# Effects of the manufacturing process on fracture behaviour of cast TiAl intermetallic alloys

A. Brotzu, F. Felli, D. Pilone
*Dipartimento ICMA, Sapienza Università di Roma, Roma (Italy)*
*andrea.brotzu@uniroma1.it, ferdinando.felli@uniroma1.it, daniela.pilone@uniroma1.it*

**ABSTRACT.** The $\gamma$ -TiAl based intermetallic alloys are interesting candidate materials for high-temperature applications with the efforts being directed toward the replacement of Ni-based superalloys. TiAl-based alloys are characterised by a density (3.5-4 g/cm$^3$) which is less than half of that of Ni-based superalloys, and therefore these alloys have attracted broad attention as potential candidate for high-temperature structural applications. Specific composition/microstructure combinations should be attained with the aim of obtaining good mechanical properties while maintaining satisfactory oxidation resistance, creep resistance and high temperature strength for targeted applications.
Different casting methods have been used for producing TiAl based alloys. In our experimental work, specimens were produced by means of centrifugal casting. Tests carried out on several samples characterised by different alloy compositions highlighted that solidification shrinkage and solid metal contraction during cooling produce the development of relevant residual stresses that are sufficient to fracture the castings during cooling or to produce a delayed fracture. In this work, crack initiation and growth have been analysed in order to identify the factors causing the very high residual stresses that often produce explosive crack propagation throughout the casting.

**KEYWORDS.** Titanium aluminides; TiAl intermetallics; Fracture toughness.

## INTRODUCTION

TiAl based alloys are interesting for high-temperature applications mainly in aerospace and automotive industries. Their potential is seen in low density, high specific yield strength, high specific stiffness, good oxidation resistance at room temperature (RT), resistance against ”titanium fire”, and good creep properties up to high temperatures [1]. In fact the good specific mechanical properties of titanium aluminide alloys push the development of these materials.
Because of their ordered structure, intermetallics have high mechanical strength both at RT and at high temperature [2,3]. Despite that, TiAl-based alloys cannot be used as single phase alloys since they have a very low ductility at RT. The presence of a second $\alpha_2$ phase ($Ti_3Al$) allows control of the microstructure. As far as mechanical properties are concerned, the addition of alloying elements such as Cr, V and Mn reduces the grain size with consequent ductility improvement. Depending on alloy composition and microstructure, these alloys exhibit good workability, medium-to-good tensile properties, tensile fracture strains in the range 1-3% at RT and fracture toughness values in the range 10- 25 MPa$\sqrt{m}$ [4-7].
Various TiAl-based alloys have been developed. Adding transition metals of a high melting temperatures is generally beneficial to increase the high temperature strength of these alloys [8-10]. More recently, the so-called 2$^{nd}$ and 3$^{rd}$ generation alloys have been developed in order to improve their mechanical properties and high temperature properties

[1]. The 4th generation alloys, also called "air-hardenable", have been intensively studied as potential materials for investment casting of low pressure turbine blades [11].

The casting of γ-TiAl alloys remains a challenge to industry and often leads to some deleterious defects: (a) misrun, which arises if the melt has too low superheat, (b) surface shrinkage due to the collapse of entrained bubbles and shrinkage during HIPing, (c) hot-tearing and cracking due to mould restraint during solidification and cooling. Different casting methods such as conventional sand casting, tilt and counter-gravity casting, investment casting, low pressure casting, centrifugal casting, shell mould casting, vacuum arc remelting, electron beam melting, plasma arc melting have been used for producing TiAl based alloys [12, 13]. Extrusion and forging have been also used to produce compressor blades for engine testing, but the processing costs are very high. A further approach to component production is powder technology, which may offer a lower process cost. Powder processing is potentially important, especially for larger products where segregation limits the homogeneity of products. Recently a relatively simple and efficient powder metallurgy processing method, based on mechanical milling and spark plasma sintering of PREP (Plasma Rotating Electrode Processed) pre-alloyed powders, has been proposed to prepare full density fine-grained Ti–Al alloys with controlled microstructure [14]. The obtained fine grained Ti–Al compacts exhibited high average fracture strength, over 900 MPa, irrespective of the nature of microstructure. The fine-grained "lamellar + equiaxed" microstructure demonstrated a combination of higher fracture strength and ductility than the fine-grained γ-based equiaxed structure.

