Original

Microstructure Formation in Cast β-Solidifying γ-Titanium Aluminide Alloys

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Abstract. In view of the development of improved TiAl cast alloys the potential of the β/α transformation and its dependence on the addition of several alloying elements has been investigated. It was found that microstructural refinement in β solidifying alloys can be attributed to the alloying effect on the kinetics of the β/α transformation. This also holds for grain refinement through Borides which apparently serve as nucleation sites for the α phase in the solid-state transformation.

Introduction

In the last decade significant progress has been achieved in the development of γ titanium aluminide cast alloys for high-temperature applications and corresponding processing routes [1-3]. However, alloys like Ti-48Al-2Cr-2Nb suffer in the cast condition generally from coarse microstructures and pronounced casting textures [4]. According to the binary phase diagram [5], solidification of engineering γ titanium aluminide alloys occurs via the β and/or the peritectic α phase. Naka et al. [4] probably were the first to clearly point out that β solidifying alloys offer significant potential for grain refinement, because in the β/α transformation 12 orientations of α precipitates can be obtained from one parent β grain. Küstner et al. [6] paid attention to the marked difference in the solidification microstructure and texture between β and peritectically solidifying alloys and highlighted the isotropic and fine microstructure as well as the weak texture of β solidifying alloys. The difference to peritectic alloys was attributed to the columnar/equiaxed transition, which for the β solidifying alloys seemed to be favoured. Later, Küstner et al. [7] and Oehring et al. [8] explained this difference with the simultaneous growth of the β and α phase during solidification of peritectic alloys and noted that the α phase formed from the melt in peritectic alloys grows during the subsequent β/α transformation into primary β dendrites and prevents further nucleation of α from β. This observation was supported by a microstructure selection map calculated for binary TiAl alloys [8] according to the nucleation and constitutional undercooling model proposed by Hunziker et al. [9]. Imayev et al. [10] studied the influence of alloying elements on microstructure formation in β solidifying alloys and showed that grain refinement can be obtained by slowing down the transformation kinetics of the β/α solid-state transformation. It should be mentioned here that several authors have emphasized the role of the β phase with respect to grain-refinement and the multitude of phase transformations involving the β phase in multi-component alloys [11-16]. With

respect to grain refinement through the β/α solid-state transformation it has been observed that Boride particles in the alloys play a significant role [10, 14]. This is remarkable since the grain refinement effect by B additions first reported by Larsen [17] often has been considered to originate from solidification [18]. However, Hecht et al. [19] recently have shown that in Boron containing β solidifying alloys the α phase is formed with a random orientation from the solid β phase clearly indicating that the mechanism of refinement is based on heterogeneous nucleation of α on Borides. Despite this clear result B could also effect grain refinement during solidification, e.g. at higher concentrations of B and Al. In the present work, we have investigated the influence of several alloying elements including B on microstructure formation of β solidifying alloys and, in particular, paid attention to separate effects resulting from solidification from those occurring during subsequent solid-state transformations.

Experiments

Buttons of about 32 g weight were melted in a laboratory arc-melting furnace on a water-cooled copper plate under Ar atmosphere. The investigated materials included binary as well as multi-component alloys, which contained Al concentrations in the range 42 – 47 Al, varying Nb (5 – 8 at.%), B (0.1 – 0.2 at.%) and other alloying additions (Mo, W, Fe, C). The buttons were melted at least 7 times to ensure sufficient homogeneity.

For microstructural and analytical investigations slices were taken from the buttons, which contained the symmetry axis. Specimens for scanning electron microscopy were prepared by grinding and subsequent electropolishing at -19 V and a temperature of -45 °C in a solution of 600 ml methanol, 300 ml n-butanol and 60 ml perchloric acid. Scanning electron microscopy was performed exclusively in the back-scattering mode using a Zeiss DSM962 and a LEO Gemini 1530 microscope. Micro-analytical investigations were carried out at one of the microscopes equipped with an energy-dispersive X-ray (EDX) analysis system (Link Oxford), which was calibrated using TiAl alloy standards. The overall compositions of all buttons determined by EDX were found to match the nominal composition within a maximum deviation of 0.4 at.% in the Al concentration.

