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Solidification and grain refinement in Ti(48-50)Al2Mn2Nb1B alloys

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ABSTRACT

Two TiAl-based alloys, Ti48Al2Mn2Nb1B and Ti50Al2Mn2Nb1B, were prepared via conventional ingot melting and unidirectional solidification (Bridgman process) to investigate their solidification behaviour and the effect of boron addition on grain refinement. Both of them are found to be α solidifying alloys. Their microstructures are refined, featuring fine and randomly oriented lamellar colonies about 150 µm in size, in conventional ingots but the unidirectionally solidified alloys still have columnar microstructures. The entirely different grain refinement behaviour in ingots and Bridgman samples gave rise to the notion that the enhanced α nucleation from the liquid is the key to grain refinement in the two α solidifying alloys although the specific mechanisms are yet to identify.

1. Introduction

The successful insertion of titanium aluminides blades into low pressure turbines (LPT) in commercial aeroengines by General Electric and Pratt & Whitney in the past few years heralded the era of fruition of titanium aluminides research and development starting about forty years ago [1,2]. The application of light weight titanium aluminides in aeroengines enables great fuel saving and low impact to the environment by increasing fuel efficiency and reducing exhaust gases. Of the TiAl LPT blades for the two commercial aeroengines, GEnx and PW1100G, the large ones (GEnx) were manufactured using investment casting technology and the relatively small ones (PW1100G) were produced through forging. Investment casting is a well-established processing technology with a relatively low cost, especially for large sized low pressure turbine blades. However, the as-cast microstructures of many titanium aluminide alloys feature very large gains (or lamellar colonies in another term). Large grains leads to low tensile ductility and large property scatters. In order to maintain certain ductility and high property repeatability of components refined grains/lamellar colonies are essential [3]. To achieve fine grains in cast Ti48Al2Cr2Nb low pressure turbine blades great efforts have been put into developing and improving heat treatment schemes consisting of multi steps of solution treatments and hot isostatic pressing (HIPping) by GE [4,5]. Such activities were also succeeded by Safran S.A. later to develop a simplified heat treatment scheme for their cast semi-products of Ti48Al2Cr2Nb blades [6]. Despite the removal of HIPping the improved heat treatment scheme still takes a long time. Hence it is desirable to have fine microstructures in as-solidified TiAl alloys for being cost-effective and boron addition has been shown to serve the purpose. The practice of adding boron into TiAl alloys to refine as-solidified microstructures stretches back to the debut of Ti45Al2Mn2Nb(XD) around 1990 and TiAl alloys containing boron have been intensively studies since then [7,8]. Ti45Al2Mn2Nb1B has been investigated for cast LPT blade application and only a simple HIPping + ageing combination is needed after casting. Boron addition even is used to help refine the grain size in the latest wrought beta-solidifying TNM alloys [9].

Alongside technological advance in manufacturing aero-engine components the demand for scientific understanding into grain refinement mechanisms has been increased and so have been the research activities in this field. The dedicated and unambiguously evidenced research relating grain refinement to alloys' solidification pathways was first published in 2007 and has been continued even since [10-12]. The findings so far are summarised here. The most studied TiAl alloys have aluminium concentrations ranging from 43 to 52% (atomic percentage is used throughout, unless otherwise stated). At the low aluminium end the alloys solidify via the beta phase ($L \rightarrow \beta$), and at the high Al end via the alpha phase (L $\rightarrow \alpha$). In between a peritectic reaction, L + $\beta \rightarrow \alpha$, occurs. The extent of the peritectic reaction is dependent on Al concentration and with increasing Al the peritectic reaction is reduced in terms of the involved β phase volume. Whether the primary solidification phase is borides or metallic phases depends upon boron concentration in TiAl-B alloys. The dividing value lies approximately at 1% B, being slightly reduced with increasing Al. Below it alloys solidify via metallic β or α phase and titanium boride is the primary solidification phase when boron concentration is high, which can be seen from the measured or/and simulated phase diagrams [13,14].

Alloys with high boron concentration has long been studied. For example, the Ti45Al2Mn2Nb(XD) was derived from $TiAl-TiB_2(XD)$

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composites by reducing boron concentration from 7% to 1% [7]. Nonetheless only recently has the grain refinement mechanism in high B-containing alloys been seriously investigated by Gosslar et al. although a hypothesis of boride inoculation was mentioned for the XD composites much earlier [7,15]. Gosslar et al. found that in Ti45Al2B it was the heterogeneous nucleation of the β phase by primary titanium boride particles in the liquid metal that led to grain refinement [15]. It was also hinted, though no direct evidence was presented, that such heterogeneous nucleation could occur to the α phase in α solidifying alloys.

For most studied allovs the boron concentration is at 1% or below and in those allows the primary solidification phase is β or α . In the allovs of this category, grain refinement is closely related to solid B phase in the fully beta solidifying alloys and the alloys with significant extent of peritectic reaction. Grain refinement in beta solidifying alloys has been well documented and the refinement was achieved during $\beta \rightarrow$ α solid phase transformation rather than the solidification [10,16]. It is the already existing boride precipitates that stimulate α phase nucleation during solid phase transformation, enabling all the possible Burgers α variants, to achieve fine α grains, giving rise to fine lamellar colonies in subsequent $\alpha \rightarrow \gamma$ phase transformation. The critical boron concentration is very low and 0.1% is enough for some high-Nb containing alloys. In alloys with higher B concentrations, up to 1%, the excessive boron forms boride particles from the liquid in the interdendritic areas, leading to formation of non-Burgers α during $\beta \rightarrow \alpha$ solid phase transformation [16-18].

