



Induction melting of γ -TiAl in CaO crucibles

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ABSTRACT

In this work a Ti–48Al alloy was induction melted in a CaO crucible using different superheating temperatures. In the first stage, samples were allowed to cool to room temperature inside the crucibles, in order to simulate a low cooling rate, and in the second stage samples were centrifugally poured into a steel mould, in order to study the effect of the melting operation on the alloy contamination with oxygen. The effect of superheating temperature on the metal–crucible interaction, alloy chemical composition, microstructure and microhardness is evaluated. The CaO crucible was found to be slightly dissolved by the molten alloy and the extent of that dissolution depends on the superheating temperature. A relationship was found between oxygen concentration and microhardness profiles of the $\alpha_2 + \gamma$ microconstituent, from the surface to the inside of samples, which depends on the superheating temperature and cooling rate.

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1. Introduction

Gamma titanium aluminides are the most serious candidates to replace traditional steels and Fe or Ni-based superalloys for high temperature applications, mainly in those areas where the combination of high tensile properties, creep strength and corrosion resistance, associated with superior strength-to-weight ratio are important, and cost is not a crucial factor, like aeronautical, aerospace and military equipments [1–3].

A major effort has been made over the last years to introduce titanium aluminides into the market-place as engineering components, however only a few niche markets in the automotive industry have been established [4,5]. Regarding aeronautical applications, although engine testing has been carried out by Rolls-Royce, P & W and GE no component made of a TiAl-based alloy is in use in any current aero engine [6,7].

At the present stage, the main factors limiting the manufacture of mass market TiAl-based components are the intrinsic characteristics of TiAl alloys – microstructure and chemical heterogeneity, brittleness, low room temperature ductility and poor hot workability [3], and the high production costs. Nevertheless, significant improvements are being made to improve the mechanical performance of these alloys, with the development of new ternary and quaternary TiAl-based alloys, namely those from the systems Ti–Al–Nb–Cr and Ti–Al–W–Si [6,8]. To decrease production costs it is

imperative to improve the actual processing techniques of TiAl. Present production routes of TiAl components are limited to forging and rolling, powder metallurgy and casting. The first two are very prone to chemical and microstructure heterogeneity and powder metallurgy is highly expensive and limited to simple geometries. Casting, using induction skull melting and the investment casting moulding process is the most versatile technology to manufacture intermetallic components. However, induction skull melting has a high impact in the castings final cost and in the high level of castings rejections, mainly due to the difficulty to achieve suitable superheating. To overcome this problem, high mould preheat temperatures are frequently used, thus increasing the probability of a metal–mould reaction occurring, and consequent metal contamination and development of solidification defects.

A possible way to reduce production costs might be the use of the ceramic crucible induction melting process, using suitable and low cost ceramic materials for crucible production, combined with the use of traditional melting stocks, avoiding expensive master alloys [9–11]. However this would only be possible in those cases where high melting point constituents like Nb or W, for example, are not used in the melting stock or their concentration in the cast alloy is low enough to avoid prolonged heating at high temperature, and consequent excessive oxygen pickup from the crucible wall.

Until now, no material was found to be absolutely inert against titanium alloys and during melting some interaction between the alloy and the crucible materials always occurs, leading to metal contamination [10–12]. Taking into account the free Gibbs energy of formation, CaO presents the most negative value among all

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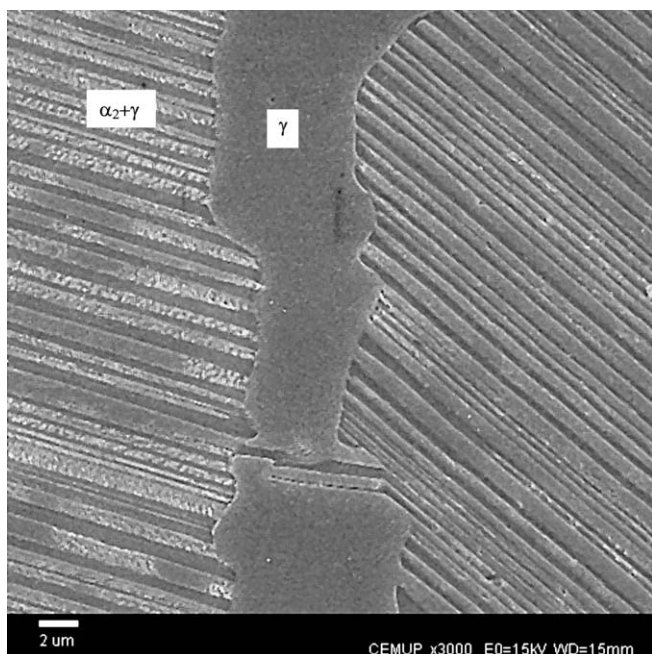