Results and discussion

In this work, the influence of alloying elements was investigated by systematically varying the Al and Ti concentration for a certain combination of the other alloying elements. After etching, in all alloy series a clear transition from equiaxed to pronounced columnar macrostructures was observed if the Al concentration exceeded a particular concentration between 45 and 46.5 at.%. The transition concentration depended on the amount of other alloying elements and can be explained by stabilizing the β with respect to the α phase. Alloys with lower Al concentrations as the critical value that contained no B showed relatively isotropic but coarse microstructures, while B containing alloys with lower Al concentrations exhibited equiaxed, fine and homogeneous microstructures. For Al contents above the transition concentration also grain refinement through B was found, however, the lamellae colony size generally was larger than in Al lean alloys.

By scanning electron microscopy of the alloy Ti-44Al-5Nb-0.2B (at.%) a microstructure similar to that of lamellar near-α Ti alloys was observed (Figure 1a). This morphology is explained by the solid-state reaction path β→β+α→α→α+γ. In the first transformation, Widmannstaetten α plates are precipitated from the β phase with different orientation relationships to the parent phase. This leads to the segregation of Ti and Nb to layers of the remaining β phase. Subsequently, γ lamellae are formed within the α plates. This type of morphology was also observed in micrographs of all alloys with Al contents below the above mentioned transition concentration. Similar microstructures have often been reported to occur in γ(TiAl) cast alloys [4]. From the microstructural observations

reported here it is concluded that in agreement with the phase diagram solidification occurred solely via the β phase for the foregoing alloys and no α phase is formed from the melt.

The microstructural observations were supported by microchemical analyses. In concentration line profiles taken perpendicular to the heat flow direction concentration fluctuations between 38 and 46 at.% Al were found in several alloys that exhibited this type of microstructure. These inhomogeneities indicate that the peritectic α phase was not formed, i.e. the alloys solidified completely through the β phase.
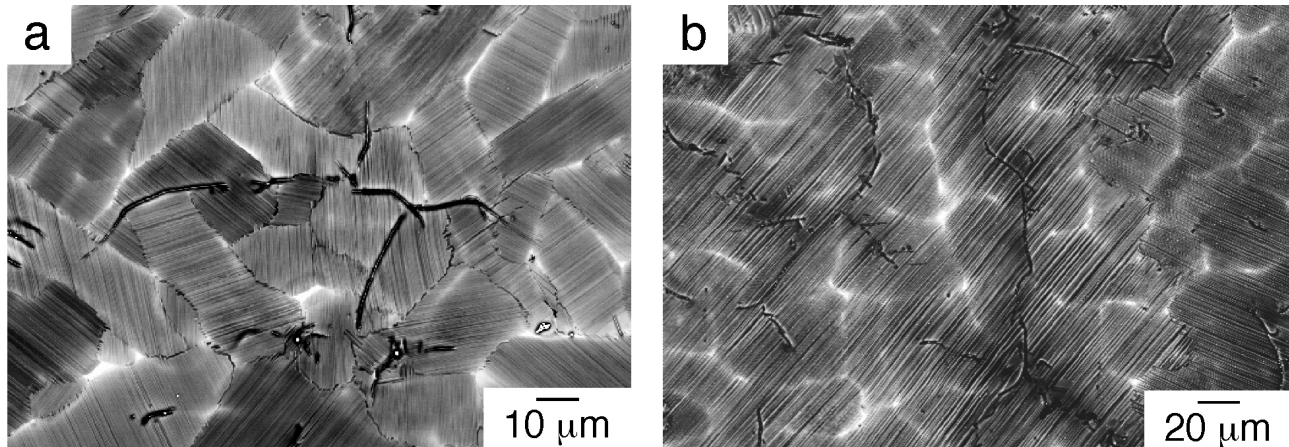

Figure 1. Scanning electron micrographs taken in the back-scattering electron mode from arc-melted buttons in the as-cast condition: a) Ti-44Al-5Nb-0.2B showing bright layers of the β phase located predominantly along colony boundaries. b) Ti-46Al-5Nb-0.2B. Please note the dendritic microstructure with brightly imaging dendrite cores and the uniform alignment of lamellae across interdendritic regions. In both images Borides are imaged with black contrast.