For many years our research group produced and studied many TiAl intermetallic alloys with the aim of optimising both high temperature oxidation behaviour and fracture toughness. The difficulty of doing so is that alloying elements that are beneficial for improving oxidation resistance are usually detrimental for fracture toughness. In order to achieve reliable results a high number of specimens have to be produced and tested. In our research we obtained compact tension and tensile specimens via direct centrifugal casting. During specimens' manufacturing a large number of them fractured during cooling, while others showed a delayed fracture.

Considering that a large number of fractured specimens was available, a study has been carried out with the aim of finding the factors that determine this phenomenon. A previous work [15] based on the analysis of a first set of specimens highlighted some critical factors affecting the alloy soundness. In this work structure and composition were analysed and crack paths were studied in order to verify whether the adopted precautions allow to reduce the quantity of defects. Moreover the analysis of a higher number of specimens allowed to better understand the causes determining high residual stresses that in many cases are able to produce an explosive crack propagation throughout the castings.

## EXPERIMENTAL

The alloys used in this work were produced by induction melting both under an Ar atmosphere and in vacuum from pure Ti, Al, Cr, Nb, Mo, Ni and B. The molten metal was cast directly into the rotating mould. In this work several samples fractured during cooling or showing a delayed fracture were studied and analysed. Tab. 1 shows the composition (at.%) of a representative sample of them.

In order to perform metallographic examinations on the specimen surfaces they were ground to a mirror-like surface using SiC papers up to 1200 followed by 0.3 μm alumina and then etched in Keller's reagent. Metallographic structure, crack paths and fracture surfaces were inspected by scanning electron microscope (SEM) and microanalyses were carried out by energy dispersion spectroscopy (EDS).

## RESULTS AND DISCUSSION

The composition of the alloys considered in this study is reported in Tab. 1. Specimens from "A" to "I" and those from "N" to "P" fractured either during cooling or after the extraction from the mould, while specimens "L" and "M" are the only ones that did not break up. As an example Alloy "F" showed an explosive fracture two hours after extracting it from the mould. After remelting, the casting showed again an explosive fracture after 3 days. The considered alloys are TiAl-Cr-Nb-Mo alloys with an aluminium content ranging from 38.3 to 53 at.%. Alloys "G" and "H" are the only ones containing nickel: they were reported in Tab. 1 because, despite the different composition, they showed the same behaviour as the Ni-free alloys. Alloys from "L" to "P" were cast under vacuum . For those alloys the mould was preheated at 550 °C and the castings were subjected to slow cooling in a furnace.

As far as the first nine alloys are concerned, a close observation of the preferential paths of spontaneous fractures occurred in all the specimens highlighted that these paths are very similar (Fig. 1).

| Alloy | Al | Ti | Cr | Nb | Mo | B | Ni |
|---|---|---|---|---|---|---|---|
| A | 50.3 | 39.6 | - | 7.1 | 2.5 | 0.5 | - |
| B | 47.1 | 46.5 | 2.5 | 3.1 | - | 0.8 | - |
| C | 46.2 | 45.7 | 3.1 | 4.6 | - | 0.7 | - |
| D | 49.0 | 41.1 | 2.5 | 4.9 | 2.5 | - | - |
| E | 46.7 | 43.2 | 2.4 | 4.7 | 2.3 | 0.7 | - |
| F | 43.5 | 47.7 | 2.7 | 3.6 | 1.8 | 0.7 | - |
| G | 47.7 | 42.9 | 2.7 | 0.3 | 1.0 | - | 5.4 |
| H | 53.0 | 29.6 | 1.6 | 0.5 | 1.3 | - | 14.0 |
| I | 46.0 | 43.5 | 3.5 | 3.5 | 3.5 | - | - |
| L | 43.0 | 48.0 | 2.5 | 5.2 | 1.3 | - | - |
| M | 42.5 | 45.6 | 2.8 | 5.3 | 3.8 | - | - |
| N | 46.7 | 40.3 | 4.2 | 3.5 | 5.3 | - | - |
| O | 38.3 | 47.6 | 4.5 | 3.5 | 6.1 | - | - |
| P | 39.3 | 47.5 | 4.8 | 3.3 | 5.1 | - | - |

Table 1: Chemical composition (at.%) of fourteen TiAl based alloys. Specimens from “A” to “I” were cast under argon atmosphere, while specimens from “L” to “P” were cast under vacuum.