Fig. 1. Microstructure of a Ti–48Al sample, induction melted in a CaO crucible.

metallic oxides if Y_2O_3 is excluded, suggesting that it could be a good option to produce melting crucibles for titanium alloys.

2. Experimental

A Ti–48Al alloy was selected for melting, prepared from commercially pure titanium and aluminium. Melting stocks weighing 1.3×10^{-1} kg were melted in a ceramic crucible induction melting furnace equipped with a commercially available 0.15 l CaO crucible, under a dry argon atmosphere at 10^{-1} bar. Before the heating cycle, the chamber was evacuated up to 10^{-3} bar and backfilled with argon 5 times, in order to reduce the oxygen content to a minimum level. The superheating temperatures were 1550 °C and 1600 °C, measured with a type B (Pt–PtRd) thermocouple with a Mo–Al₂O₃ protection sheath, corresponding to approximately 50 and 100 °C superheating (assuming that the *liquidus* temperature of Ti–48Al is 1500 °C [13]). After reaching the superheating temperature the alloy was held at that temperature for 60 s in order to get chemical homogenization. In the first series of tests, samples were allowed to solidify and cool to room temperature inside the crucible, in order to simulate the worst practical situation

concerning the occurrence of a metal–crucible interaction. In the second series of tests, 20 mm × 80 mm test samples were centrifugally poured at 400 rpm into a cylindrical steel mould pre-heated to 250 °C, in order to isolate the effect of oxygen pickup exclusively from the crucible, during the melting operation.

During cooling, temperature measurement was performed using a wireless receiver/transmitter–receiver data acquisition system compatible with the centrifugal casting process, developed at the University of Minho.

Samples for characterization were collected from the middle of the cast cylinders by sectioning them at 50% of their height. Phase identification and chemical composition were performed by quantitative EDS analysis with standards of pure Ti, Al and Ca using a JEOL JSM 35C scanning electron microscope. Overall oxygen content was measured by the IGF technique (Inert Gas Fusion), and SIMS (Secondary Ion Mass Spectrometry) was used to determine the diffusion profile of oxygen from the surface to the inside of the samples. Microhardness was evaluated on a Shimadzu hardness tester using a 50 g load for 15 s.

3. Results

3.1. Microstructure

Those samples allowed to cool inside the crucible revealed a microstructure containing a dendritic lamellar constituent with two phases – $\alpha_2 + \gamma$ – and an interdendritic γ phase (Fig. 1). The volume fraction of the lamellar constituent was higher near the surface in every sample (fully lamellar at distances from the surface lower than 25 μ m) and decreased towards the inside of them where average values between 70.5 and 75% were found, following the decrease in the cooling rate. This is the typical TiAl bi-phase structure ($\alpha_2 + \gamma$) in which the amount of aluminium is slightly below 49%, and the amount of γ constituent increases with increasing Al content [3,12,14]. In those samples poured into steel moulds the microstructure was fully lamellar at distances from the surface of less than 200 μ m (Fig. 2) due to the high cooling rate achieved in the metallic mould (Fig. 3). At higher distances from the surface small grains of γ phase appear at the boundaries of the lamellar grains, in a volume fraction between 5 and 10%.

