

# Structural and mechanical evolution of the thermo-mechanical processed Ti-46Al-6.5Nb alloy

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## Abstract

The formation of a high quality as-cast structure for the Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si (at.%) alloy, with a new alloying concept, and also the application of a thermo-mechanical (TM) processing scheme was the subject of present investigation. The investigations consisted of structural and mechanical characterizations of the alloy in 6 different structural conditions: S1 – in cast state, S2 – after a heat treatment of homogenization, S3–S6 – after a TM processing scheme. The micro-structural investigations were performed using a SEM analyzing system, and an X-ray diffractometer. The investigations of mechanical properties have been based on tensile strength, modulus of elasticity, elongation to fracture measurements and fracture surface analysis for the as-cast sample, and on micro-hardness measurements for the TM processed samples. The alloy in cast condition, with specific near-lamellar morphology, has a homogeneous, fine-grained structure ( $d \sim 30 \mu\text{m}$ ), and no axial texture. Thus, a grain refinement of new proposed alloy was achieved. For the TM processed specimens, the micro-hardness measurements have revealed a proper concordance between HV values and micro-structural evolution of the samples, being argued and analyzed after each TM processing step. The presented results can underline that the new proposed alloy can be TM processed around 1200 °C, starting from an optimized as-cast structure.

**Key words:** intermetallics, thermo-mechanical processing, microstructure, scanning electron microscopy (SEM), XRD spectra, micro-hardness test

## 1. Introduction

Near gamma titanium aluminides are considered by the aerospace industry as proper high-temperature structural materials, which offer opportunities for substantial weight reductions, compared to Ni-based materials. Compared to Ti alloys, they offer better creep oxidation and burn resistance, and increased strength at elevated temperatures [1]. However, poor ductility and low fracture toughness, not only at room temperature but also at high temperatures, still represent the main obstacle against full utilization of these alloys [2–6]. Therefore, in the last years many studies have been performed to improve the structural and chemical homogeneity of near gamma TiAl-based alloys [2, 4, 7, 8], especially for most technically interesting alloys based

on Ti-(44–49)at.%Al but with several other containing elements. For this composition range, the structurally stable solid phases are: the hexagonal  $\alpha$  phase, the ordered  $\alpha_2$  phase, the disordered bcc  $\beta$  phase, and the ordered fcc  $\gamma$  phase. Generally, all phase separation and transformation during the solidification and subsequent solid alloy cooling, lead to coarse microstructures with casting textures and micro-segregation of alloying elements [9]. These structural characteristics of  $\gamma$  TiAl alloys, together with its inherent brittleness, make ingot to break-down and subsequently very difficult to be hot worked.

For that reason, over the last years, many studies [10–18] have been performed to describe and analyze structural and mechanical properties of various near gamma titanium aluminides in order to ob-

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Table 1. Structural state of the examined and analyzed Ti-Al-Nb alloy samples

Sample No.	Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si alloy (at.%)
S1	cast
S2	cast and heat treated for homogenization at 1200 °C/6 h/vacuum cooling with a furnace
S3	cast and laminated at 1100 °C with deformation degree of $\varepsilon = 5\%$
S4	cast and laminated at 1150 °C with deformation degree of $\varepsilon = 5\%$
S5	cast and laminated at 1200 °C with deformation degree of $\varepsilon = 5\%$
S6	cast and laminated at 1250 °C with deformation degree of $\varepsilon = 5\%$

tain valuable information pertaining to the chemical composition and microstructure features and thermo-mechanical processing of these alloys. One of the main conclusions of these studies refers to the necessity of achieving an important prerequisite for the commercialization of these alloys consisting in the production of high quality cast material, with a fine-grained microstructure, weak texture, and without chemical non-homogeneities [6], characteristics that can assure a subsequently efficient thermo-mechanical processing. These characteristics can be achieved via a rational selection of the alloy composition, by introducing proper alloying elements [4, 5].

Adhering to this research direction, present paper reports some structural and mechanical properties for a new near gamma alloy composition concept, with high Nb concentration and small additions of Cr, Ta, Ni, Si, a cold crucible levitation melting technique being used for its elaboration. Based on this investigation, it hopes to obtain a class of TiAl alloys which will permit to efficiently control the microstructure and uniformity of the obtained ingots.