As an example for alloys with higher Al contents, the microstructure of Ti-46Al-5Nb-0.2B will be described in more detail in the following. As shown in Fig. 1b large lamellar colonies were observed which were elongated along the heat flow direction with a length of up 1000 μm. Clearly, a dendritic structure could be distinguished with dendrite cores that appear light in the image indicating the enrichment of Ti and Nb. The interdendritic regions contained Borides and in some cases single-phase γ grains. Quantitative EDX analysis revealed that the dendrite cores were rich in Nb and Ti and depleted in Al, whereas the interdendritic regions exhibited Al concentrations up to 48.5 at.%, clearly indicating that the peritectic α phase has formed in these regions. It should be noted here that the above-mentioned microstructure originating from the β→α transformation and reminiscent to Ti alloys was not observed in these alloys. This is attributed to the simultaneous growth of the α phase together with the primary β phase during solidification as explained in ref. 8. During further cooling the α phase grows into the β dendrites and prevents nucleation of α from β. As a consequence, Ti and other alloying elements that segregate to the β phase are enriched in the dendrite cores, where the β phase transforms at last or may remain. Küstner et al. [6] have shown that binary peritectic TiAl alloys exhibited the texture that is found for alloys solidifying through the α phase. Thus, α grew from the melt with a certain orientation to the heat flow direction and did not nucleate at the primary β phase. Here, it might be noted that peritectic solidifcation is a rather complex phenomenon that can lead to a rich variety of microstructures and to phase formation ranges differing from the equilibrium diagram [9].

From the results reported above it is concluded that grain refinement in β solidifying alloys can be explained by nucleation of the α phase at lower temperatures or higher undercoolings compared to peritectic alloys. Thus, the kinetics of the β→β+α→α-transformation seems to be the decisive

factor with respect to microstructural refinement. Besides nucleation, the kinetics of this transformation depends on the transformation temperature, the partitioning of the alloying elements between the β and the α phase and the diffusion coefficient of the alloying additions. Imayev et al. [10] have shown that by alloying with Nb and Mo the β/α transformation temperature can be reduced by approximately 50 K. Further, the authors demonstrated slight segregation of Nb and strong partitioning of Mo between the β and the α phase. Both effects result in significant refinement of the lamellae colony size if Nb or Nb together with Mo were added to a ternary alloy with the same concentration of Al and B. It is interesting to note that microstructural refinement even was obtained if the alloys were heat treated at 1450 °C followed by slow furnace cooling [10]. Similar refined microstructures could also be achieved in this work in quite a number of alloys containing (44 − 45.5) at.% Al, (0.1 − 0.2) at.% B, (5 − 8) at.% Nb and additions of Mo, W or Fe up to 2 at.%.

The microstructure refinement mechanism described so far is solely ascribed to a solid-state transformation. Thus, it could be suspected that it is also effective in B-free TiAl alloys. However, if the B-free alloy Ti-45Al-7Nb-1Mo is compared with the alloy Ti-45Al-7Nb-1Mo-0.2C-0.2B, a significant microstructural refinement by alloying with B becomes obvious (Fig. 2) as also found
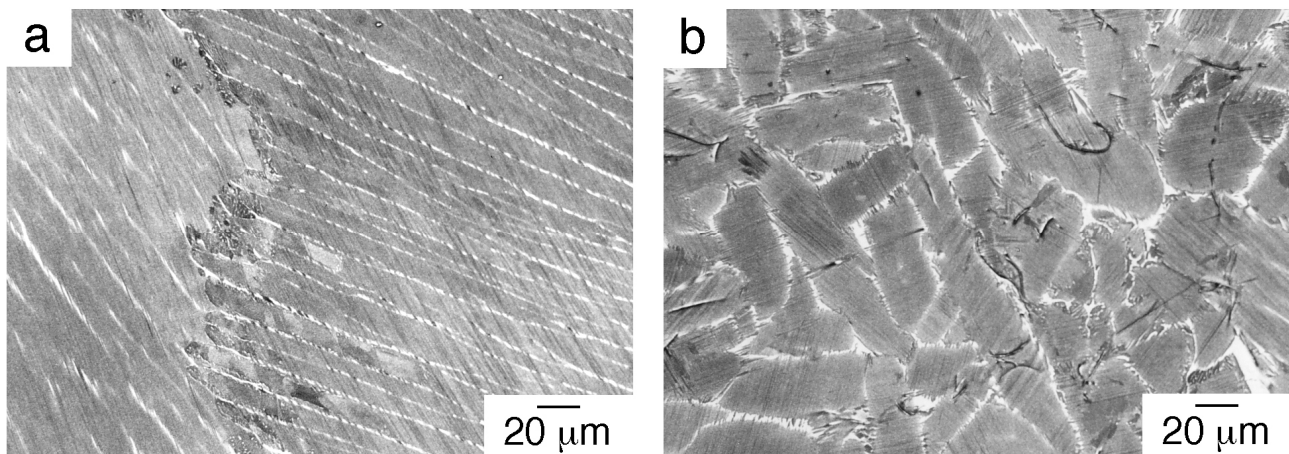