3.2. Chemical composition

Table 1 presents the chemical composition of cast samples at different distances from the surface, for each pair of temperature/cooling media. After melting, a very small amount of aluminium powder usually appeared at the furnace window (cold zone), suggesting that some Al vaporization occurred, but there was no

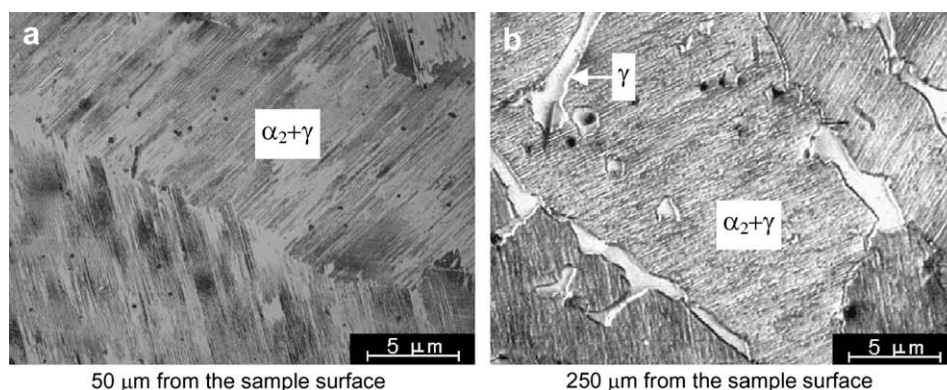


Fig. 2. Microstructure of Ti–48Al samples cast in steel moulds, at different distances from surface.

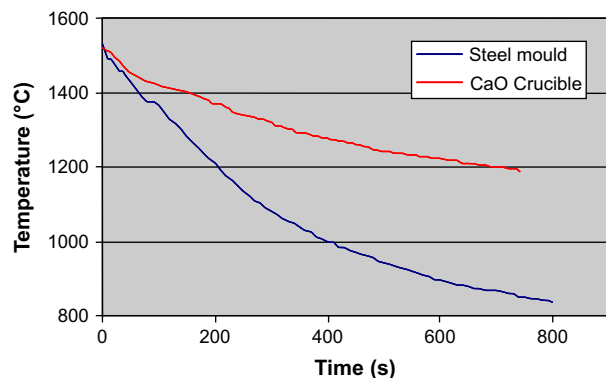


Fig. 3. Cooling curves of samples allowed to solidify inside CaO crucibles and steel moulds.

perceptible aluminium loss in the alloy composition, which fall within the range it was expected. Although a stronger exothermic reaction between Al and Ti could be expected, it might have been avoided due to the fact that Al never reached the liquid stage, but kept on a mushy state since the early moments of the melting operation, as it immediately started dissolving Ti.

In every sample the lamellar dendritic constituent was richer in titanium and the interdendritic γ phase had higher aluminium content, which agrees with the available references [3,8,15]. Every sample revealed a slight contamination with Ca that dissolved in both microconstituents, although a little more in the interdendritic γ phase.

In samples allowed to solidify inside the crucible (samples S1 and S2), Ca concentration was higher at the surface assuming

constant values for distances from the surface higher than 25 μm for $T = 1550^\circ\text{C}$ and 50 μm for $T = 1600^\circ\text{C}$. In those samples poured in steel moulds, no Ca concentration gradients were found, and a constant value around 0.10 wt% was found in the sample poured at 1600 $^\circ\text{C}$, and values below the equipment detection limit in the sample poured at 1550 $^\circ\text{C}$. Besides Ca, the cast alloys were found to be contaminated with oxygen, whose content and concentration gradients were found to depend on the superheating temperature and the cooling media (Table 1 and Fig. 4).

3.3. Microhardness

Microhardness values of the $\alpha_2 + \gamma$ microconstituent were affected both by the cooling media and the superheating temperature, but in the γ phase they were almost constant and similar in every sample (289–293 HV). In those samples allowed to solidify inside the crucibles, microhardness of $\alpha_2 + \gamma$ decreased from the surface to the inside of samples for both superheating temperatures (Fig. 5), until a certain distance from surface, which corresponds to the *alpha-case extension*. For higher distances from the surface, microhardness assumes almost constant values, of the same magnitude of those in bulk. For sample S1, poured at 1550 $^\circ\text{C}$, that value is around 45 μm , and for sample S2, poured at 1600 $^\circ\text{C}$, it is around 70 μm . This gradient did not occur in samples poured in steel moulds, which revealed almost constant microhardness values all over their mass. The cooling media and the superheating temperature also affected the $\alpha_2 + \gamma$ microhardness in the bulk of samples: average values of 303 and 316 HV for 1550 $^\circ\text{C}$ and 1600 $^\circ\text{C}$ respectively in those samples allowed to cool inside the crucible (S1 and S2), and 301 and 306 HV, for the same temperatures, in those samples poured into steel moulds (S3 and S4) (see Table 2).