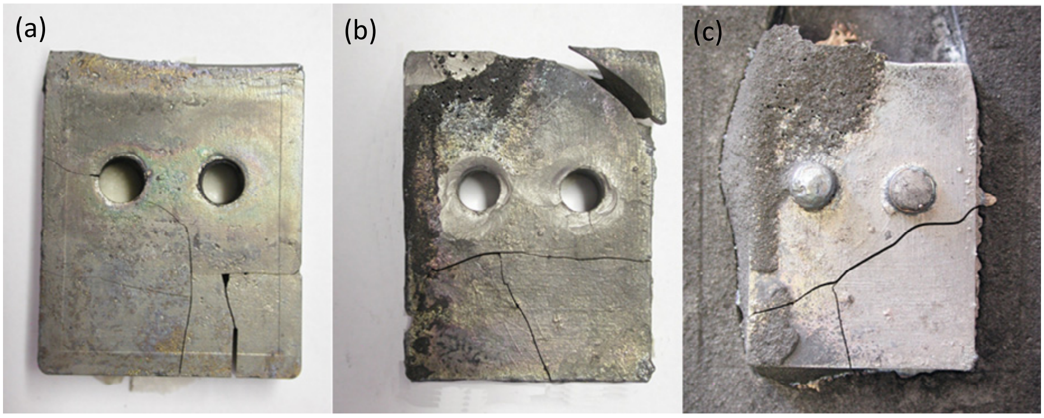


Figure 1: Macrograph showing three fractured specimens (34x40x10 mm$^3$). Specimen shown in macrograph (a) was cast under vacuum while specimens in macrographs (b) and (c) were cast under argon atmosphere.

This observation can be an important clue to identify the causes of sudden and, in some cases, explosive fractures. In particular centrifugal casting, used for manufacturing our specimens under a gas atmosphere, may generate a turbulent flow that produces gas entrapment and then air pockets or porosity formation after solidification. Other casting techniques allow a slower filling of the mould: if the liquid metal enters the mould with quiescent flow gas defects are greatly reduced.

The analysis of cracks highlights that fracture paths seem to follow the trend of the turbulent flow and fracture surfaces are characterised by the presence of gas defects. Fig. 2d shows a gas defect found on the Alloy “C” fracture surface, while Fig. 3 shows gas porosity found on the external layer of Alloy “C” specimen.

Those defects may result from entrapment of air during pouring or may be precipitated during solidification as a result of change in solubility with temperature. Defects take the form of internal blowholes, surface or subcutaneous pinholes or intergranular cavities. Gaseous elements may be absorbed by dissociation of compound gases in contact with the molten alloy. In our study the gas precipitated from the metal on cooling could be hydrogen whose source is moisture and organic compounds contaminating the charge materials. Since the most important safeguard against gas defects is a low gas content in the metal when poured, in this research the charge materials have been degreased and afterwards they have been preheated together with the crucible in a muffle to evaporate surface moisture.

