

GRAIN REFINEMENT AND PROPERTY ASSESSMENT IN LOW AND HIGH Nb-CONTENT CAST TiAl-BASED ALLOYS

Xinhua Wu, D Hu and M H Loretto

IRC in Materials, The University of Birmingham, Edgbaston B15 2TT, UK

Keywords: TiAl alloys, Microstructural refinement, Casting.

Abstract

The problems associated with producing pore-free castings of TiAl-based alloys are reviewed and recent progress in improving the quality of castings discussed. The influence of the plastic anisotropy of fully lamellar TiAl alloys on their yielding behaviour is highlighted since castings generally have structures which are near fully lamellar. Similarly the influence of borides on the ductility of castings is discussed since these are formed in castings which are grain refined by addition of boron. On the basis of these considerations it is suggested that a new approach is required if fine-grained cast components are to be manufactured which have adequate and reproducible properties. Such an approach requires improved casting technologies and improved TiAl alloys with low Al contents (about 46at%) for high strength and high alloying addition (such as 8at%Nb) to provide adequate oxidation resistance and high temperature strength.

Recent work has shown that it is possible to produce pore-free castings. In addition it has been shown that the microstructure of B-free cast alloys such as Ti₄₈Al₂Cr₂Nb can be refined by tempering massively transformed samples in the alpha plus gamma two phase region so that the brittle borides present in samples grain refined through addition of B are eliminated. The microstructure which is generated by tempering massively transformed samples is highly convoluted because the alpha precipitates are formed on all four {111} in the massive gamma and is nucleated on the high density of defects present. The room temperature tensile properties of cast samples are very significantly improved by this type of heat treatment with a ductility of 1.3% compared with 0.2% or worse in the as-cast condition. Similar work is underway on alloys such as Ti₄₆Al₈Nb and the observations suggest that even finer microstructures can be generated than in the Ti₄₈Al₂Nb₂Cr alloy, but that the ductility is not improved to the same extent. Further work involving detailed transformation studies is underway with the aims firstly, of optimising the properties of heat treated samples of high Nb-content alloys and secondly, of defining the component size for different TiAl alloys for which this type of heat treatment can be used.

Introduction

TiAl-based alloys are being used for the manufacture of turbochargers in top-of-the-range automobiles and exhaust valves for formula 1 cars. The turbochargers are made using counter-gravity casting which appears to be a commercially viable manufacturing route, but no figures are available concerning failure rates or production costs. There are current plans to use TiAl alloys in further automotive engine tests with the aim of introducing these light weight exhaust

valves (a more demanding application than turbochargers) into a wider range of cars if they can be produced via casting rather than by the extensive thermomechanical processing route used for the formula 1 valves. Applications in aero engines are likely to use thermomechanically processed alloys in the short term, but from the cost-perspective there is always interest in casting as a process-route in aero engines as well as in automotives. The aim of this paper is thus to examine the factors which control the properties of cast products and thus to suggest process-routes and alloy compositions which will allow cost-effective castings to be produced.

There are major problems that have to be addressed in the manufacture of castings of TiAl-based alloys; the first is the production of sound, pore-free castings with an acceptable reject-rate and the second is the generation of a microstructure which has acceptable properties in cast components. These two topics will be addressed in turn.

Production of TiAl Castings

An extensive amount of work has been reported dealing with attempts to melt TiAl alloys in a cold wall furnace and to use gravity casting to produce the cast product (e.g. [1], [2], [3]) either by casting into ceramic or into metal moulds. One of the major problems with this approach is the fact that the superheat which can be reached even in recently designed cold-wall furnaces is only about 60°C. Modelling of the process has been carried out and confirms that the superheat would be expected to be limited to about 60°C with current designs of cold wall furnaces [4]. In the modern cold wall furnace the contact between the molten alloy and the furnace is limited to the base of the furnace so that there is no direct contact with the wall. The skull is typically only 5% of the initial weight of the melt as can be seen from the skull left behind after pouring the molten metal where only a skull-base is left with no indication of a skull formed on the furnace walls (other than that formed during pouring) [2]. These experimental observations contrast with earlier design cold wall furnaces in which the liquid metal made contact nearly up to the top of the furnace walls, giving a skull of about 30% of the melt which reduced the superheat to nearer 30-40°C. Although the superheat has been increased it appears that it is still not adequate to allow successful and reliable gravity filling of moulds.