The TiAl alloys with a peritectic reaction at about 1490 °C can be separated into two groups: hypo-peritectic and hyper-peritectic. The first has a reaction of L + $\beta \rightarrow \alpha$ and the liquid was completely consumed before the β phase during the reaction. In the second group the liquid is in excess and some is left to solidify as the γ phase after β being exhausted. According to the phase diagram proposed by Witusiewicz et al. the Al concentration separating the two groups is 47.6% in Ti-Al binary systems [14]. The grain refinement in the alloys of the first group was found to start in the interdendritic regions but close to the β dendrites by precipitating peritectic α grains inoculated by existing boride particles in the liquid. The peritectic a grains have defined orientation relationship with the inoculating boride particles, and grow into the surrounding β dendrites, consuming both liquid and the β dendrites [11]. Owing to the randomness in orientation of the inoculating boride particles most α grains are also randomly oriented. The critical boron concentration for grain refinement, in this case, can be understood as the minimum amount of boron to form boride particles in the interdendritic liquid prior to the peritectic reaction.

As to the hyper-peritectic alloys, i.e. 47.6% < Al < 49.4%, there are no published work regarding their grain refinement mechanisms. Also no such work has been reported in α solidifying TiAl alloys to date. The purpose of this paper is to report our latest work on TiAl-1B with Al of 48–50% solidifying with limited peritectic reaction or alpha phase, in order to complete the spectrum of grain refinement mechanism in TiAl-B in the most studied Al and B ranges.

2. Experimental

The alloys used in this study were Ti48Al2Mn2Nb1B and Ti50Al2Mn2Nb1B. The alloying was to keep it consistent with previous work on Ti45Al2Mn2Nb1B, a well-studied alloy. They were prepared via cold hearth plasma arc melting in the form of 1 kg hemispherical buttons. The buttons were melted four times with turning about 90° each time before the next melting to improve homogeneity. Blocks weighing 20 g were taken from the buttons and remelted into finger-shaped ingots using non-consumable electrode arc melting in a water cooled copper crucible in Ar atmosphere and the ingots were melted twice and turned over after the first melting. The remelting was carried out under the same conditions for the two alloys to ensure the same/ similar thermal history of them. The aluminium concentration of the

finger ingots were measured using energy-dispersive X-ray spectroscopy (EDX) in a scanning electron microscopy (SEM), being 48.0% and 49.9% respectively which were calibrated with a Ti45Al2Mn2Nb standard sample.

Cylinders with 8 mm in diameter were also taken from the buttons as the feedstock for preparing Bridgman specimens. The samples were prepared using the Bridgman-Stockbarger technique in ACCESS Materials and Processes at a constant growth rate V of $8.33 \times 10^{-5} \, \text{ms}^{-1}$ and a constant temperature gradient in liquid at the solid-liquid interface G_L of $2 \times 10^4 \, \text{km}^{-1}$. Crucible tubes made of densely sintered yttria were used to minimise oxygen pickup during experiment. The furnace operates under 1000 mbar Argon. Solidification process was frozen by liquid metal quenching with a cooling rate of about 100°Cs^{-1} . Detailed description of this equipment and process can be found in Refs [16] & [19].

Samples for microstructural assessment were mounted into conductive Bakelite and ground successively to grit 1200 grinding paper. They were mechanically polished using $0.3 \,\mu\text{m}$ alumina suspension to finish. The as-polished samples were examined using SEM in backscattered electron (BSE) mode and using electron backscatter diffraction (EBSD) with an INCA software.

3. Results

3.1. Microstructures and orientation analysis of 20 g-finger ingots

The 20 g-finger ingots cooled down fairly fast during solidification owing to their small volume. It was noted that the temperature dropped by 600 °C after 1 min from power off and the cooling rate during solidification was estimated as at the order of 1° Cs⁻¹ considering the fact that cooling rate is normally high at high temperature and drops with cooling. Observation was made in areas away from the surface, especially the regions in contact with the crucible to avoid the chilling effect.

3.1.1. Ti50Al2Mn2Nb1B

According to phase diagrams Ti50Al2Mn2Nb1B should solidify via the α phase since the low alloying additions will not alter the phase diagram much. Fig. 1 shows a BSE SEM image of the ingot. The microstructure is consisted of lamellar colonies, the γ -TiAl grains with dark contrast (arrowed) and some fine rod-like TiB₂ precipitates (arrowed) also in dark contrast. The lamellar colony boundaries are delineated by white dashed lines according to their lamellar interface orientation. The lamellar colonies have a few important features which could help understand the solidification process. Firstly, they are almost equiaxed with an average size of about 150 µm determined using linear intercept method, in contrast to the columnar structure often found in boron-free alloys, indicating grain refinement. Secondly, each colony



Fig. 1. SEM BSE image of Ti50Al2Mn2Nb1B 20 g ingot.

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