Figure 2. Scanning electron micrographs taken in the back-scattering electron mode from arc-melted buttons in the as-cast condition: a) Ti-45Al-7Nb-1Mo, b) Ti-45Al-7Nb-1Mo-0.2C-0.2B.

e,g. in ref. 10. The B-free alloy exhibits the same or a slightly lower width of former Widmannstaetten α plates as the B containing alloy. However, the length of such plates is much larger and many parallel former Widmannstaetten plates have the same alignment of α_2 and γ lamellae, i.e. during the β/α transformation big colonies of equally oriented parallel Widmannstaetten plates have formed that result in lamellar α_2 and γ colonies of the same size. In contrast, in the B containing alloy adjacent former α plates often have a different alignment of α_2 and γ lamellae. This observation indicates that nucleation of differently oriented α plates was easier in the B containing alloy or not restricted to the former β grain boundary as usually the case in Ti alloys. This could indicate heterogeneous nucleation of α precipitation by Borides.

In order to examine this hypothesis an alloy of composition Ti-44.5Al-(5-7)Nb-(0.5-1.5)Mo-0.1B with a similarly fine cast microstructure as shown in Fig. 2b was heat–treated in the α single-phase field at 1310 °C and subsequently quenched in oil. After this heat-treatment the alloy showed a microstructure consisting of α grains with a size 0.5 − 1 mm (Fig. 3). This material was then subjected to a second heat treatment in the β phase field at 1450 °C followed by air cooling or by furnace cooling with an initial rate of 1 K/s. In the air-cooled specimen a relatively fine dispersion