The second main objective of present investigation is the study of the microstructure and micro-hardness of the initial optimized as-cast material, after a heat treatment of homogenization and a series of thermo-mechanical treatments, in order to analyze the effect of hot-working temperature on the phase constitution, microstructure conversion and mechanical characteristics of the alloy.

## 2. Material and methods

For present investigation, an alloy with nominal composition of Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si (at.%) has been elaborated, starting from elemental components and using a cold crucible levitation melting technique in a FIVES CELES MP 25 type furnace. The melting parameters were: furnace nominal power 25 kW, magnetic field frequency 215 kHz, melting temperature of 2000 °C, under controlled argon atmosphere and at low vacuum level of 0.2–0.3 bar.

The new selected multi-component chemical composition represents a new near-gamma alloy concept, in order to better connect an optimized chemical com-

position with improved mechanical behavior of a quality cast and thermo-mechanically processed alloy. This concept will be discussed and argued below.

After melting and casting, an ingot of 60 mm height and 30 mm diameter was obtained. After that, from the cast alloy ingot 6 specimens were prepared for 6 different structural conditions (Table 1). The sample S1 remains in the cast state. The S2 specimen was subjected to a heat treatment of homogenization at  $T = 1200\text{ °C}$ , for  $t = 6\text{ h}$ , and vacuum cooling at cooling rate of  $20\text{ °C min}^{-1}$ . The rest of the 4 specimens (S3–S6) have been thermo-mechanically processed. The thermo-mechanical process consists of plastic deformation by lamination with an imposed deformation degree of  $\varepsilon = 5\%$ , at a strain rate of  $0.02\text{ s}^{-1}$ , applied for 4 different temperatures variants: 1100, 1150, 1200 and 1250 °C. The lamination process had been performed using a MARIO DI MAIO LQR120AS laboratory mill.

The structural and mechanical characterization of the alloy ensued from following steps:

I. *Structural characterization* of the Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si alloy, in cast condition, heat treated condition and after each thermo-mechanical processing variant, has been performed using metallographic specimens prepared by cutting at 2 mm thickness size, grinding and polishing. The structure was examined using a scanning electron microscope (SEM), Tescan Vega II – XMU, and analyzed using a XRD diffractometer Panalytical X'Pert PRO MRD, with wavelength of Cu  $K\alpha$  ( $\lambda = 1.5418\text{ \AA}$ ).

II. *The mechanical characterization* of the Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si alloy in different structural condition consisted in:

– Tensile tests on samples in cast condition, with  $0.5 \times 3 \times 30\text{ mm}^3$  size (samples were cut from Ti-Al-Nb as-cast alloy ingot). Tensile tests have been performed at room temperature using a tensile-compression module, of GATAN MICROTTEST 2000N type, located inside the chamber of the aforementioned SEM, through this procedure, the ultimate strength and the elongation to fracture of the sample in cast condition have been determined.

– Analysis of the modulus of elasticity, using Ramberg-Osgood model, of the S1 specimen of as-cast alloy.

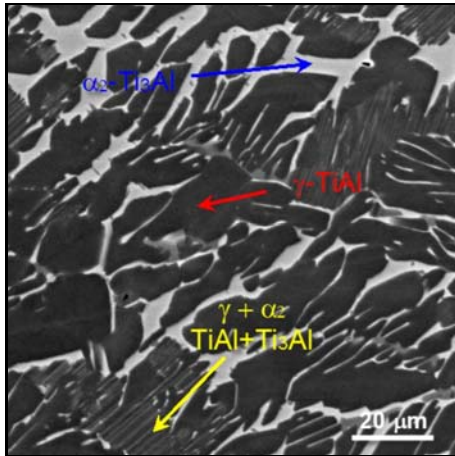


Fig. 1. SEM image of the sample S1 in as-cast condition.

– Analysis, using SEM microscopy, of the fracture surfaces of the S1 specimen corresponding to as-cast alloy.

– Micro-hardness determination for the Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si alloy in as-cast condition and after each thermo-mechanical processing variant, using a micro hardness tester, of WILSON-WOLPERT 401MVA type. The conditions for the micro-hardness test were the following: the micro-indentation of Vickers pyramid type, testing force = 500 gf, maintaining time under constant loading (dwell time) = 30 s, number of tests = 3, for each processing variant.

### 3. Results and discussion

Regarding the *structural characterization* of the studied alloy, there is presented a succession of SEM and XRD images in which the structural evolution of the proposed alloy can be viewed, starting from cast state (S1), passing through heat treated state (S2), and finally through each thermo-mechanical treatment step (S3, S4, S5, S6).