Because the superheat is so limited preheated moulds tend to be used and the molten alloy is poured from the cold wall furnace very rapidly. This is of course not good practice and the resultant turbulent filling results in very poor yields with many of the individual castings not filling properly and/or containing a number of large pores [2]. Some typical porosity is illustrated in figure 1.

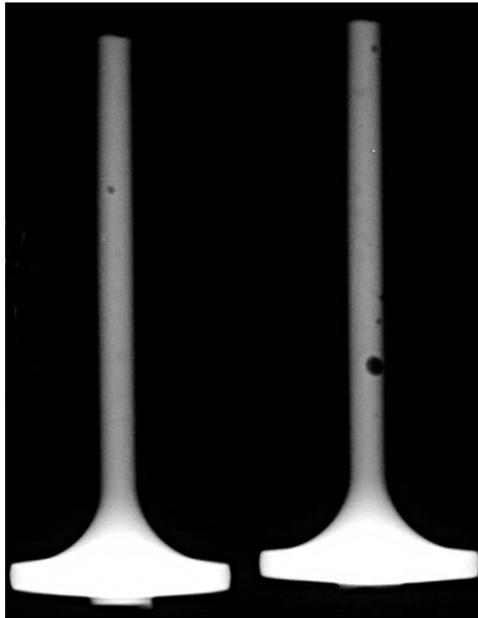


Figure 1 X-ray radiographs showing pores in valves of Ti48Al8Nb1B cast using a cold wall furnace to melt the alloy which was heated to about 50°C above its melting point before pouring into a ceramic mould pre-heated to 1000°C

Two different approaches are being investigated in order to overcome these problems. Firstly, counter gravity casting is being used in the IRC in conjunction with a cold-wall furnace (as has been done earlier for production of superchargers) and secondly conventional centrifugal casting is being used in conjunction with a cold-wall furnace and metal moulds (in Germany) and in conjunction with CaO crucibles and ceramic moulds in China [5,6].

In the work in the IRC the counter-gravity work is at an early stage, but already it has been found that the pore population can be significantly reduced, as indicated in figure 2 where the automotive valves cast using this technique do not show any large pores [5]. Sections taken from such castings show that there is still some fine porosity, but this is small enough not to lead to surface “dimpling” on HIPping. The reproducibility of the casting quality is not yet acceptable using this approach, but further development is underway.

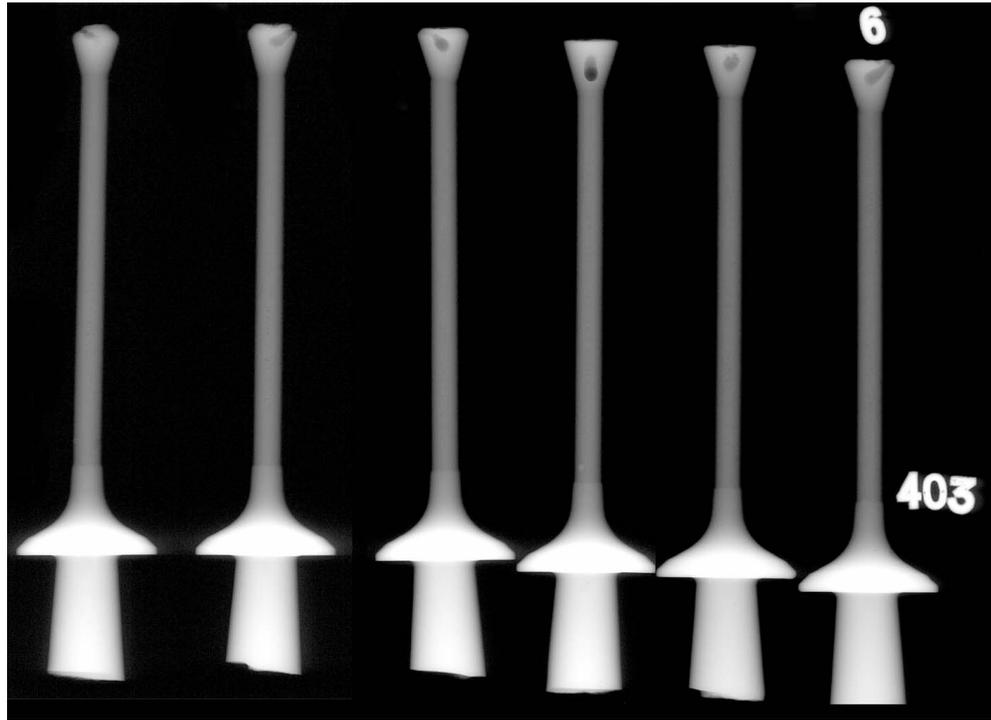


Figure 2 X-ray radiographs Ti44Al8Nb1B valves cast using counter gravity casting showing no obvious pores. Compare figure 1. (Courtesy R A Harding).

