Directionally Solidified Ti-48Al-2Cr-2Nb Made by Power Down or Bridgeman Processing

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ABSTRACT

The microstructural attributes of directionally solidified Ti-48Al-2Cr-2Nb (in at%) produced by the Power Down and Bridgeman techniques at various cooling rates/growth rates are presented. Fully lamellar α_2-Ti_3Al+γ-TiAl microstructures with γ particles on grain boundaries are present in all conditions. The directionally solidified structure consists of discrete regions of columnar grains either parallel or radial to the growth direction, as well as a region of equiaxed grains. The phase structure and lamellar structure made by the two techniques are examined. Increasing the cooling rate or growth rate decreases the portion of columnar grains parallel to the growth direction and decreases the lamellar interface spacing.

1. INTRODUCTION

A major research effort has occurred world-wide directed to the study of the intermetallic TiAl. This effort has been driven by the potential of TiAl intermetallics for applications as high temperature structural materials /1-3/. Among the possible microstructural conditions, the two-phase lamellar mixture of α_2-Ti_3Al and γ-TiAl offers a potentially useful combination of tensile and creep properties. Furthermore, this microstructural condition can be modified in terms of the size, shape, distribution and relative content of α_2 in the gamma matrix to achieve an improved balanced of mechanical properties at both elevated and room temperatures. Ti-48Al-2Cr-2Nb (hereafter referred to as 4822), as one of the second generation of the gamma TiAl alloys, may replace a nickel-based superalloy for a stationary structural application imminently /4/.

Research focussing on the processing options for TiAl is crucial for successful application of these intermetallics. Component microstructure and mechanical properties are sensitively dependent on the processing history, however the strong chemical activity of titanium introduces processing difficulties. Directional solidification (DS) processing has been successfully used in industrial production of superalloys for many years /5/. It is natural to apply the DS processing in development of TiAl alloys, particularly if the α_2+γ lamellar structure and the grain structure can be aligned parallel to the growth direction. Considerable previous research on DS TiAl alloys /6-14/ has focussed on obtaining an improved microstructure for mechanical properties. In particular it has been proposed /8/ that the primary phase and growth rate (withdrawal rate) during DS will influence the alignment of the lamellar structure with respect to the growth direction. Previous work has produced aligned columnar grains each with a length of several centimeters /11,13/.

On the other hand, very little work has been done on DS 4822. London *et al.* /11/ produced DS 4822 at a growth rate of 254 mm/h resulting in an aligned columnar grain structure. The angle of the lamellar structure varied with respect to the growth direction in each grain. A detailed examination of the microstructure and lamellar structure has not been presented. The lamellar boundaries in Ti-48at% at a growth rate of 90 mm/h /12/ and 200 mm/h /14/ are almost normal to the growth direction. It was postulated /14/ that hexagonal α was the primary phase with a preferred growth direction parallel to [0001] during the solidification. The gamma precipitated from α during further cooling with an orientation relationship of $(0001)_\alpha//(111)_\gamma$ and $<1120>_\alpha//<110>_\gamma$. Hence, the lamellar boundaries were all perpendicular to the growth direction.

Most of the previous research on DS processing of TiAl has utilized the floating zone method /5/, which is only useful for laboratory research since only small section sizes can be produced. In contrast, the Bridgeman (BD) processing technique uses a moving mould or furnace providing a controllable growth rate. Furthermore, the BD technique can be scaled up to industrial requirements. In addition to BD processing, the Power Down (PD) method is also a convenient DS processing technique, that controls the cooling or growth rate of the casting by reducing the power supply after melting and holding. The major difference between the PD and BD techniques is that the temperature gradient in a bar made by PD is progressively reduced from the bottom of the casting, which is in contact with a chill plate, to the top, while during BD processing the temperature gradient is constant.

To further study the effects of different DS processes on the microstructure of the 4822 alloy, DS bars were made by the PD and BD techniques with various cooling/growth rates. The DS structures, phase structures and lamellar structures are examined and the influence of the cooling/growth rate are evaluated. The two processes for making DS TiAl alloys are also compared.

2. EXPERIMENTAL

The 4822 master alloy ingots for the DS processing were cast by Wah Chang Co. using an induction skull melting processing. The DS processing was performed under flowing purified argon in a Power Down furnace or Bridgeman furnace. The two furnaces are shown schematically in Figure 1. The cooling rate close to the surface of a DS bar during PD solidification was