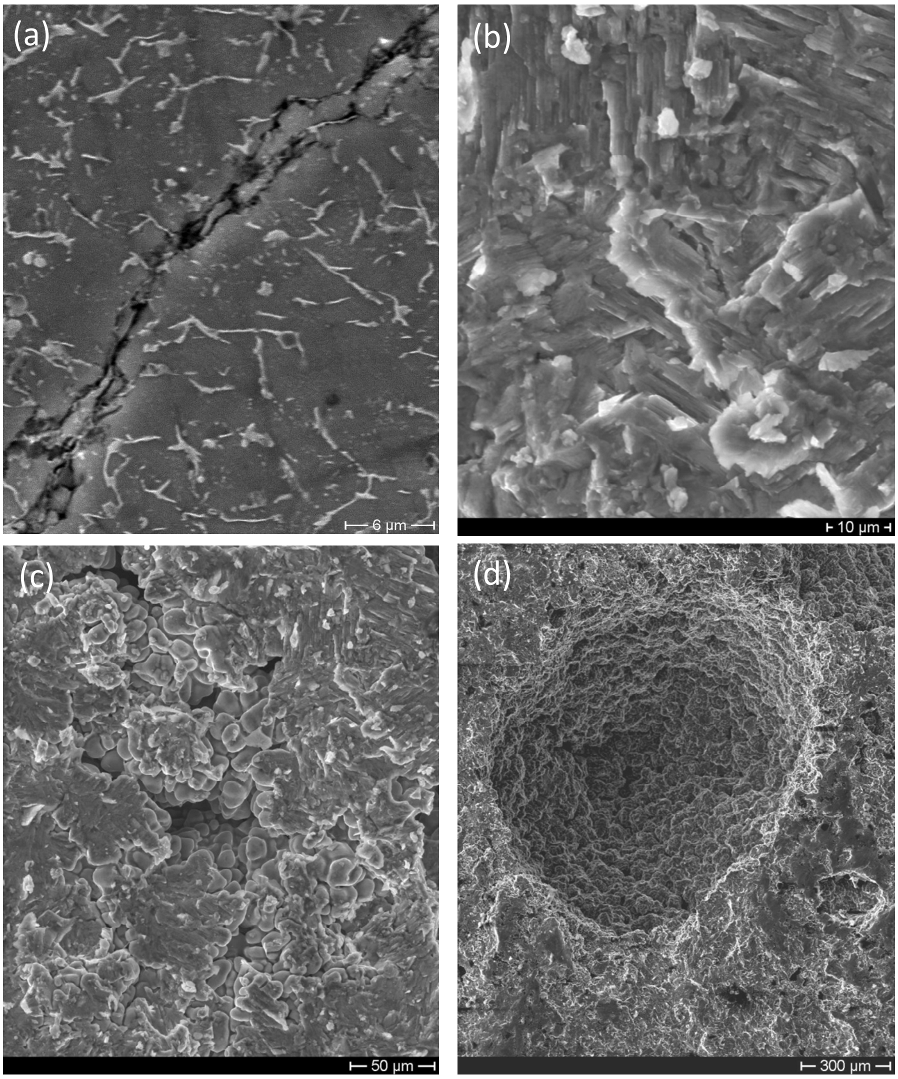


Figure 2: SEM micrografs of the Alloy "C" specimen showing one of the cracks (a). On the fracture surface the lamellar structure (b), the presence of microshrinkage cavities (c) and gas porosities (d) can be clearly observed.

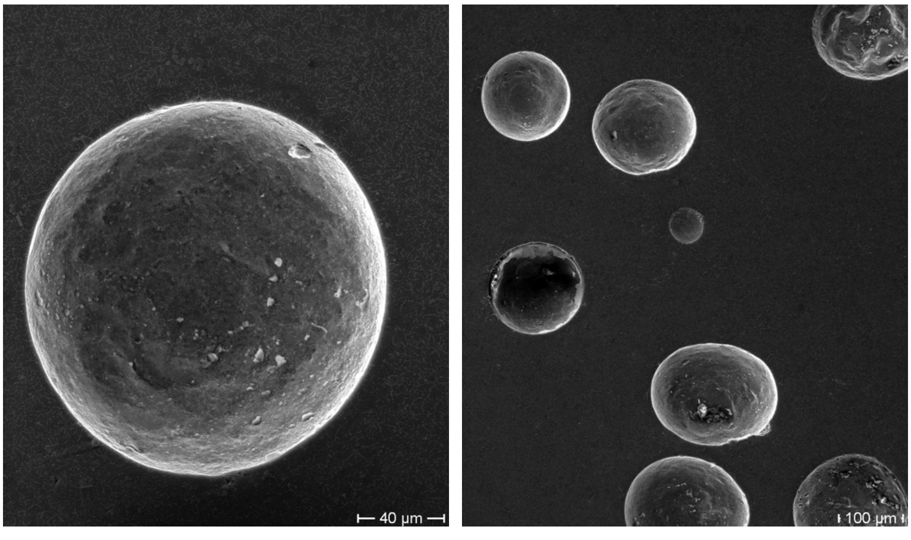


Figure 3: SEM micrographs showing gas porosity on the external surface of Alloy "C" specimen.