Table 1

Chemical composition of cast Ti–48Al samples poured at different superheating temperatures and cooled in different cooling media

			Depth from surface (μm)									
Sample	Const.	Element	25	50	100	150	200	250	300	500	1000	Bulk
S1 1550 °C Crucible	α ₂ + γ	Ti	51.6	50.2	51.7	51.9	52.6	52.0	51.9	52.0	52.5	52.7
		Al	48.3	49.8	48.3	48.1	47.4	48.0	48.1	48.0	47.5	47.3
		Ca	0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
	γ	Ti	45.0	44.9	46.5	46.0	45.8	45.9	45.9	46.5	45.8	45.6
		Al	54.7	55.0	53.5	54.0	54.2	54.1	54.1	53.5	54.2	54.4
		Ca	0.14	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
Sample average composition:			Ti – 51.7			Al – 48.3		Ca < 0.1		Oxygen content – 0.26 wt%		
S2 1600 °C Crucible	α ₂ + γ	Ti	52.1	51.6	51.7	51.5	52.0	51.8	51.8	52.3	52.2	52.5
		Al	47.8	48.4	48.3	48.5	48.0	48.2	48.2	47.7	47.8	47.5
		Ca	0.12	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
	γ	Ti	46.2	45.4	45.8	45.8	45.9	46.3	45.5	46.1	46	45.9
		Al	53.6	54.5	54.1	54.2	54.0	53.6	54.5	53.9	53.9	54.0
		Ca	0.16	0.11	0.10	<0.10	0.11	0.11	<0.10	<0.10	0.11	0.10
Sample average composition:			Ti – 52.1			Al – 47.8		Ca – 0.11		Oxygen content – 0.29 wt%		
S3 1550 °C Steel mould	α ₂ + γ	Ti	51.4	51.2	52.1	52.2	51.9	51.7	52.3	52.3	52.1	52.2
		Al	48.6	48.8	47.9	47.8	48.1	48.3	47.7	47.7	47.9	47.8
		Ca	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
	γ	Ti	45.5	44.9	45.2	46.1	46.0	45.5	45.9	46.3	45.5	45.9
		Al	54.4	55.0	54.8	53.9	54.0	54.5	54.1	53.7	54.5	54.1
		Ca	n.a.	n.a.	n.a.	n.a.	<0.10	<0.10	<0.10	<0.10	<0.10	0.10
Sample average composition:			Ti – 51.9			Al – 48.1		Ca < 0.1		Oxygen content – 0.24 wt%		
S4 1600 °C Steel mould	α ₂ + γ	Ti	51.2	52.2	52.0	52.8	52.9	52.7	51.2	52.8	52.5	53.0
		Al	48.8	47.7	48.0	47.2	47.1	47.3	48.8	47.2	47.5	47.0
		Ca	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
	γ	Ti	45.0	44.9	45.2	45	45.6	46.7	46.0	46.4	45.5	46.4
		Al	55.0	55.0	54.8	55.0	54.3	53.2	53.9	53.5	54.5	53.5
		Ca	n.a.	n.a.	n.a.	n.a.	0.10	0.10	0.10	0.11	0.10	0.10
Sample average composition:			Ti – 52.3			Al – 47.6		Ca – 0.10		Oxygen content – 0.26 wt%		