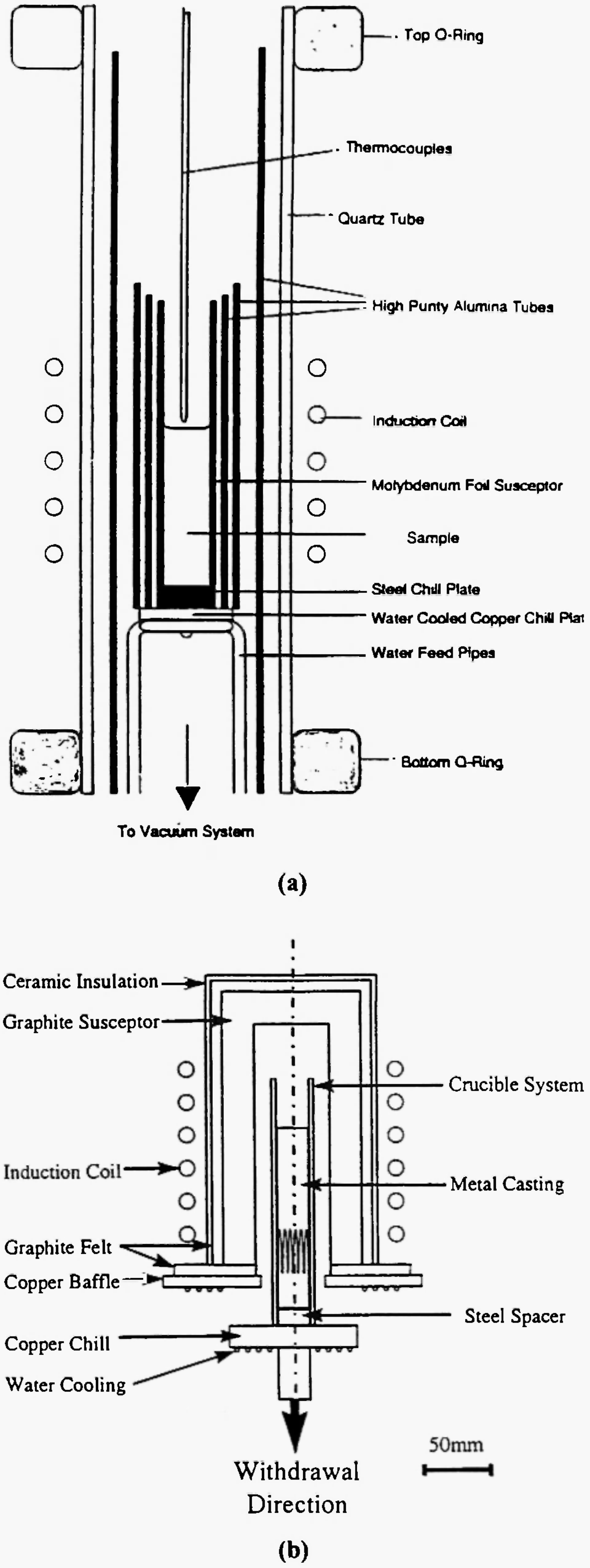


Fig. 1: Schematic drawings of DS furnaces (a) Power Down furnace (b) Bridgeman furnace.

estimated according to the thermal history recorded by a thermocouple. The withdrawal rates or growth rates of bars made by the BD process varied from 2 mm/min to 6 mm/min. All DS bars studied in this paper and corresponding characteristic processing parameters are listed in Table 1. The size of the DS bar made by PD is typically 30-40 mm in length and 20 mm in diameter and that by BD is typically 95-135 mm in length and 19-22mm in diameter (Figure 2).

Table 1

DS 4822 and related characteristic processing parameters (V: cooling rate, R: growth rate)

DS bars made by Power Down processing		DS bar made by Bridgeman processing	
No.	V (°C/s)	No.	R (mm/min)
PD1	2.05	4822-2	2
PD2	0.11	4822-4	4
		4822-6	6

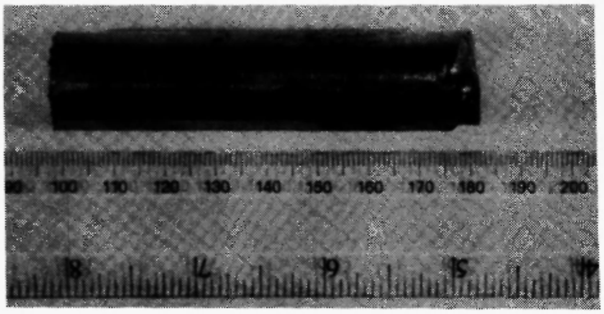


Fig. 2: A typical DS bar of 4822 alloy made by Bridgeman processing with a withdrawal rate of 4mm/min.