Considering the high affinity between the alloying elements and oxygen, initially casting and pouring were performed in a centrifugal furnace after 8 vacuum-argon washing cycles. Afterwards, in order to improve casting soundness, castings have been manufactured by means of vacuum induction melting. By adopting this preventing measure we obtained castings that did not contain gas defects.

A further critical aspect that comes to light in the presented investigation is the presence of shrinking cavities that seem to favour crack initiation and propagation. Shrinkage should not be a problem when manufacturing by centrifugal casting, since the material is constantly forced to instantly fill any vacancies that may occur during solidification and then it is important to evaluate if gas porosities and blowholes favour the formation of shrinkage cavities by avoiding locally compensation of liquid and solidification contraction. Figs. 2c and 4b show shrinkage cavities found on the fracture surfaces of Alloy "C" and Alloy "B" respectively. By pouring under vacuum in a preheated mould and by performing a slow cooling in a furnace gas defects are eliminated. Despite that SEM analyses showed that shrinkage cavities are still present as shown in Fig. 5. A considerable reduction of shrinkage cavities could only be achieved by means of a close control of process temperatures. Although melting under vacuum allows to get rid of gas porosities, as already stressed in some papers [16-18], it causes aluminium loss that requires during the charge material preparation a careful calculation of aluminium excess. Usually, the melting conditions of TiAl lie in the free evaporation area, thus the evaporation loss must be taken into account for the composition control. Experiments reported in literature [18] highlight that there exists a critical pressure and an impeding pressure for the evaporation losses of Al in a TiAl melt during the melting process under vacuum. When the vacuum pressure is less than the critical pressure, the evaporation of components takes on a state of free evaporation.

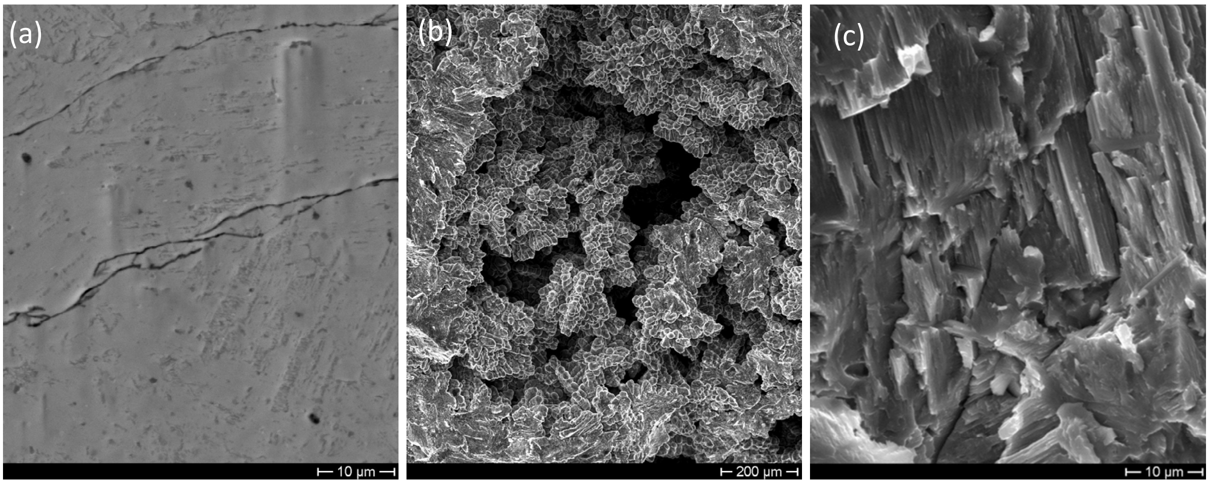


Figure 4: SEM micrographs of the Alloy "B" specimen showing a crack (a), a shrinkage cavity on the fracture surface (b) and the transgranular fracture (c).