In the work in Germany centrifugal casting has been under development using metal moulds in a facility intended for mass production of car valves [7]. Reports on that project are not detailed but it is understood that there are still some problems to be overcome.

In the work in China the use of CaO crucibles has allowed superheats up to 180°C and this in conjunction with pre-heated moulds and centrifugal casting has yielded excellent castings with no porosity visible in the cast product [6]. An example of a roughly machined casting and a corresponding X-ray radiograph are shown in figure 3. No pores are visible on the X-ray radiograph but sectioning of these as-cast valves shows that there are a few extremely small pores in some parts of the centre line. HIPping these does not generate any surface dimpling and the evidence at this stage suggests that this route can produce reliable castings. Engine testing has not yet been completed since surface engineering is also required and optimisation of this process is underway.

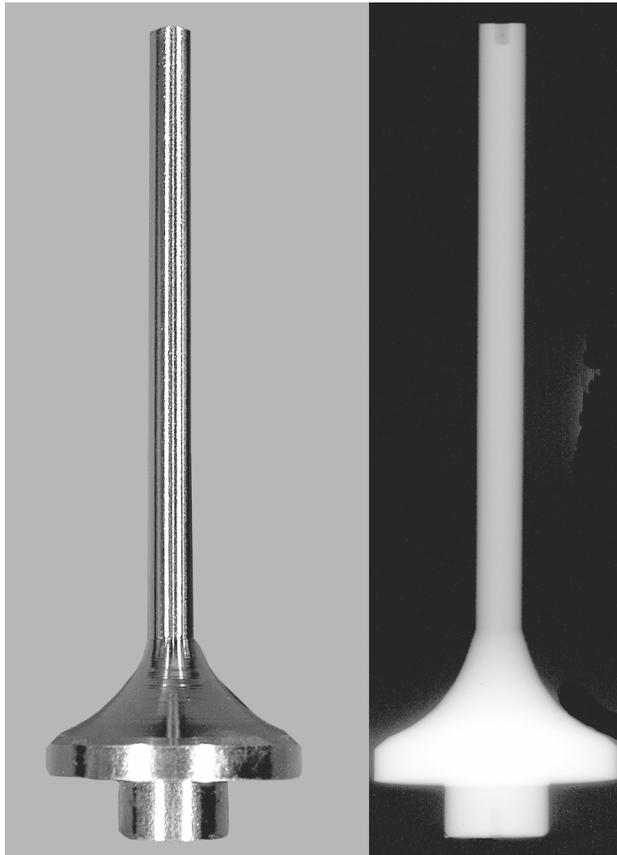


Figure 3 showing a roughly machined exhaust valve of Ti44Al8Nb1B and the corresponding X-ray radiograph, which does not reveal any porosity. Melting was carried out using a CaO crucible which allowed a superheat of 160°C, and the valve was cast using centrifugal casting into a pre-heated ceramic mould. (Courtesy IMR, Shenyang, China).

The colleagues who have developed this casting facility in China believe that if it is used to its full potential the cost of individual valves will approach that of top of the range valves used in the more expensive cars [8]. Clearly the problems caused by failure of an exhaust valve would be much greater than failure of a turbocharger and failure rates must be comparable with those of conventional steel valves. It appears however that casting technology required for a reasonably demanding application is now available. Further work is required before cast products of TiAl alloys are acceptable for aero engines but applications of cast products in stationary parts should be realised reasonably soon.

TiAl Alloy Development For Cast Components

Unless directional properties are required in a cast component it is generally accepted that grain refinement gives rise to castings which have optimal properties. In the case of cast samples of TiAl-based alloys the microstructure is virtually always a fully or near fully lamellar structure. As is well-known this microstructure is developed as the gamma phase precipitates on the (0001) planes in the alpha grains. The (0001) planes within each grain form parallel plates of gamma (twinned) and thus the lamellar length is defined by the pre-existing alpha grain size. The grain size is controlled by the residence time and cooling rate through the alpha phase field and by the presence of any grain refining additions. Typically in the absence of grain refining additions the grain size is 300µm for high Al-content alloys (about 48at%Al) and 150µm for lower contents (about 44at%Al). B additions can reduce these sizes to about 120 and 50µm

respectively. Complex heat treatments, which involve cycling samples through the alpha transus have been suggested as an alternative way to obtain fine grained castings [e.g. 9].