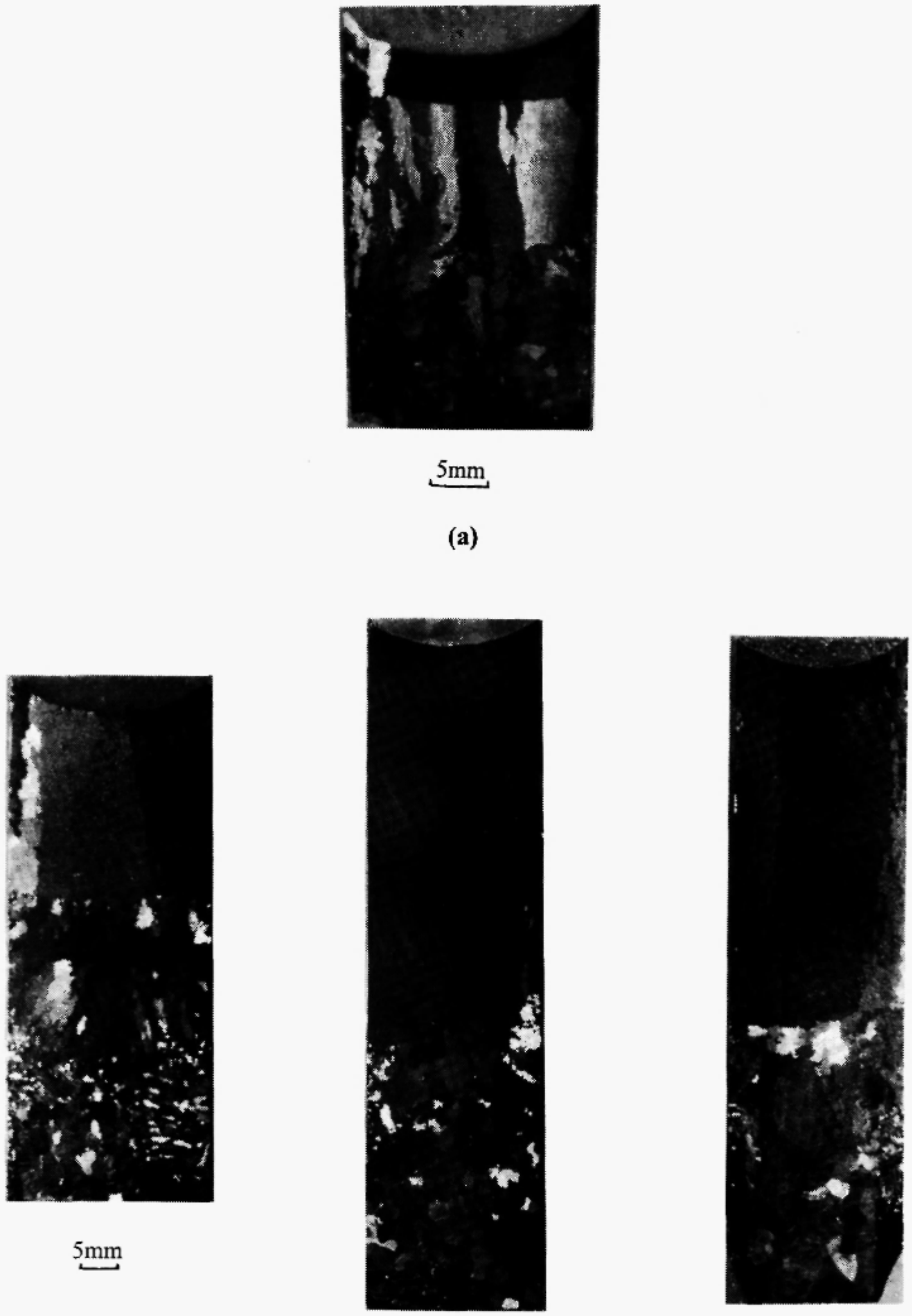
All DS 4822 bars were longitudinally cut into two halves by EDM (electric discharge machining). The microstructures of longitudinal sections were studied by optical microscopy and using a Hitachi S-570 scanning electron microscopy (SEM). The micro-analysis of the composition was obtained by energy dispersive spectroscopy (EDS) with a consistency of about 0.5at% for the elements in 4822. Each test was repeated at least two times or until the variation was <0.5at%. The average lamellar spacing, including both γ and α_2 lamellae, was measured using a linear intercept method in at least three random SEM micrographs. More than 100 lamellae were counted for determination of the lamellar spacing.

3. RESULTS

3.1 DS structures

The DS structure of the bar made by PD processing is shown in Figures 3(a) and 4(a). The structure consists mainly of four parts. The grains in the bottom part exhibit an irregular shape, but many nearly equiaxed grains can be seen as a result of the high temperature gradient corresponding to a large degree of undercooling in this region solidified close to water cooled chill plate. Above this equiaxed region there are short columnar grains oriented radially from the metal mould interface to the centre of the bar – these are hereafter referred to as radial columnar grains. The radial columnar grains are probably associated with the temperature difference between the centre of the bar and the wall of the mould at the same distance from the chill. Longer columnar grains are present in the middle part and their orientation is roughly parallel to the growth direction. The top part of the bar has a few coarse grains, probably nucleated from the surface cooled by the flowing argon during solidification. The temperature gradient from the equiaxed to parallel columnar grains decreases. The grain structure of the bar is consistent with thermal analysis of the PD process /5/ that indicates a progressively reduced temperature gradient from the bottom to the top of the bar with an increasing distance from the chill. The DS structure of PD1 is similar to PD2 except for the shorter length of the part containing parallel columnar grains.

Figures 3 (b) and 4 (b) show the typical DS structure of 4822 made by the BD process with different withdrawal rates. The DS structure in the three 4822 bars can be divided into three parts from the bottom to the top, similar to that in PD bars: equiaxed or nearly equiaxed grains (Part I), radiant columnar grains directed to the centre (Part II), and columnar grains parallel to the growth direction (Part III). The radiant columnar grains are not as apparent in 4822-4; however



(a)

(b)

Fig. 3: DS structures in longitudinal section of 4822 alloys (a) PD2 (b) 4822 (with a growth rate from left to right: 6mm/min, 4mm/min and 2mm/min).

the reason for this feature is not clear at the present time. Parts I and II are associated with unstable solidification before the thermal exchange between hot zone and cold zone reaches equilibrium during withdrawal. The top part observed in the PD bar is not seen in the BD bar, since during BD processing stable solidification corresponds to part III. The major area of the DS bars made by BD is the parallel columnar grains

Fig. 4: Typical DS structures of 4822 (a) PD1

which grew with a temperature gradient only in growth direction. It can be seen in Figure 4 that all of the columnar grains in both PD bars and BD bars have a poorly developed dendritic structure. Figure 5 shows that a larger portion of the bar is parallel columnar grains in BD bars than PD bars. The DS structure in 4822 BD bars mentioned above is similar to that found in another DS TiAl-based alloy made by BD processing /16/.