Figure 1 shows the SEM image of Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si as-cast alloy structure from which it can be asserted that this structure represents a *two-phase near-gamma alloy* with *near-lamellae* NL morphology. The alternation of two phases –  $\alpha_2$  and  $\gamma$  – can be observed in lamellar colonies with an average grain size of about 30  $\mu\text{m}$ , and with high thickness of  $\alpha_2$  and  $\gamma$  lamellae. The lamellar colonies have a mainly equiaxed shape. No  $\beta$  phase is found for this as-cast condition, fact asserted by XRD spectra from Fig. 3b, which indicates the presence of the  $\text{Ti}_3\text{Al}$ - $\alpha_2$  and  $\text{TiAl}$ - $\gamma$  phases. The specific near-lamellar morphology in as-cast conditions is also highlighted by images of fracture mode presented in Fig. 10, in the section of mechanical characterization of studied alloy.

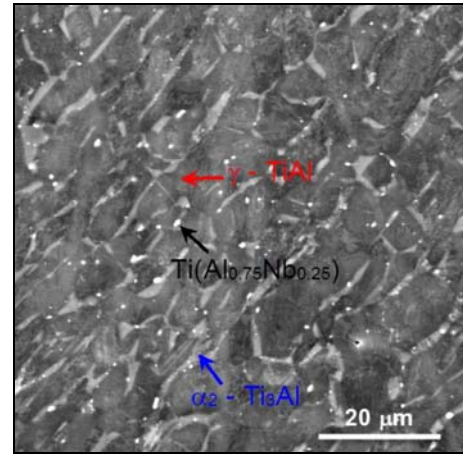


Fig. 2. SEM image of the sample S2, in cast and heat treated for homogenization state.

One must have in view that for obtaining this kind of structure/morphology there has been selected a specific new chemical composition that, by nature and quantity of alloying elements, can assure the proposed main objective – the obtaining of a high quality cast material. The most important goal in selecting the alloying elements for the present case was the concept, according to which a high quality cast material can be obtained by means of solidification through beta phase, which allows avoiding significant chemical non-homogeneity induced by peritectic reactions [4, 5, 11]. For this reason, niobium is the most important alloying element in this case, because it is a beta stabilizing element and also has low diffusion mobility in the  $\gamma$  and  $\alpha_2$  phases, thus facilitating a decrease rate of grain growth [2, 10, 11, 20]. Whereas Nb and Ta produce hardening through solubilization and reduction of ductility, the addition of Cr produces grain decreasing, with a significant increasing of ductility [22]. Elements like Cr, Si act like Al substitutes, whereas Nb and Ta act like Ti substitutes [19, 22].

As a result, present alloy obtained in cast state is relatively chemically homogeneous, with fine-grained structure ( $d \sim 30 \mu\text{m}$ ) and with no axial texture. Thus, a refinement in the colony size of the TiAl-based alloy was achieved merely through alloying.

For the second part of present work, with regards to another effective way to refine the grain size of the TiAl-based alloys, meaning through thermo-mechanical (TM) processing of the as-cast alloy, papers, such as [21, 23, 24], report different possibilities of TM processing, for which the dimensions of the initial grains are indicated to be at the same value or even higher than those obtained for present studied alloy (about 30  $\mu\text{m}$ ). For this reason, this initial dimension of the obtained cast alloy can be considered that represents a good value for further successful thermo-mechanical treatments.

Figure 2 shows the SEM image of the studied al-

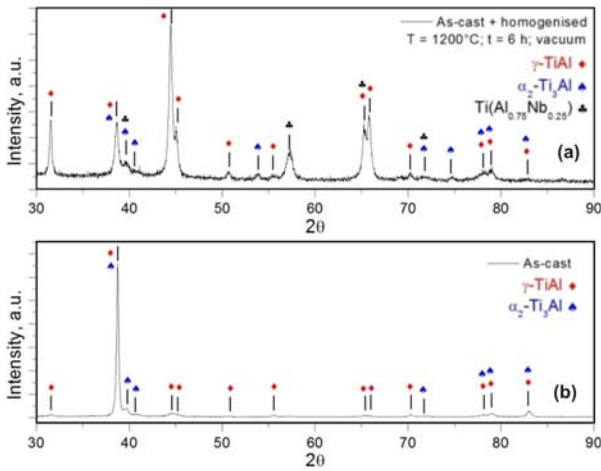


Fig. 3. XRD spectra of the studied alloy in as-cast condition – sample S1 (b) and after heat treatment for homogenization – sample S2 (a).