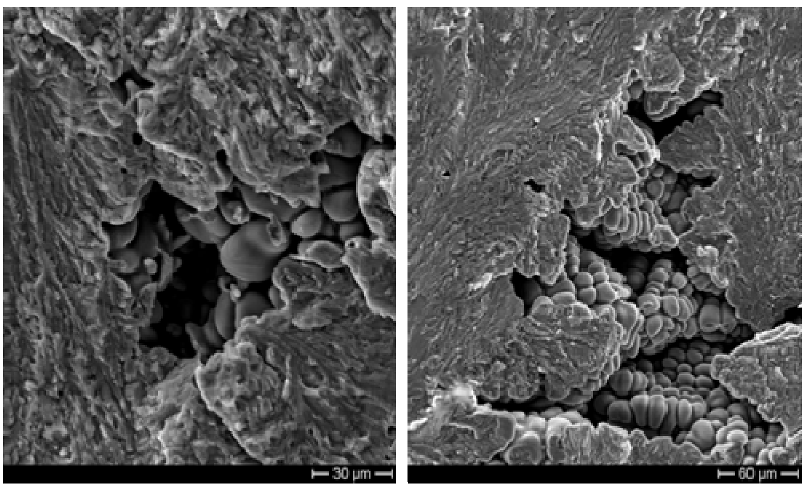


Figure 5: Shrinkage cavities found in the Alloy "P".

Investigations carried out on several specimens highlighted also that boron, added to the alloy to refine the microstructure, considerably increases its tendency to crack during or after cooling. Recently, it has been suggested that grain refinement using boron addition can increase the number of potential crack propagation sites, due to the appearance of aciculated boride particles. Therefore, a new heat treatment process based on massive transformation, that does not rely on boron, is under development as a way of achieving grain refinement and thus improved mechanical properties [12, 13].

As far as the fracture is concerned it appears transgranular since it travels through the grains. Figs. 2b and 4c show also that fracture propagates predominantly in a translamellar mode perpendicular to the lamellar interfaces although in localized areas propagation occurs in an interlamellar mode with crack advance occurring along $\alpha_2/\gamma$ interfaces.

The TiAl based alloys produced in this research are characterized by uniformly distributed fine lamellar colonies and a small quantity of residual primary β phase (so defined as it forms at high temperature) distributed around colony boundaries (bright phase in Figs. 2a, 6 and 7). Residual β phase is due to the presence in these alloys of β stabilising elements such as Nb and Mo. The disordered bcc structure of β phase is softer than α and γ phases at elevated temperature and it is expected to facilitate thermomechanical processing of TiAl alloys. However, it has been reported that both the coarse β particles existing in colony boundaries and excessive β phase precipitating from lamellar interfaces deteriorates creep behaviour and room-temperature ductility, while the precipitation of fine β particles is considered as intrinsic toughening mechanism [19]. By examining the path of cracks developing through the TiAl alloys produced in this work it can be seen (Figs. 2a, 4a and 6) that cracks do not propagate along the β/lamellar colonies interfaces and it seems that β phase distribution does not affect crack propagation. Experimental results highlighted that alloys characterised by a high content of β phase tend to fracture during either cooling or machining. In fact, alloys "N", "O" and "P" (Tab. 1) containing a high quantity of β stabilising elements broke up during cooling although they were cast under vacuum and subjected to slow cooling.