In addition to the major parts, a narrow layer of equiaxed or nearly equiaxed grains with an inhomogeneous size exists between part II and part III. Based on solidification theory /15/, the ratio G/R, between the temperature gradient G and growth rate R, dominates the morphology of the solidified grains. According to the change of grain shape from part I to part II, it is clear that the temperature gradient G was lower in part II than part I. Since the growth rate was constant during withdrawal, the solid/liquid interface stability was decreased. It is difficult to explain the

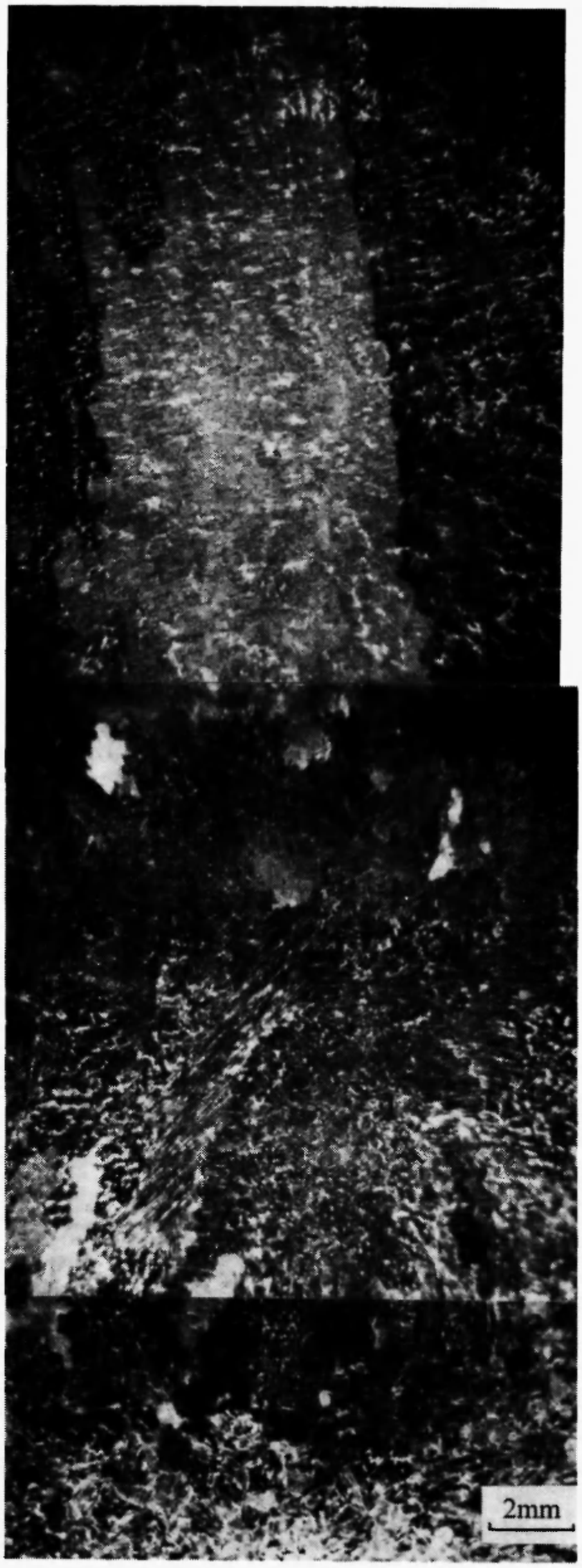
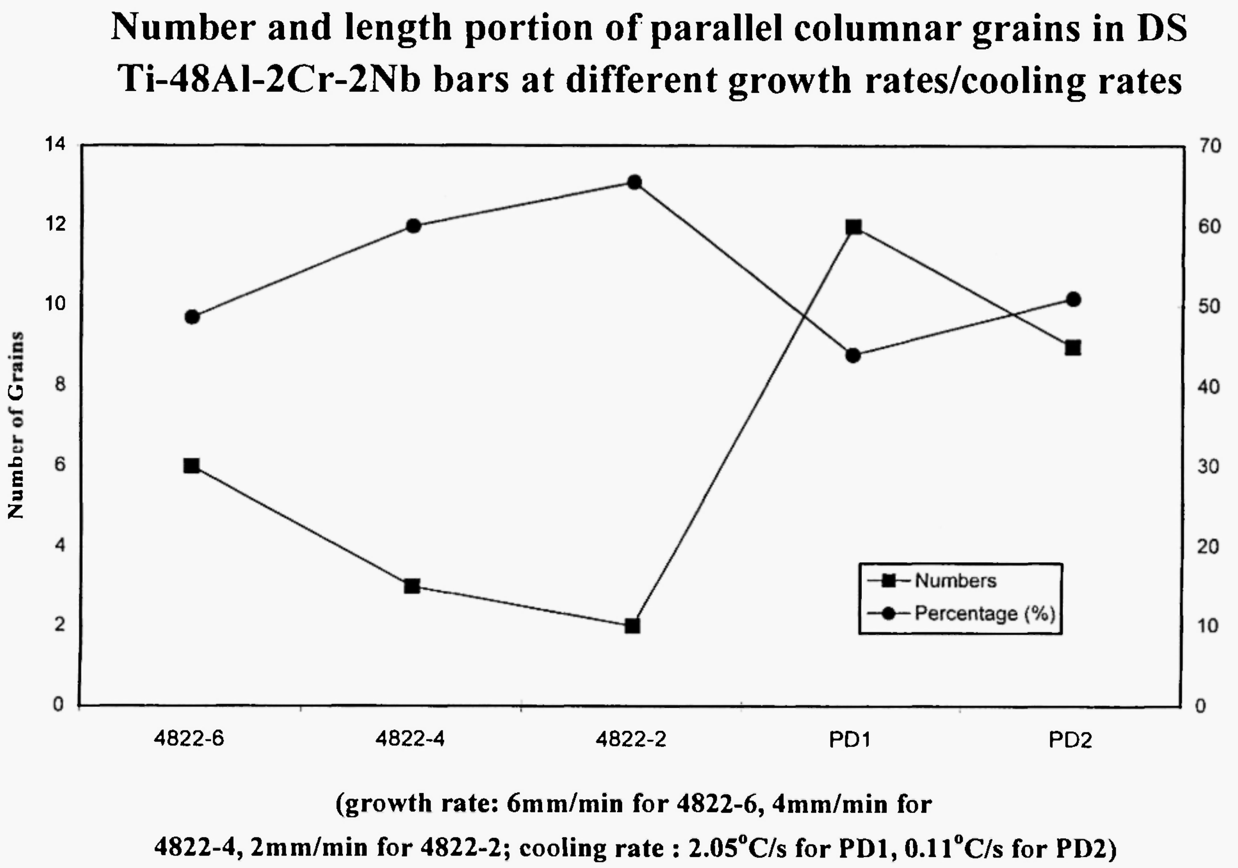