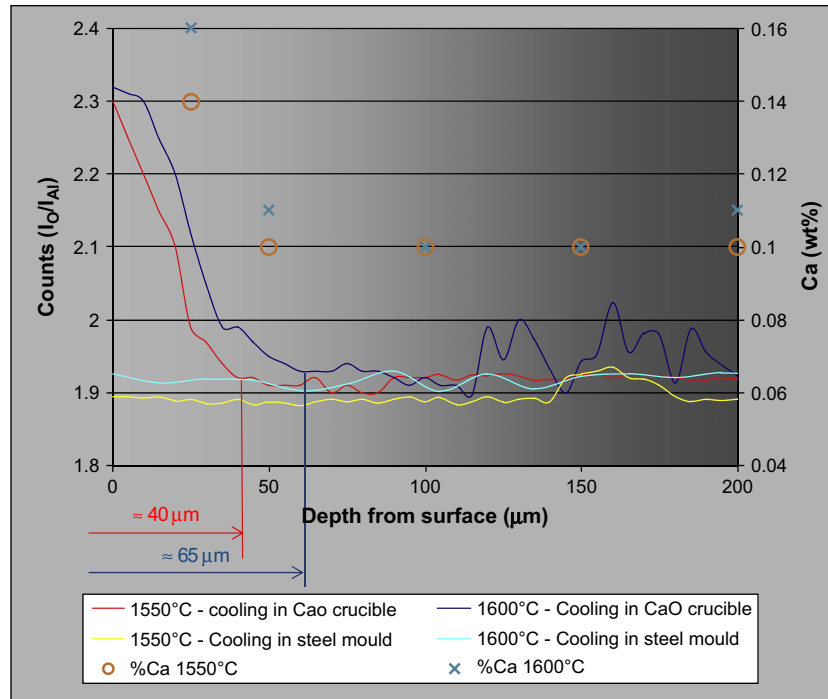


Fig. 4. Oxygen and Ca concentration profiles from the samples surface to the inside, for different superheating temperatures and cooling media. The diffusion depth of oxygen seems to be around 65 μm for $T = 1600^\circ\text{C}$ and around 40 μm for $T = 1550^\circ\text{C}$, in samples cooled inside CaO crucibles. Ca diffusion depth seems to be of the same magnitude. The fluctuation of the oxygen content of sample S2 (1600 $^\circ\text{C}$, cooling in CaO crucible) between 120 and 200 μm from the sample surface was due to difficulties to stabilize the energy of the primary Ar^+ ion beam source during the corresponding lap time.

3.4. Metal-crucible/mould interface

Fig. 6 shows the samples microstructure at the metal-crucible wall and metal-steel mould interfaces for 1550 $^\circ\text{C}$ pouring temperature. Those samples allowed to solidify inside the crucible revealed a continuous film of calcium oxide at the surface with average thickness less than 5 μm . This layer could be easily removed with a finger, revealing that no metal penetration occurred at the crucible wall. The morphology and thickness of that layer were not affected by the superheating temperature.

4. Discussion

The average chemical composition of every sample was very close to the desired Ti–48Al standard alloy, as presented in Table 1, suggesting that vacuum induction melting could provide good homogenization of the melted feed stocks. However, the presence of Ca and O in solution reveals that some sort of interaction occurred between the CaO crucible and the cast alloy. According to its free energy of formation, CaO is thermodynamically much more stable than titanium and aluminium oxides, so no reaction of the crucible with the cast alloy should be expected. Thus, the most probable cause of alloy contamination is the dissolution of the CaO crucible by the metal, which confirms the results of other researchers [9].

The dissolution of the CaO crucible can be confirmed by the oxygen content of the alloys. According to the composition of the melting stock, the maximum oxygen content of the alloys should be 0.16 wt%. Nevertheless, the oxygen content of the cast samples varies from 0.26 wt% to 0.29 wt% in those samples cooled inside the CaO crucibles (0.10–0.13 wt% enrichment, depending on the superheating temperature), and between 0.24 wt% and 0.26 wt% in samples poured into steel moulds (0.08–0.10 wt% enrichment for 1550 and 1600 $^\circ\text{C}$ superheating temperature, respectively)

(see Table 2). Taking into account that the entire processing (melting + pouring/cooling) was performed under a dry argon atmosphere, which excludes the possibility of oxygen pickup, the only possible source of oxygen was the crucible material. The values presented above are slightly higher (≈ 0.03 wt%) than those referred by Barbosa et al. [11] and Kuang et al. [16] for Ti–48Al obtained in Y_2O_3 crucibles using the same superheating