In the case of thermomechanically processed samples, which are generally processed in the alpha + gamma two phase field, a duplex microstructure is formed which is made up of gamma grains (formed during hot working) and lamellar grains (formed from the alpha grains produced during hot working, during cooling from the hot working temperature). The grain size is controlled by the extent and the temperature of hot working but is typically 20-30µm.

Cast products can be obtained with alloys such as Ti44Al8Nb1B which have ductilities of about 1% and UTS up to 600MPa in small diameter castings [10]. For larger sizes of castings the borides, which are inevitably formed in alloys refined by addition of boron, are coarse and act as failure initiation sites[11] and the influence of diameter of cast rods on tensile properties is shown in figure 4. The minimum ductility observed is thus reduced in larger samples and the scatter in properties is greater. Alloys such as Ti44Al8Nb1B can thus be used for producing fine grained castings up to 10mm in diameter and will give ductilities of about 1%. Perhaps slightly lower B contents would allow larger castings to be made. In view of the limitations associated with the addition of B for grain refinement of cast components some work is being carried out aimed at developing heat treatable castings, which have refined microstructures which do not contain borides.

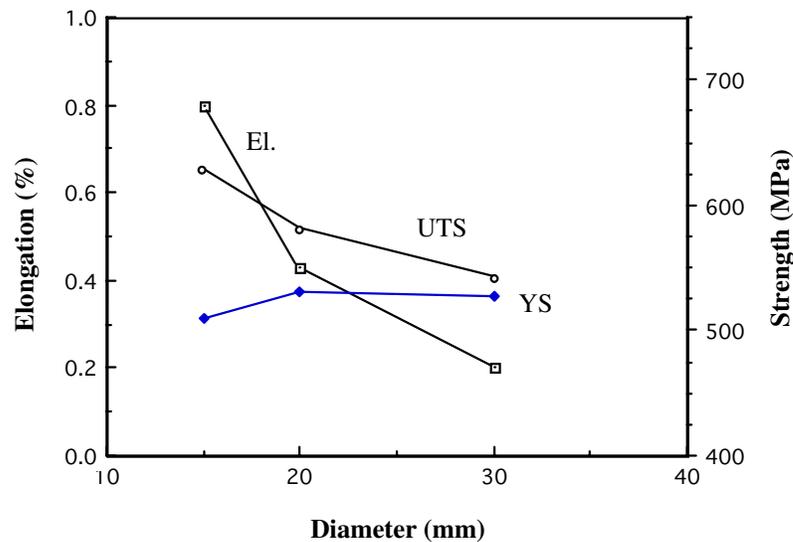


Figure 4 showing the influence of the diameter of cast bars of Ti44Al8Nb1B on the room temperature tensile properties.

Before discussing the development of heat treatable castings it should be noted that it has been reported that the fully or near fully lamellar structure has a disadvantage which is associated with the extreme plastic anisotropy [12] of this structure. The problem arises because soft grains (i.e. those with the lamellae inclined at angles between about 10 and 70° to the stress axis or perhaps larger gamma grains in near fully lamellar samples) yield at stresses which can be

very much less than those grains where the lamellae are oriented at 0 or 90° to the stress axis [12]. These soft grains generate dislocations at stresses well below the macroscopic yield stress and these dislocations build up large internal stresses. Acoustic emission and direct observations have shown that these stresses lead to cracking in fully lamellar Ti44Al8Nb1B at stresses well below the 0.2% proof stress of 625MPa [13, 14]. Several alloys were shown in

this earlier work to crack well below their yield points if they were lamellar but none showed pre-yield cracking if they were processed to produce a duplex microstructure [13]. Importantly it was also shown by tinting experiments [14] in combination with acoustic emission, that pre-stressing fatigue samples in tension or by fatiguing at low stresses, introduces cracks which then act as failure initiation sites on subsequent fatigue testing. Further detailed work has been carried out on samples which were tested in the as-machined condition, after deep electropolishing, after shot peening and after hand polishing, which confirmed that the cracking is controlled virtually totally by the microstructure, with surface preparation playing only a limited part [15]. Interestingly a forged TNB alloy (Ti45Al8Nb0.2C) which can be processed to give a heterogeneous microstructure with some regions being fine grained duplex and others relatively coarse fully lamellar shows pre-yield cracking in some samples but not in others. It has been found that in samples where pre-yield cracking is detected it always occurs in regions which are lamellar [16]. Fully duplex samples do not show pre-yield cracking.