Fig. 4: Typical DS structures of 4822 (b) 4822-6.


Number and length portion of parallel columnar grains in DS Ti-48Al-2Cr-2Nb bars at different growth rates/cooling rates

(growth rate: 6mm/min for 4822-6, 4mm/min for 4822-4, 2mm/min for 4822-2; cooling rate : 2.05°C/s for PD1, 0.11°C/s for PD2)

Fig. 5: The number and length portion of parallel columnar grains in DS 4822 bars made by PD and BD processing.

sudden change of the grain shape from radial columnar to equiaxed and then from equiaxed to parallel columnar only using thermal flow theory, without accounting for the effects of segregation. The criterion of G/R is effective mainly in binary alloys for which the segregation in the melt would affect the thermal physical constants of the melt, which finally influences the above criterion. In the current alloy containing more alloying additions segregation or compositional changes around the layer may play more important role leading to its formation, although we did not find apparent composition differences across the layer of equiaxed grains. Similar equiaxed grains with even wider distribution were reported in DS Ti-46.5Al-1.5Mo-0.5B /13/, and were thought to be α phase, but no experimental results were presented to support this supposition. Further study is necessary to understand the mechanism responsible for the formation of this region.

3.2 Phase structures

The phase structure in 4822 is quite simple. Before DS processing the cast alloy has a duplex structure /1-2/ (Figure 6(a)), which is a mixture of α_2+γ lamellar grains and small equiaxed gamma grains. After DS processing, the majority of the DS bar consists of a fully lamellar structure (Figures 4 and 6 (b)). The average composition of the parallel columnar grains is listed in Table 2, with the compositions of the DS bars made by different processing conditions quite close. There is a minor volume fraction of a small blocky phase on grain boundaries (Figure 6 (c)-(e)). According to the average composition of this blocky phase (Table 2) and the lack of a lamellar structure, the blocky phase is concluded to be γ. The size of γ blocks increases a little with a reduction in the growth rate, as shown in Figs. 6(d) and (e). Generally, the phase structure in DS 4822 is similar to cast 4822 with a normal cooling rate /17, 18/ and cast