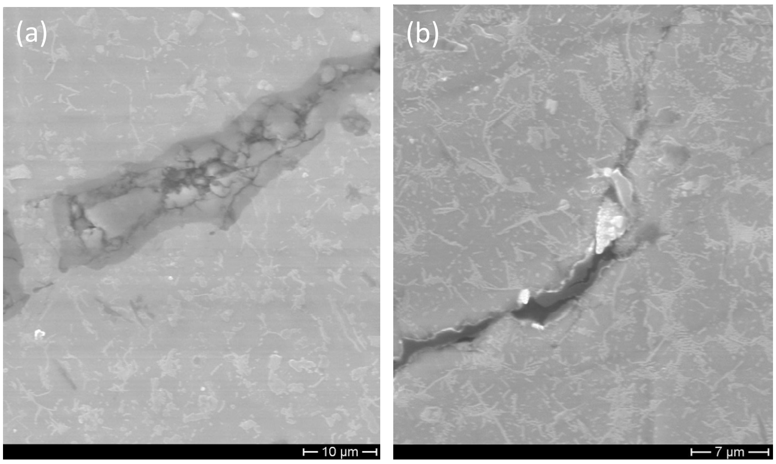


Figure 6: SEM micrographs showing two details of cracks that propagated in the Alloy "A" casting

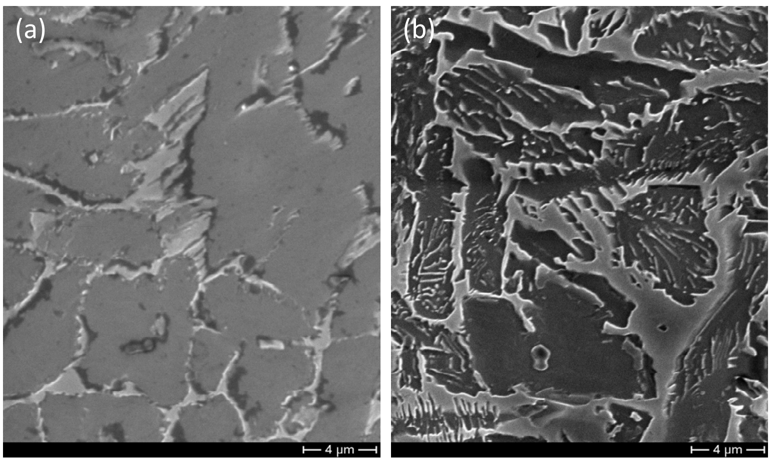


Figure 7: SEM micrographs showing the microstructures of Alloy "M" (a) and Alloy "N" (b).

By observing Fig. 7, it can be noticed that the addition of beta stabilising elements, over a certain limit, strongly increases the quantity of beta phase formed. This figure shows that by increasing the quantity of beta stabilising elements in the alloy from 10.9 at.% to 13 at.% the amount of beta phase (bright in Fig.7) noticeably raises, thus affecting the alloy behaviour.
A final factor that has to be considered is the effect of residual stresses. It is well known that internal stresses play an important role in determining the mechanical properties of materials. It is therefore worthwhile to evaluate the influence of the internal stresses on the fracture behaviour of TiAl-based alloys. Owing to the tetragonal crystal structure of γ phase and the hexagonal crystal structure of $\alpha_2$ phase, there is a lattice misfit between them. Deformation incompatibility across the lamellar interfaces and grain boundaries may occur. Moreover the presence of coarse β particles may increase this incompatibility: in fact it seems that the tendency to cracking increases by increasing the amount of β stabilising elements added to the alloy. Local accumulation and non-uniform distribution of internal strain and stresses introduced by all these effects may relate to brittle fracture behaviour. In order to understand whether internal stresses play an important role in the phenomenon we observed, as already said, many of the considered alloys were poured in a mould preheated at 550 °C and the castings were subjected to a very slow cooling in a furnace. By using this methodology the alloys' tendency to cracking was strongly reduced. The selected preventive measures proved to be ineffective for the alloys that contain a higher quantity of β stabilising elements and then a higher quantity of β phase.
A close control of superheating parameters, such as time and temperature, and of pouring speed could strongly affect the casting soundness. The effect of these parameters will be further investigated in subsequent studies.

## CONCLUSIONS

The study carried out on cracks and fracture surfaces of TiAl based alloy specimens fractured during or after cooling highlighted that there are many concurrent factors that produce specimen fracture. SEM analyses showed that microshrinkage cavities and gas porosity coupled with relevant residual stresses, probably related to the quantity of β phase, favour the explosive fracture of the considered alloys. In order to prevent this phenomenon the charge materials has been degreased and afterwards they have been preheated together with the crucible in a muffle to evaporate surface moisture. Further improvement have been obtained by pouring the alloy in vacuum in a preheated mould, leaving the casting to cool down in a furnace.
Further studies are required to understand the influence of quantity and distribution of β phase on internal residual stresses as well as the effect of superheating temperature and pouring speed on casting soundness.