loy structure (sample S2), after a heat treatment of homogenization at 1200 °C/6 h vacuum cooling with furnace. Correlated with corresponding XRD spec-

tra from Fig. 3a, both the Figs. 2 and 3a indicate the apparition of the  $\text{Ti}(\text{Al}_{0.75}\text{Nb}_{0.25})$  precipitates, an increasing of the  $\gamma$  phase and a decreasing of the  $\alpha_2$  phase. These specific features indicate that the niobium segregation in specific precipitates hinders the development of the  $\alpha_2 + \gamma$  lamellar formations. In addition, this heat treatment slightly decreases the colony size from about 30  $\mu\text{m}$  in cast state to about 20  $\mu\text{m}$  after heat treatment of homogenization.

Figure 4 indicates the SEM images of the samples S3 (Fig. 4a), S4 (Fig. 4b), S5 (Fig. 4c) and S6 (Fig. 4d), after thermo-mechanical (TM) treatment consisting in rolling at 1100, 1150, 1200, and respectively 1250 °C, with the same deformation degree of  $\varepsilon = 5\%$ , at a strain rate of  $0.02\text{ s}^{-1}$ . Figures 5–8 indicate the XRD spectra for the samples S3 (Fig. 5), S4 (Fig. 6), S5 (Fig. 7) and S6 (Fig. 8).

The selection of the above indicated temperatures for the TM treatment had in view the reason of hot working the alloy in the interval between eutectoid temperature –  $T_e$  and  $\alpha$ -phase *transus temperature* –  $T_\alpha$ , both indicated to be approximately at 1175 °C for the  $T_e$ , and at  $1290 \pm 5^\circ\text{C}$  for the  $T_\alpha$ , for a similar alloy ( $\text{Ti-45Al-7Nb}$ ) in [25].