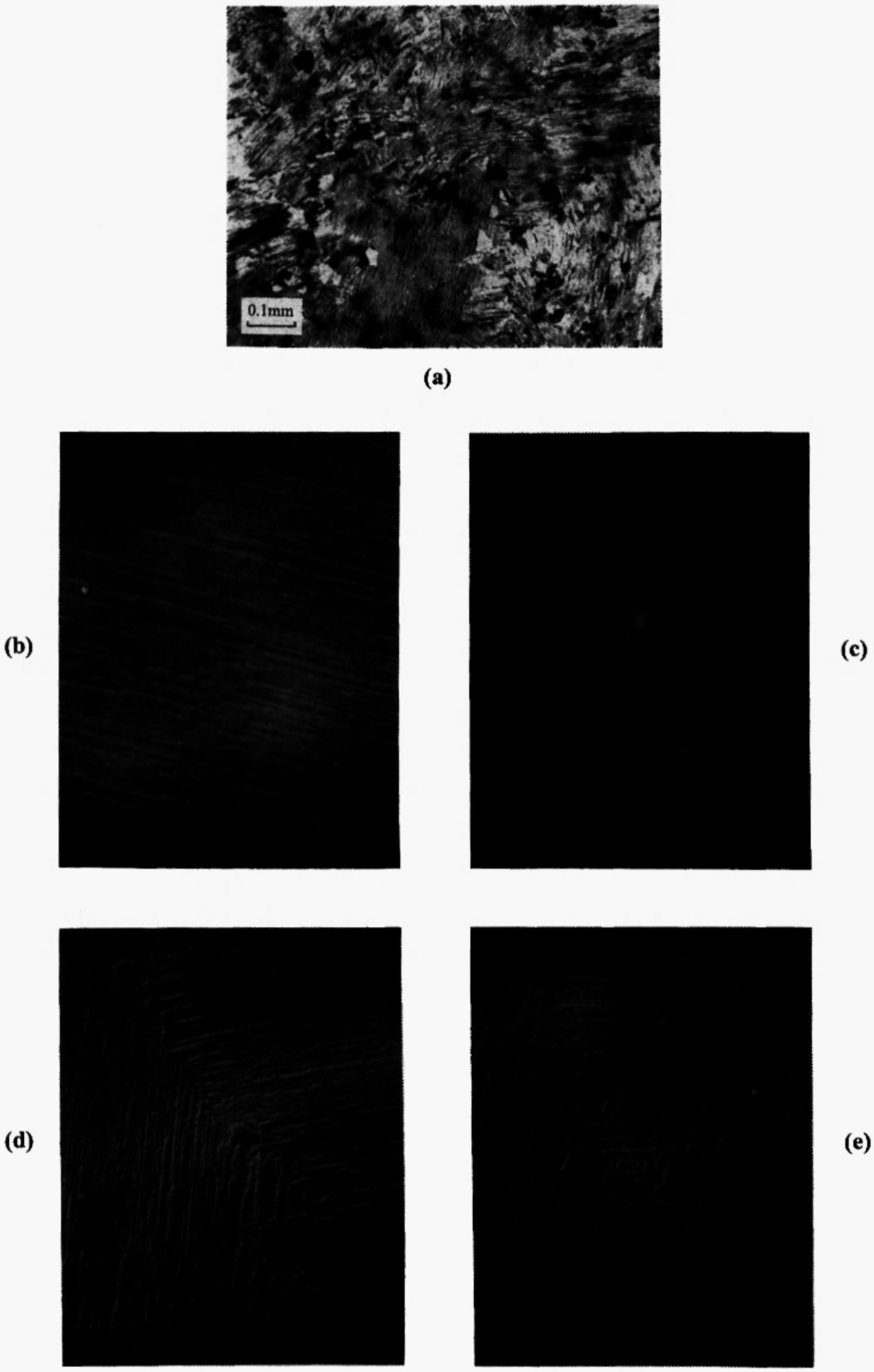


Fig. 6: Typical microstructures of 4822 (a) master cast alloy (optical micrograph) (b) lamellar structure in parallel columnar grains of 4822-2 (c) a few block-like γ (white) on grain boundaries in PD1 (backscattering image in SEM) (d) grain boundaries in 4822-6 (e) grain boundaries on 4822-2 ((b)(d)(e) secondary electronic image in SEM).

Table 2

Compositions of major structure and phase in DS 4822 (at%)

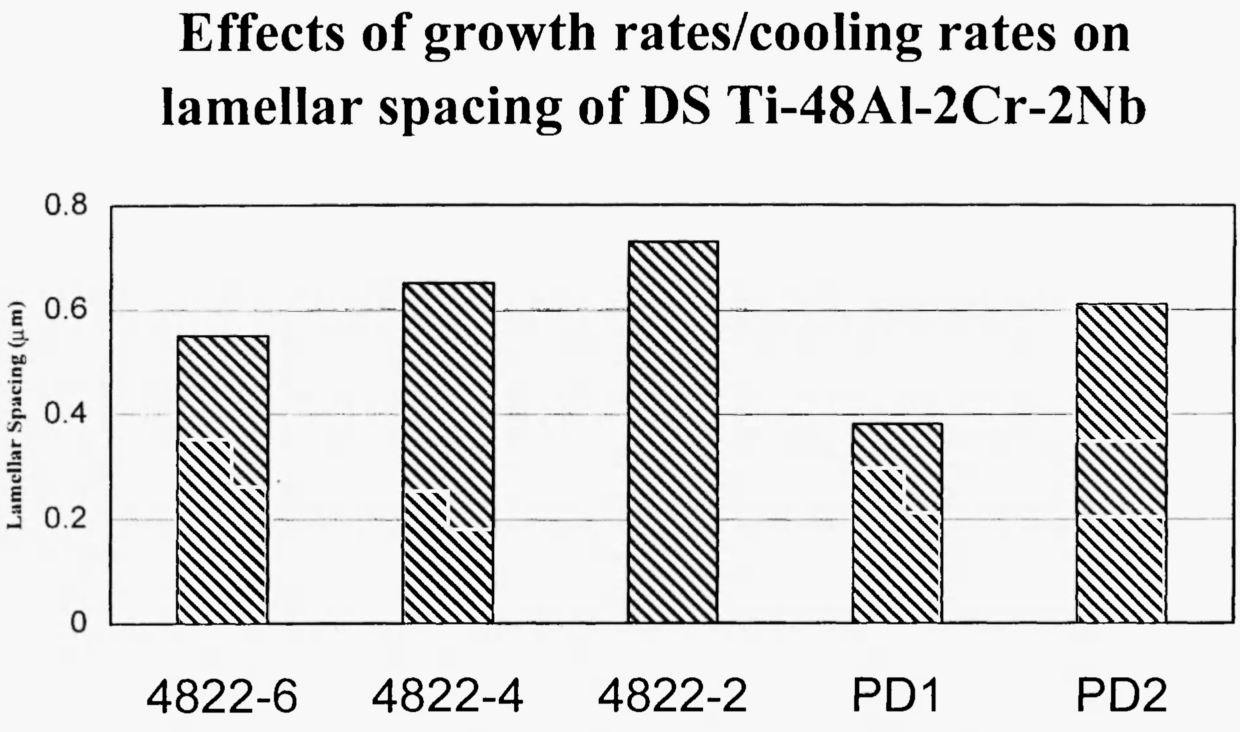
Structure or phase	Al	Ti	Cr	Nb
Average in columnar grains	46.5	49.6	1.8	2.1
Block-like γ phase on grain boundaries	51.0	44.7	1.8	2.5

plus HIP'ed Ti-47.3Al-2Nb-1.7Mn /19/, except for a smaller volume fraction of blocky γ phase in DS 4822. The phase structure also is similar to that of Ti-48Al, suggesting that the alloying addition did not have a major influence on the microstructure.

3.3 Lamellar structure

The average spacing of the lamellar structure in the PD bar is about 0.38 to 0.61μm, which is close to the value previously measured in DS Ti-48Al /20/. The lamellar spacing decreases with increasing cooling rate from PD2 to PD1 (Figure 7). Similarly, the lamellar spacing in BD bars decreases with increasing the growth rate corresponding to an increasing cooling rate.