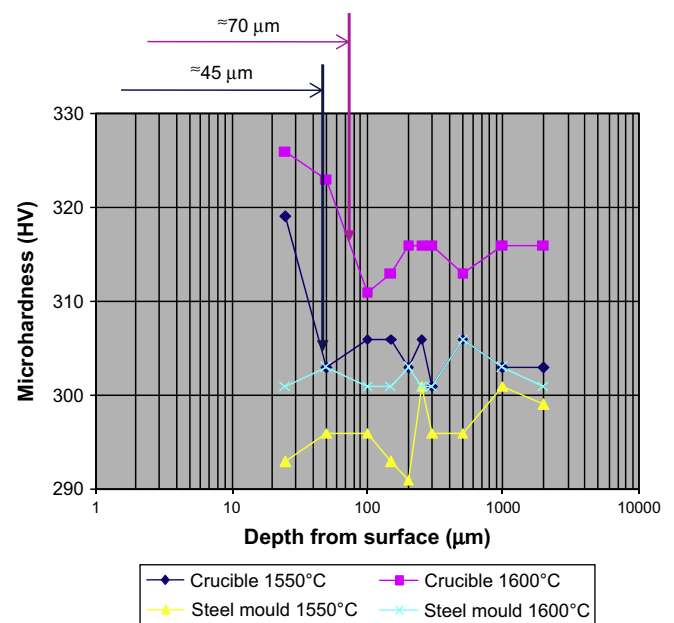


Fig. 5. Microhardness variation at the $\alpha_2 + \gamma$ microconstituent in Ti–48Al samples allowed to solidify inside the crucibles and poured into steel dies, for different superheating temperatures.

Table 2

Influence of superheating and cooling media in some characteristics of Ti–48Al samples induction melted in CaO crucibles

Temp. (°C)	Cooling media	Alpha-case extension (μm)	Inside the sample						
			Composition (at%)			Dissolved oxygen (wt%)		Microhardness (HV)	
			Ti	Al	Ca	Final	Pickup	$\alpha_2 + \gamma$	γ
1550	S1 crucible	≈ 40	51.7	48.3	<0.1	0.26	0.10	303	291
	S3 steel mould	–	51.9	48.1	<0.1	0.24	0.08	301	291
1600	S2 crucible	≈ 70	52.1	47.8	0.11	0.29	0.13	316	289
	S4 steel mould	–	52.3	47.6	0.10	0.26	0.10	306	293

temperatures, and are similar to those obtained by Sato et al. (0.2–0.4 wt%) [17] and Liu et al. (0.082–0.29 wt%) [9] for CaO crucible induction melted unalloyed titanium and Ti–45Al–8Nb respectively.

According to Fig. 4 and Table 1, the oxygen and calcium contents of samples allowed to solidify inside the crucibles decrease from the surface to the inside of the samples following curves with similar profiles, for the same superheating temperatures. This behaviour suggests that the extent of the CaO crucible dissolution is a function of that parameter. When the metal reached the liquid state and its fluidity became high enough, it could easily penetrate the interstices between the CaO grains (notice that CaO crucibles used on this work had 20% open porosity), thus increasing the erosion effect and the grain dissolution rate. Although the stirring effect of induction heating was not very high due to the high frequency of the melting furnace (1.3 MHz), it helped in the distribution of those CaO particles detached from the crucible in the liquid metal, which dissolved them, and the dissolution products (Ca and O) went into solution with the alloy. Thus, a uniform composition was easily reached in the molten alloy, the value of which depended on the dissolution extent and in turn on the melt temperature. That is why the average oxygen enrichment is higher in those samples superheated to 1600 °C (samples S2 and S4). However, when the heating supply stopped and cooling started, the metal fluidity decreased very quickly and the CaO dissolution rate became much slower, as well as the diffusion mechanisms, leading to the Ca and O concentration gradients found in samples S1 and S2. Nevertheless, until the metal solidifies, diffusion of Ca and O is still possible, slightly increasing the Ca and O content inside the samples. However, due to different crucible temperatures, the cooling conditions were not the same for every sample. As a consequence, they led to different dissolution kinetics and diffusion rates, thus to different average contents and concentration gradients of Ca and O from the surface to the inside of the cast samples.