In the light of the work summarised above further research is underway which has the dual aim of obtaining a fine grained structure in cast samples without using boron and of avoiding the fully or near fully lamellar structure. The current state of this work is discussed below.

The heat treatment which has shown the most promise involves rapid cooling to produce a massively transformed microstructure, followed by tempering at a high temperature (typically about 1320°C) in the alpha + gamma two phase field [17]. This process has many advantages but also presents some difficulties. One of the main advantages lies in the type of fine microstructure which is developed during the heat treatment, which is very different from that found in either cast or in thermomechanically processed samples. One of the difficulties is the fact that rapid cooling is required (air cooling for some alloys, but oil quenching for others) in order to induce the massive transformation and this requirement may limit the size of component which can be treated in this way and may, as discussed below, result in cracking during quenching.

For samples which are transformed massively and then tempered, the microstructure is formed by precipitation of alpha on all four {111} in the gamma formed by the massive transformation. This transformation is thus very different from the transformation which yields a fully lamellar structure where precipitation occurs only on the {0001} planes in the alpha phase. The massively transformed gamma contains a high density of defects, which act as sites for nucleation of the alpha and this taken together with the fact that growth takes place on all four {111} means that a highly convoluted microstructure is developed. The scale of the microstructure is also influenced by the tempering conditions for 1h to 2h at temperatures which are high in the two phase region have been found to generate optimum structures. This type of tempering treatment has the obvious advantage that it will be stable at the highest temperatures at which TiAl alloys will be used. A typical example of a structures generated using this type of heat treatment is shown in figure 5.

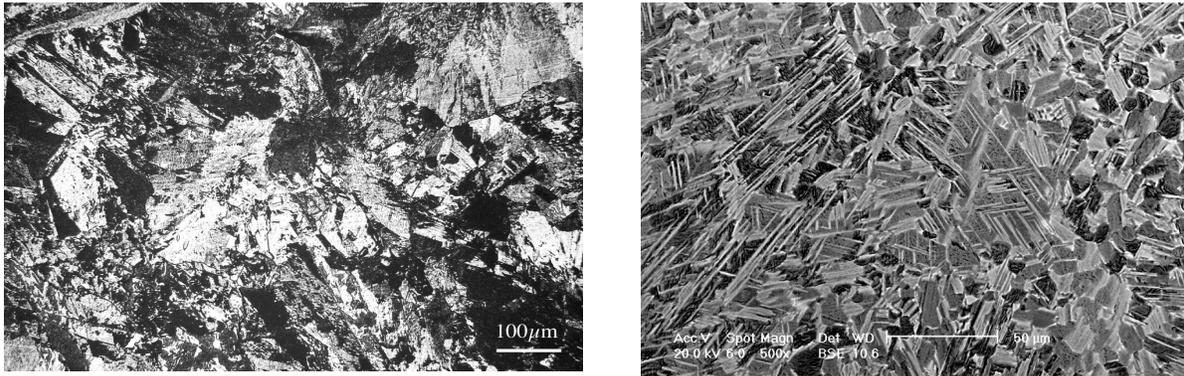


Figure 5. Backscattered scanning electron micrographs of Ti48Al2Nb2Cr showing in (a) the massively transformed structure in a sample air cooled from the alpha phase field and in (b) the convoluted microstructure formed on tempering the massively transformed structure for 2h at 1320°C. See text.

The size of the alpha grains, which existed prior to the transformation, cannot be discerned in this complex microstructure and it is this fact, together with the non-directional nature of the microstructure, that makes this structure of interest for cast products [17].

The potential of the heat treatment in overcoming the problems in fully lamellar castings refined by B addition is illustrated in the table where the ductility and the strength of two high Al-content, low alloy addition TiAl-based alloys, treated to produce the convoluted microstructure are compared with that of the same alloy after forging or after addition of B [17] to refine the grain size of the forged sample.