of β layers in lamellar colonies was observed, however, the colony size was approximately in the range 0.5 – 1 mm. In contrast, the furnace-cooled specimen exhibited a homogeneous microstructure of lamellar colonies with a size of around 50 – 100 μm. The orientation of the lamellae appeared to be randomly distributed. These results clearly show that grain refinement by B additions in β solidifying alloys has to be attributed to heterogeneous nucleation of the α phase during the β/α transformation as earlier proven by Hecht et al. [19]. Notably, it is only achieved if the cooling rate is not to fast, i.e. the formation of Widmannstaetten colonies nucleated at former β grain boundaries seems to be faster than nucleation of α at Borides inside β grains but the former transformation mode apparently requires higher undercooling.
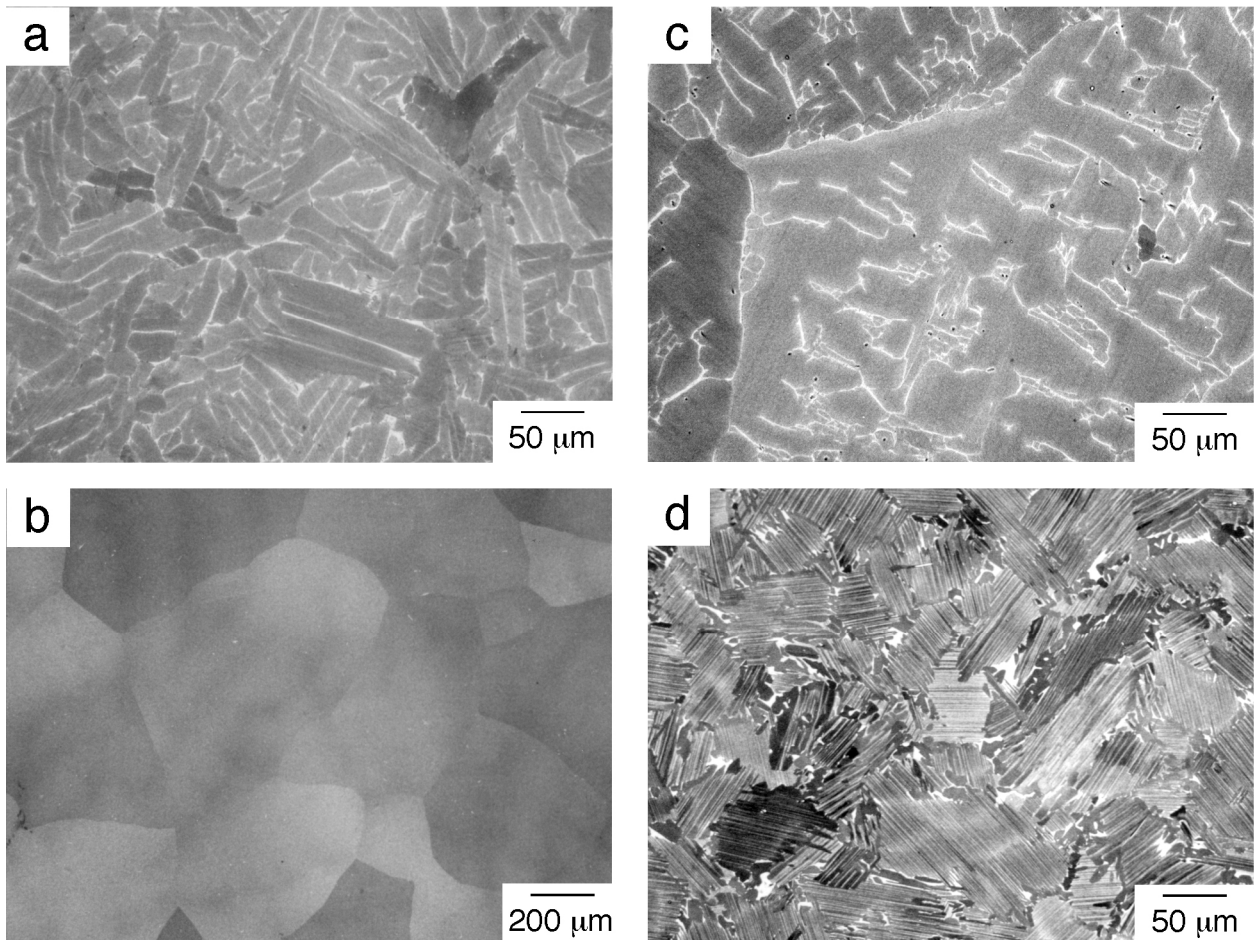

Figure 3. Scanning electron micrographs taken in the back-scattering electron mode from arc-melted buttons of an alloy Ti-44.5Al-(5-7)Nb-(0.5-1.5)Mo-0.1B: a) as-cast condition, b) heat-treatment 2 h/1310 °C/OQ, c) heat-treatment 2 h/1310 °C/OQ + 0.5h/1450 °C/AC, d) heat-treatment 2 h/1310 °C/OQ + 0.5h/1450 °C/FC.

Conclusions

Our experimental observations strongly indicate that the β/α transformation is the key factor to understand grain refinement in β solidifying alloys. In peritectic alloys, the α phase is formed from the melt which inhibits further nucleation of α from the β phase at higher undercoolings and thus, results in coarse microstructures. Further, it has been shown that grain refinement in β solidifying alloys with small additions of B can be obtained through the β/α transformation, without melting the material. Thus, in such alloys grain refinement through B is a solid-state transformation effect, as earlier has been proven by Hecht et al. [19].

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