Results obtained by allowing the alloys to solidify inside the melting crucibles can be a useful indication for the use of CaO to produce ceramic moulds for TiAl. In fact, it can be assumed that if

CaO moulds are used for TiAl castings, the maximum alloy contamination, “alpha-case” extension and microhardness will be those obtained by allowing the alloy to solidify inside the crucible (samples S1 and S2) as in practice the cooling rate will be much higher due to the lower mould temperature (for samples S1 and S2, the crucible acted as a mould, thus the “mould” temperature was in fact 1550 and 1600 °C respectively). Nevertheless, due to the hygroscopic nature of CaO, its use as mould material will always have to be done with great care to avoid moisture pickup, otherwise mould destruction and unacceptable oxygen pickup by the cast alloy will easily occur. Although some researchers claim to have successfully produced automotive components using CaO moulds, it is believed that if a suitable handling technique of this material will not be developed, the use of CaO moulds will always be limited to non-critical applications.

The oxygen content of the cast alloys is the main factor responsible for microhardness variation as it dissolves interstitially in the matrix. For such reason, the microhardness of the $\alpha_2 + \gamma$ constituent and oxygen concentration profiles of samples allowed to solidify inside the CaO crucibles are quite similar, decreasing from the sample surface inwards, reaching constant values after similar distances from the sample surface (see Figs. 4 and 5). In the γ phase the microhardness is quite constant for every cast sample because oxygen dissolves preferentially in the lamellar constituent being almost constant in the γ phase, where its solubility limit is around 230 ppm [18]. As a consequence, if no other residual elements are present in solution, the microhardness variation profile in $\alpha_2 + \gamma$ represents, in fact, the oxygen variation profile in the cast alloy. Due to such a strict relationship, the microhardness depends significantly on the superheating temperature and cooling rate, because as those parameters increase and decrease respectively, the more extensive is the crucible material dissolution, with a consequent increase in the alloy oxygen content. Although the microhardness increases at the surface, the maximum value – 326 HV – is significantly lower than those reported by other researchers for as cast Ti–48Al [10,14] and agree with the suggested microhardness values for this kind of alloy [19].

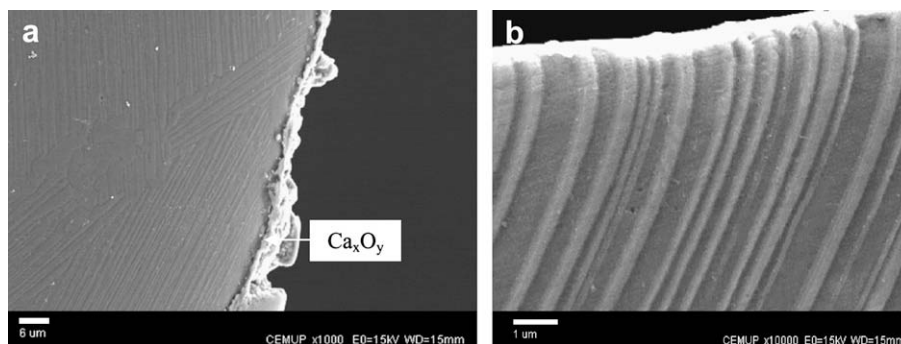


Fig. 6. Microstructure of the surface of Ti–48Al samples a) allowed to cool to room temperature inside a CaO crucible and b) poured into a steel mould, using 1550 °C superheating temperature.

5. Conclusions

- For the processing conditions used in this work, it is possible to obtain cast Ti–48Al by melting commercially pure titanium and aluminium, using 1550 °C superheating temperature for 60 s;
- For superheating temperatures of 1550 and 1600 °C, melting in CaO crucibles led to oxygen enrichment of a Ti–48Al alloy between 0.08 and 0.10 wt%, making it difficult to produce components for critical applications;
- Besides superheating, the cooling rate is an important factor in the interaction between CaO and molten TiAl, with direct influence in the concentration of residual elements and microhardness;
- If CaO moulds and superheating temperatures up to 1600 °C are used to produce TiAl castings the maximum “alpha-case” extension and surface microhardness that may be expected will be around 50 µm and 326 HV, respectively. However, due to CaO hygroscopic nature, a suitable moulding technique has to be developed to avoid moisture pickup and consequent mould destruction and excessive alloy contamination with oxygen.

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