of carbon in the γ -TiAl phase in a way that the solubility of carbon is raised [14]. In addition, most of the studies on carbide precipitates in TiAl alloys in literature did not involve complex heat treatment schemes [6,15–20]. There were already some reports concerning carbide precipitates in TNM/TNB alloys in short-term annealed condition or in HIPed condition [10,15]. But for applications such as turbine blades in aircraft engines, the long term stability of the carbides is decisive. Therefore it is important to characterise and understand the nucleation, growth and coarsening of carbide precipitates in this new generation of TiAl alloys as a first step to optimise the carbide hardening of these alloys.

In the present work the precipitation and thermal stability of carbides in Ti–45Al–5Nb–xC alloys (high Nb and low Al concentration) in different heat treatment conditions are investigated. By use of diffraction methods and transmission electron microscopy it

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is cleared how crystallographic defects (i.e. grain boundaries and dislocations), local variations in carbon concentration and the thermal history of the specimens influence the carbide nucleation, growth and coarsening.

2. Experimental

2.1. Materials

Two alloys were used in this research with constant contents of aluminium and niobium and variations in the content of carbon balanced by an appropriate change in titanium content. The base composition of the alloys is Ti–45Al–5Nb–xC (all in atomic percent; $x = 0.5$ and 0.75). To minimise side effects of chemical inhomogeneities of the specimen powder metallurgy (PM) was chosen for the production. The samples were compacted from pre-alloyed Ti–45Al–5Nb–xC powders produced by the plasma melting induction guiding gas atomization (PIGA) technique. Details about the powder production can be found in Ref. [21]. The actual contents of C in the Ti–45Al–5Nb–0.5C and Ti–45Al–5Nb–0.75C powders are 1530 and 2170 $\mu\text{g/g}$ respectively, which are very close to the nominal concentrations (1480 and 2210 $\mu\text{g/g}$). The size fraction of powder particles with diameters $<180 \mu\text{m}$ was separated by sieving, then filled into Ti cans, degassed, sealed and consolidated by hot isostatic pressing (HIP) at $1250 \text{ }^\circ\text{C}$ and 200 MPa for 2 h.

It is of interest to note that in all alloys after HIP consolidation already carbides were present. These results were published in detail elsewhere [10] but for a complete understanding of the following results it is of benefit to briefly describe type, location and amount of carbides already present in the material prior to any further heat treatments. The alloy with 0.5 at% carbon was virtually free of carbides after HIP consolidation. By tedious search using TEM few P-type carbides could be found with a fraction so low that no respective peaks were discernable by high energy X-ray diffraction (HEXRD) of the specimens. When the carbon content is increased to 0.75 at% the amount of carbides increases to levels where respective peaks are visible in the HEXRD patterns. The carbides present are predominantly of P-type while H-type carbides were observed only occasionally. The carbides of both types are located at internal boundaries. These are γ -grain boundaries in the case of P-type and former powder particle boundaries in the case of H-type carbides. For the P-type carbides an orientation relationship with a neighbouring γ -grain of $[101]_P \parallel [101]_\gamma$ and $(010)_P \parallel (010)_\gamma$ is determined while no orientation relationship exists between H-type carbides and neighbouring γ -grains. All alloys exhibit a so-called near-globular microstructure after the HIP consolidation consisting of γ -grains with smaller α_2 -grains located in between. Occasionally, few lamellar colonies are also present. A typical example for Ti–45Al–5Nb–0.75C is shown in Fig. 1.