As shown in Figure 4(b), the lamellar boundaries in all grains of three BD bars and PD2 are inclined at an angle of about 45-90 degree to the growth direction. However, an exception is PD1 produced with a rapid cooling rate during solidification. As shown in Figure 4(a), a parallel columnar grain of about 2.5 mm width, has lamellar boundaries nearly parallel to the growth direction (inclined at an angle to the growth direction <15°). In addition, lamellar structures nearly aligned to the growth direction were found in large nearly equiaxed grains immediately below the columnar grain, three radial columnar grains (in the lower left of Figure 4(a)) and a few of small equiaxed grains. A similar situation has not been reported before in DS 4822 and DS binary γ alloys such as Ti-48Al. This phenomenon will be discussed later.



(growth rate: 6mm/min for 4822-6, 4mm/min for 4822-4, 2mm/min for 4822-2; cooling rate: 2.05°C/s for PD1, 0.11°C/s for PD2)

Fig. 7: Effects of cooling rate/growth rate on lamellar spacing in DS 4822 made by PD and BD processing.

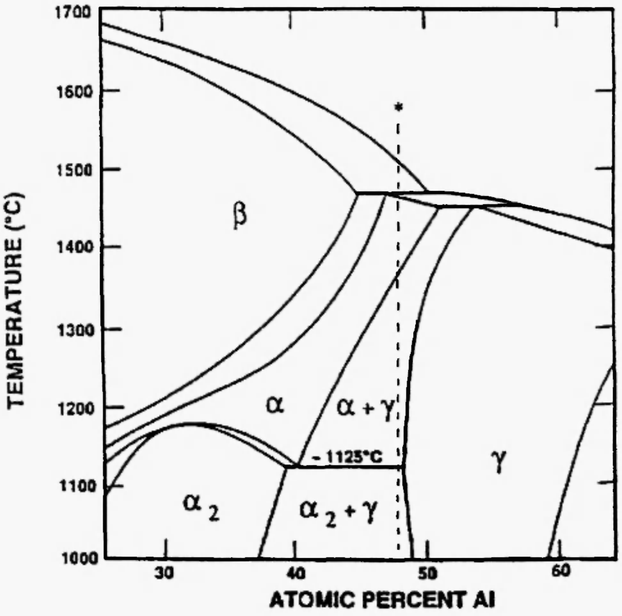


Fig. 8: A high temperature part of pseudo binary Ti-Al phase diagram /19/. The dotted line indicates 4822.

4. DISCUSSION

4.1 Solidification sequence

A high temperature portion of the binary Ti-Al phase diagram /19/ is shown in Figure 8. Since no quaternary phase diagrams are available for 4822, the binary diagram of Figure 8 is used as an aid for the following discussion. The aluminum/titanium content between β and α can be quite close in ternary and quaternary TiAl alloys, so the symmetry of the prior solidified dendrites often provides additional useful information for identification of the primary phase to EDS testing /13,17/. Although the dendrite morphology within the columnar grains in DS 4822 (Figure 4) is not very clear, the dendrites at the top of the DS bars have a cubic symmetry. Combined with the composition of the dendrite (Table 2), β is likely the primary phase during solidification in DS 4822. Since β contains less aluminum than the average content of the alloy the remaining liquid becomes aluminum rich leading to the formation of the interdendrite blocky gamma at the end of the solidification.

No indications of a peritectic reaction can be seen (Figures 3-4), suggesting that the peritectic reaction between β and liquid was completely suppressed in all conditions. For example, 4822-4 consists of almost one single parallel columnar crystal. The microstructures of the DS 4822 in all conditions from PD1 with quick cooling to 4822-2 with slow cooling are very close to each other. To further clarify this point, the five parallel columnar grains close to each other in 4822-6 were selected to determine the average composition. As shown in Table 3, the compositions of these columnar grains do not show any significant differences, which suggests that all of them solidified as the β phase. However, it was found that a similar peritectic reaction between the α and melt in DS Ti-52Al-2W-0.5Si with a growth rate of 3 mm/min was only partially suppressed /16/. It would be valuable to study further the effects of alloying additions on the high temperature part of the binary Ti-Al phase diagram, particularly that related to the peritectic reaction.

Table3

The area average compositions of parallel columnar grains closed each other in 4822-6 (at%)

Grains	Al	Ti	Cr	Nb
1	46.2	49.9	1.8	2.1
2	46.3	49.8	1.7	2.2
3	46.8	49.5	1.8	1.9
4	46.6	49.7	1.7	2.0
5	46.7	49.6	1.8	1.9

The limited size and quantity of the gamma phase in all conditions (Figure 6) implies that segregation in DS 4822 is minimal. However, the partition coefficient in the melt of 4822 during the solidification is quite large so that the interdendritic phase is γ instead of α with an aluminum content close to the primary solidified β. This deduction is consistent with the previous results /17/. During cooling after solidification the β transforms into α before an α+γ lamellar structure is finally formed, but the interdendrite γ remained.

In related work on 4822 /11,17/ and binary TiAl with a similar aluminum content /7,20,21/, β was also concluded to be the primary phase. As already mentioned, α has been proposed to be primary phase in

DS Ti-48Al /8,14/ and this result was used to explain the orientation of the lamellar structure. However, this conclusion was subsequently modified /10/, and based on approximate calculations of the solidification kinetics, it was suggested that a fast growth rate favours primary α, with a transition from primary β to α occurring at about 300 mm/h (5 mm/min) in Ti-48.5Al. However, in the 4822 of the current investigation, β is the primary phase at growth rates ranging from 2 mm/min to 6 mm/min. It can be speculated that the alloying additions to 4822 influence the primary phase selection. Further research is necessary to verify the calculated results.