Table showing some typical properties obtained in variously treated TiAl-based alloys

Alloy and treatment	0.2% yield (MPa)	UTS (MPa)	Ductility %
Ti48Al2Cr2Nb, forged +FC from 1380°C	312	347	0.5
Ti48Al2Cr2Nb1B forged +FC from 1380°C	345	445	1.4
Ti48Al2Nb2Cr. Cast and OQ from 1380°C. Tempered 1320°C 1h	425	622	1.3

FC is furnace cooled and OQ is oil-quenched.

At this stage no fatigue data are available for the oil quenched and tempered samples and no work has been carried out to assess the susceptibility of this convoluted microstructure to pre-yield cracking; the arguments presented earlier suggest that this microstructure would not show such cracking. Further testing work is underway.

The alloys for which the properties are shown in the table are first generation TiAl-based alloys with a total level of alloying addition of about 4at% and a Al content of 48at%. More recently, higher strength alloys, which also show much better oxidation resistance than the first generation alloys, have been developed. A typical recent alloy contains about 45-46at%Al and about 8 at% of a major alloying addition such as Nb, together with minor additions such as C or

Si to further improve creep properties. The low Al leads to a high room temperature strength, but reduced oxidation resistance but the high Nb more than compensates for the reduced oxidation resistance and also provides improved high temperature strength. The strength of these alloys in the wrought condition ranges from about 600MPa to 1000MPa with ductilities varying between about 1.5% (for the lower strength alloys) to only about 0.2% for the strongest. In view of the improvement in properties which can now be obtained in these TiAl alloys, in the wrought form it is clearly of interest to assess the response of cast samples of these alloys to the type of heat treatment used to produce the property improvements in Ti48Al2Cr2Nb shown in the table.

The microstructure that can be generated in Ti46Al8Nb is shown in figure 6, which was obtained by oil quenching a 10mm diameter bar from 1360°C and tempering it at 1320°C for 1h. It is clear that a very fine microstructure that shows no obvious anisotropy has been generated and the response to quenching and tempering is clearly very similar to that of the alloys shown in the table. The properties are however totally dominated by the presence of small cracks which form at some stage (presumably during quenching) and the samples all fail well below yield. Hot isostatic pressing does not improve the properties, presumably because the cracks are directly connected to the surface and encapsulation would be needed before there was any possibility of closing these cracks.

Further work is underway in which samples are being cooled at rates which should allow the massive transformation to occur but to temperatures well above room temperature to limit the magnitude of the internal stresses generated during cooling. For example experiments are underway in which samples are held at temperatures between 800 and 1100°C after being rapidly cooled from the single phase alpha field and then either up-quenched directly to about 1320°C or cooling (relatively slowly) to room temperature before tempering (or HIPping) at about 1320°C. The work carried out so far indicates that samples quenched from 1360°C into a fluidised bed transform massively only partially, with feathery structure being observed throughout most of the sample. It appears that somewhat more rapid cooling directly to lower temperatures may be required and this work is currently underway. The possibility of inducing the massive transformation (perhaps even in TiAl-based alloys with Al contents below 45at%) above the ductile-to-brittle transformation temperature would eliminate cracking and should thus allow large cross section samples to be heat treated in this way.