2.2. Heat treatments

A number of heat treatments were performed to observe the development of the carbide microstructure with varied annealing times and temperatures. To generate defined initial conditions for these annealing treatments the Ti–45Al–5Nb–xC alloys were solution heat-treated to dissolve primary carbides and distribute carbon homogeneously in the material. The solution heat treatment was performed at $1250 \text{ }^\circ\text{C}$ for 5 h in air and the specimens were subsequently oil quenched. Annealing was done with specimens solution treated and with specimens in as-HIP condition. To distinguish these two series of specimens we will denote specimen states which were HIP'ed or HIP'ed and subsequently annealed with xC-HIP and xC-HIP + anneal. and specimen states

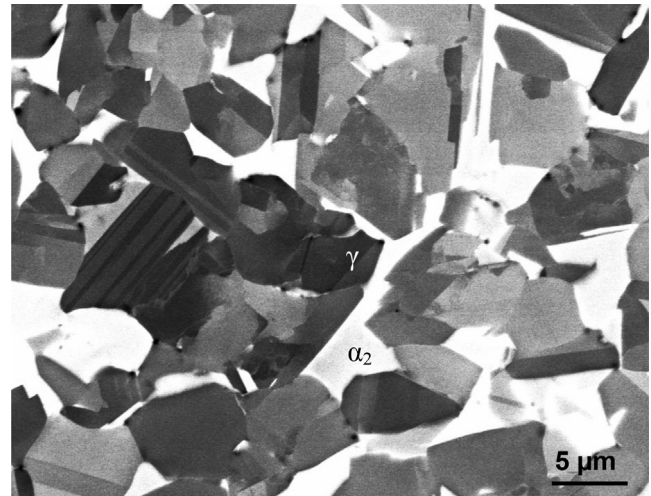


Fig. 1. SEM micrograph (in back-scattered electron mode) of HIP'ed Ti–45Al–5Nb–0.75C alloy: The microstructure is mainly composed of the γ phase (dark or grey contrast) and α_2 phase (bright contrast).

solutionised or solutionised and annealed with xC-SOL and xC-SOL + anneal. (x being carbon content in at.%). The annealing took place within the temperature range $800\text{--}1000 \text{ }^\circ\text{C}$ for 24, 48, 96 and 168 h. Additional long term annealing treatments at $800 \text{ }^\circ\text{C}$ for over 1000 h were performed to investigate the long term thermal stability of carbides. The atmosphere for all annealing treatments was air and the treatments were finished by furnace cooling of the specimens.

2.3. Characterization methods

High-energy X-ray diffraction (HEXRD) experiments were conducted at the HEMS and HARWI-II beam lines run by the Helmholtz-Zentrum Geesthacht at the Deutsches Elektronen-Synchrotron (DESY) in Hamburg. The X-ray beam had a cross section of $1 \times 1 \text{ mm}^2$ and a photon energy of 100 keV at HARWI-II and 87.1 keV at HEMS. These energies correspond to wavelengths of 0.1240 \AA and 0.1425 \AA , respectively. The reflections were either recorded with a mar345 image plate or a PerkinElmer XRD 1622 flat panel detector at the HEMS beamline and a mar555 flat panel detector at HARWI-II. An in situ heating HEXRD experiment was performed with a modified dilatometer DIL 805A/D [22,23] at HARWI-II, heating a cylindrical sample (4 mm in diameter and 10 mm in length) from room temperature to $1450 \text{ }^\circ\text{C}$. Due to the low fraction of carbides the Rietveld method could not be applied to determine the exact lattice parameters or phase fractions of carbides. Thus, the fraction of carbides was obtained by the Reference Intensity Ratios (RIRs) method [24] and the lattice parameters of P-type carbides were calculated as the average value from the peak positions of the (111), (200) and (220) reflections.

Scanning electron microscopy (SEM) was performed using a LEO Gemini 1530. SEM specimens were cut, ground and then electro polished with a solution of 26 ml perchloric acid (70%), 359 ml 2-butanol and 625 ml methanol at 30 V and $-30 \text{ }^\circ\text{C}$.

A Philips CM200 transmission electron microscope (TEM) operated at 200 kV was used for transmission electron microscopy investigations. TEM foils with a diameter of 2.3 mm were drilled, ground to a thickness of less than $120 \mu\text{m}$, and then thinned by twin-jet polishing at $25\text{--}35 \text{ V}$ and a temperature of $-40 \text{ }^\circ\text{C}$ with a solution of 26 ml perchloric acid (70%), 359 ml 2-butanol and 625 ml methanol.

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