4.2 Effects of processing parameters on microstructures

For DS bars made by PD processing a more rapid cooling rate refines the grain size and reduces the lamellar interface spacing (Figure 7), but reduces the portion of the parallel columnar grains (Figure 5). Similarly, for bars produced by BD processing, a more rapid withdrawal or growth rate causes the same trends, in terms of the portion of parallel columnar grains and lamellar interface spacing. Both reduced lamellar interface spacing and grain size can improve the mechanical properties of the TiAl in the fully lamellar condition /22/. Hence, to achieve a DS bar with useful mechanical properties by either the PD or BD techniques will require balancing the portion of parallel columnar grains, the grain size and lamellar interface spacing.

4.3 Lamellar orientation

As mentioned in the Introduction, Yamaguchi *et al.* propose /8,12/ that the primary phase during the solidification, in addition to the growth rate, controls the orientation of the subsequent lamellar structure. Accordingly, when β is the primary phase, the preferred growth direction is <100>, so the $(110)_\beta$ forms with an angle of 0° or 45° to the growth direction. The α formed during subsequent solid state transformation would have an orientation with β of $(110)_\beta // (0001)_\alpha$. The γ lamellae then precipitates from α according to the orientation relationship of $(0001)_\alpha // (111)_\gamma$. This sequence produces lamellar boundaries inclined at an angle of 0° or 45° to the growth direction. In addition, they concluded /12/ that a more rapid growth rate does not generally favour the alignment of the lamellar structure possibly since the β region is shifted to the titanium-rich side in binary Ti-Al phase diagram.

According to the current experimental results and the foregoing analysis, β is the solidifying phase, except for a few γ on grain boundaries in all of the tested conditions. However, in one grain within the parallel columnar region of PD1 lamellae aligned in the growth direction, Figure 4(a). Furthermore, lamellae aligned in the growth direction are present in several other grains. Considering that aligned lamellar structures are present in several grains, this result is unlikely due to a random phenomenon, but such aligned lamellar structures have not been previously reported in DS 4822 or binary TiAl alloys. Hence, the results cannot be explained on the basis of Yamaguchi *et al.*'s suggestions. Even if α is the primary phase, as suggested in /8,14/, the question about the mechanism controlling the lamellar orientation remains, since in such a case no lamellae aligned in the growth direction should exist. The fully aligned lamellae can neither be accounted for by interference of α that may form subsequent to the primary β formation /9/, since as demonstrated above, no evidence of the formation of peritectic α was observed in the current investigation.

Even according to the suggestions of Yamaguchi *et al.*, when β is the primary phase, a probability of 1/3 of the grains would have lamellar boundaries parallel to the growth direction, with a probability of remaining two thirds of the grains having lamellae oriented at 45° to the growth direction. These proportions are due to the $\{110\}_\beta // (0001)_\alpha$ orientation relationship between β and α. For any one of the three <100> growth directions, only 1/3 of the $\{110\}_\beta$ would be parallel to the growth direction of the β, assuming columnar grains grow equally likely on each {110} plane. However, the microstructures of the current results do not respect these lamellae orientation proportions.

Consequently, based on the results it appears that whether the primary phase is β or α is not the only factor that determines the subsequent lamellar alignment. In fact, controlling the alignment of lamellar boundaries in TiAl alloys appears to be a complicated issue. It is clear, however, that the lamellar orientation is influenced both by the DS process parameters and the subsequent solid state transformations, neither of which occurs under equilibrium conditions. Further detailed research is required to elucidate this important factor.

4.4 Comparison between PD and BD

Based on Figures 3 and 5 it is apparent that using BD processing results in a DS bar with a larger portion of parallel columnar grains. This is because the BD process provides a more homogeneous temperature field along the growth direction, whereas the temperature field is less uniform during PD processing. Suitable control of the PD process to overcome this deficiency would be difficult. However the PD process is mechanically much simpler, since a withdrawal system is not required. Nevertheless, the BD technique is frequently used in industrial production, with the PD technique more suitable for research purposes when the inhomogeneous temperature field can in fact be advantageous.

5. CONCLUSION

(1) DS 4822 bars were made by PD and BD processing respectively. The DS bars made in all conditions in the current work consist of columnar grains parallel to the growth direction, radiant columnar grains and equiaxed grains. A narrow layer of equiaxed grains exists between the radiant columnar grains and parallel columnar grains.

(2) Reducing the cooling rate in PD or decreasing the growth rate in BD can increase the portion of the parallel columnar grains and increases the lamellar interface spacing in the DS structure. However, BD can provide a homogeneous temperature field along the growth direction making this technique more appropriate for producing a TiAl bar with a larger effective length of columnar grains parallel to the growth direction.

(3) β is the primary phase in DS 4822 in all conditions. A few blocky gamma grains are present at grain boundaries. The β transformed to alpha and finally to a $\gamma+\alpha_2$ lamellar structure during the cooling after solidification but the interdendrite gamma remains.

(4) The lamellar boundaries of DS 4822 are inclined at an angle of 45 to 90° to the growth direction in almost all conditions. However, the nearly parallel lamellar structure to the growth direction is present in several grains, including a parallel columnar grain, radial columnar grains and equiaxed grains, in a DS 4822 bar made by PD, the top surface of which solidified at a cooling rate of 2.05°C/s. The phenomenon can not be satisfactorily explained at this time. Further study of the factors controlling the orientation of the lamellar structure is needed.

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