## REFERENCES

[1] Clemens, H., Smarsly, W., Light-Weight Intermetallic Titanium Aluminides – Status of Research and Development, Adv. Mat. Res., 278 (2011) 551-556.
[2] Djanarthany, S., Viala, J.C., Bouix, J., An overview of monolithic titanium aluminides based on Ti3Al and TiAl, Mater. Chem. Phys., 72 (2001) 301.
[3] Dimiduk, D.M., Gamma titanium aluminide alloys - An assessment within the competition of aerospace structural materials, Mat. Sci. Eng. A-Struct., 263 (1999) 281.
[4] Kim, Y-W., Clemens, H., Rosenberger, A., Gamma Titanium Aluminides 2003, The Minerals, Metals and Materials Society (TMS), Warrendale, PA, USA (2003).
[5] Kim, Y-W., Morris, D., Yang, R., Leyens, C., Structural Aluminides for Elevated Temperature Applications, The Minerals, Metals and Materials Society (TMS), Warrendale, PA, USA (2008).
[6] Peters, M., Leyens, C., Titanium and Titanium Alloys, Wiley-VCH, Weinheim, Germany (2003).
[7] Brotzu, A., Felli, F., Pilone, D., Fracture toughness of TiAl-Cr-Nb-Mo alloys produced via centrifugal casting, Frattura ed Integrità Strutturale, 22 (2012) 20-25.
[8] Ye, H.Q., Recent developments in $Ti_3Al$ and TiAl intermetallics research in China, Mat. Sci. Eng. A-Struct., 263 (1999) 289-295.
[9] Yamaguchi, M., Inui, H., Ito, K., High-temperature structural intermetallics, Acta Mater., 48 (2000) 307-322.
[10] Lin, J.P. , Xu, X.J., Wang, Y.L., He, S.F., Zhang, Y., Song, X.P., Chen, G.L, High temperature deformation behaviors of a high Nb containing TiAl alloy, Intermetallics, 15 (2007) 668-674.

[11] Lapin, J., Gabalcová, Z., Solidification behaviour of TiAl-based alloys studied by directional solidification technique, Intermetallics, 19 (2011) 797-804.
[12] Wu, X., Review of alloy and process development of TiAl alloys, Intermetallics, 14 (2006) 1114-1122.
[13] Jarvis, D.J., Voss, D., IMPRESS Integrated Project-An overview paper, Mat. Sci. Eng. A-Struct., 413–414 (2005) 583-591.
[14] Vajpai, S. K., Ameyama, K., A novel powder metallurgy processing approach to prepare fine-grained Ti-rich TiAl-based alloys from pre-alloyed powders, Intermetallics, 42 (2013) 146-155.
[15] Brotzu, A., Felli, F., Pilone, D., Fracture behaviour of cast TiAl based intermetallic alloys, in: F. Iacoviello, G. Risitano, L. Susmel, XXII Convegno Nazionale IGF, Roma, Italy, (2013) 66-72.
[16] Gomes, F., Puga, H., Barbosa, J., Ribeiro, C.S., Effect of melting pressure and superheating on chemical composition and contamination of yttria-coated ceramic crucible induction melted titanium alloys, J. Mater. Sci., 46 (2011) 4922-4936.
[17] Yanqing, S., Jingjie, G., Jun, J., Guizhong, L., Yuan, L., Composition control of a TiAl melt during the induction skull melting (ISM) process, J. Alloy. Compd., 334 (2002) 261-266.
[18] Guo, J., Liu, G., Su, Y., Ding, H., Jia, J., Fu, H., The critical pressure and impeding pressure of Al evaporation during induction skull melting processing of TiAl, Metall. Mater. Trans. A, 33 (2002) 3249-3253.
[19] Chen, Y., Niu, H., Kong, F., Xiao, S., Microstructure and fracture toughness of a β phase containing TiAl alloy, Intermetallics, 19 (2011) 1405-1410.