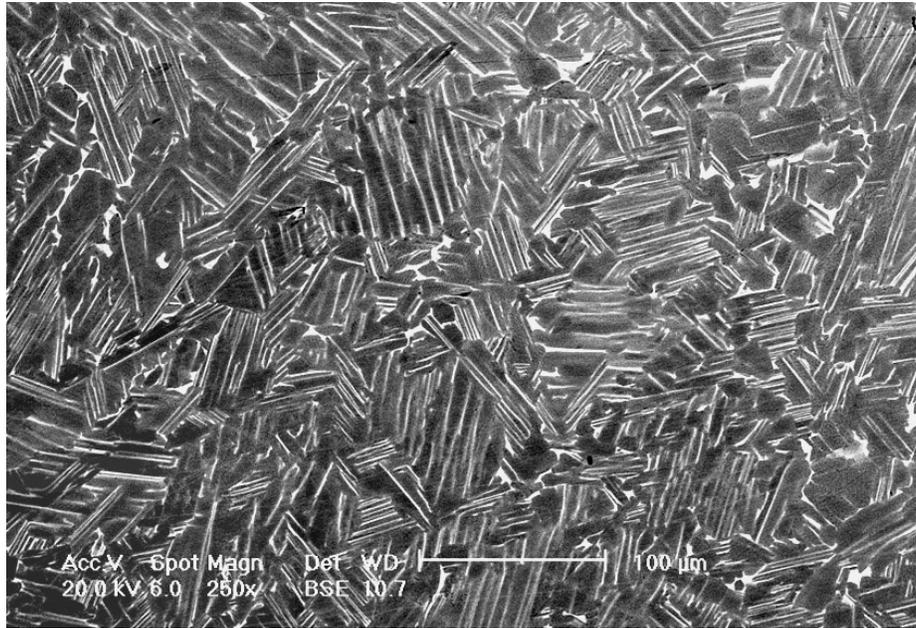


Figure 6 showing the convoluted microstructure formed in Ti46Al8Nb after oil quenching to room temperature and tempering the massively transformed sample at 1320°C for 1h.

Summary

It has been argued that the development of improved casting technologies and the development of high strength heat-treatable TiAl casting alloys will improve the chances of TiAl-based alloys being used both in aero engines and perhaps more importantly in mass market automobiles. Current work is aimed at assessing how generally the techniques discussed here can be applied.

Acknowledgements

The work reported in this review paper has been supported by EPSRC, by Niobium Products limited and by Rolls-Royce. Several research students have contributed to the work and their contribution is acknowledged in the text. Finally the work carried out at IMR in Shenyang has been supported by a Royal Society - Chinese Academy of Sciences Joint Research Programme.

References

- 1 J.P. Kuang, R.A. Harding, J. Campbell. Examination of defects in gamma Ti aluminide investment castings. *Intl J of Cast Metals Research*. Sept 2000 13, 125-134
- 2 J.P. Kuang, R. A. Harding, J. Campbell Microstructures and properties of investment castings of gamma titanium aluminides. *Mat Sc and Engg A* 329-331C, (2001), 31-37.
- 3 J.P. Kuang, R.A. Harding, J. Campbell A study of refractories as crucible and mould materials for melting and casting gamma TiAl alloys. *Intl J of Cast Metals Research*. 13(5), (2001), 277-292
- 4 V. Bojarevics, G Djambazov, R.A. Harding, K. Pericleous, M. Wickins. Investigation of the cold crucible melting process: experimental and numerical study. Accepted for publication in *Magnetohydrodynamics* 2003.

- 5 R.A. Harding (private communication)
- 6 W. Ke (private communication)
- 7 M. Blum, H. Franz, G. Jarczyk, P. Seserko, H.J. Laudenberg, K Segtrop, P. Busse. To be published in the *Proceeding of the 10th World Conference on Ti* (Hamburg) 2003.
- 8 Private communication W. Ke et al
- 9 J. Yang, J.N. Wang, Q. Xia. Refining of grain size of a TiAl alloy by cyclic heat treatment through discontinuous coarsening. *Intermetallics*. 11, (2003), 971-974
- 10 D. Hu “Effect of boron addition on tensile ductility in lamellar TiAl alloys.” *Intermetallics*, 10, (2002), 851-858
- 11 D. Hu, M. Wickins, R.A. Harding *Scripta Materialia* 47, (2002), 273-278
- 12 H. Inui, A. Nakamura, M.H. Oh, M. Yamaguchi. *Acta met* 40, (1992), 3095-
- 13 R. Botten, X. Wu, D. Hu, M.H. Loretto. “ The significance of acoustic emission during stressing of TiAl-based alloys. Part 1: Detection of cracking during loading up in tension”. *Acta Materialia* 49, (2001), 1687 -1691
- 14 X. Wu, D. Hu, R. Botten, M.H. Loretto. “ The significance of acoustic emission during stressing of TiAl-based alloys. Part 2: Influence of cracks induced by pre stressing on the fatigue life”. *Acta Materialia* 49, (2001), 1693-1699
- 15 X. Wu, M. Preuss, P. Withers, M.H. Loretto (accepted for publication *Intermetallics*)
- 16 N. Mota Solis, D. Hu, M.H. Loretto, Wayne Voice, M. Preuss, P J Withers, Xinhua Wu “The influence of microstructure and residual stress on pre-yield cracking in TiAl-based alloys” *Proc Thermec Conference* Madrid 2003.
- 17 Xinhua Wu, D. Hu, M.H. Loretto. “The influence of microstructure and surface residual stresses on pre-yield cracking in TiAl alloys” *Proc International symposium on gamma Ti aluminides (San Diego) 2003*. To be published.