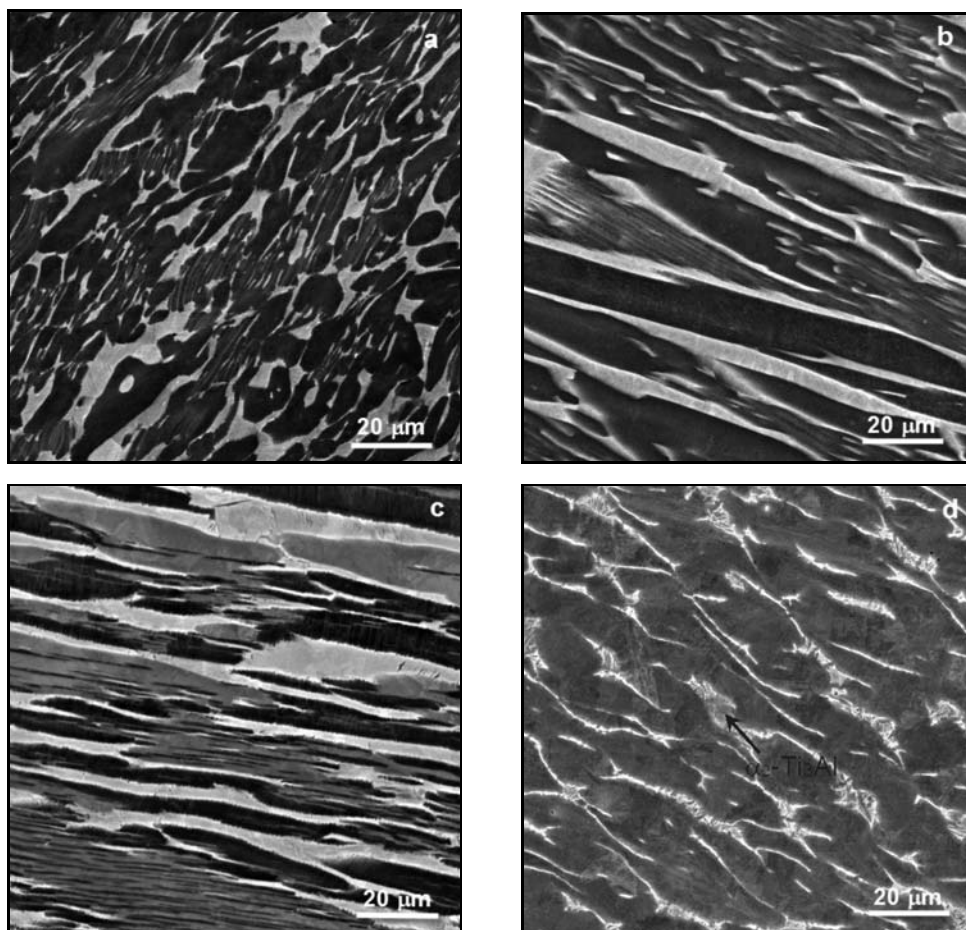


Fig. 4. SEM images of the samples S3 (a), S4 (b), S5 (c) and S6 (d) – cast and laminated at 1100 °C (a), 1150 °C (b), 1200 °C (c), 1250 °C (d), with deformation degree of  $\varepsilon = 5\%$ .

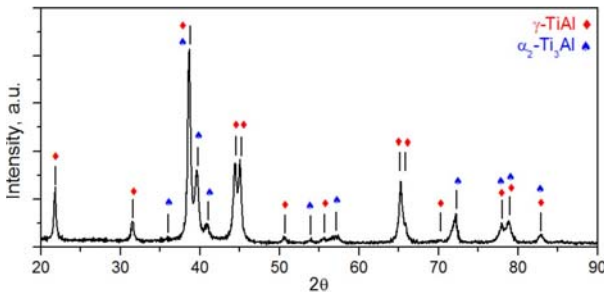


Fig. 5. The XRD spectra of the Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si studied alloy, laminated at 1100°C with deformation degree of  $\varepsilon = 5\%$  – sample S3.

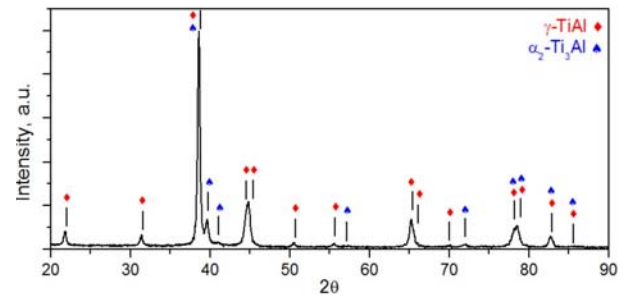


Fig. 8. The XRD spectra of the Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si studied alloy, laminated at 1250°C with deformation degree of  $\varepsilon = 5\%$  – sample S6.

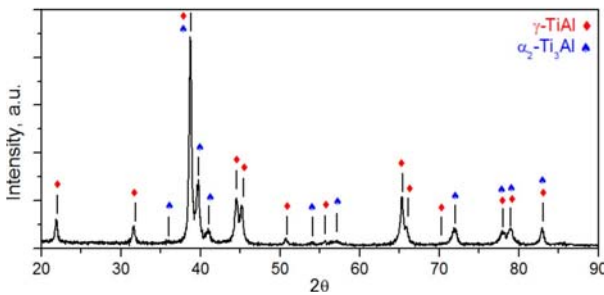


Fig. 6. The XRD spectra of the Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si studied alloy, laminated at 1150°C with deformation degree of  $\varepsilon = 5\%$  – sample S4.

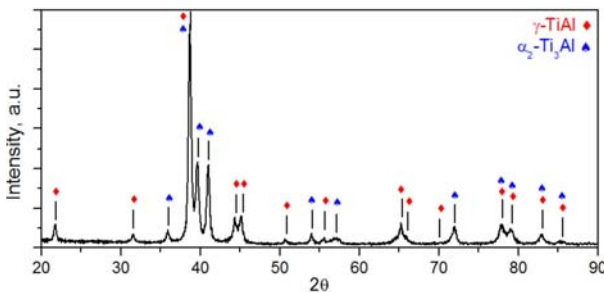


Fig. 7. The XRD spectra of the Ti-46Al-6.5Nb-0.5Ta-1Cr-0.3Ni-0.2Si studied alloy, laminated at 1200°C with deformation degree of  $\varepsilon = 5\%$  – sample S5.

Observing the evolution of the structural characteristics in Fig. 4, it can be asserted that above  $T_e$ , as the heating temperature increases from 1100 to 1200°C, the structure remains in the same state like as-cast one (two-phase near-gamma, with near-lamellae morphology), but with a gradual grain flattening on deformation direction. At 1150°C (Fig. 4b for S4) and 1200°C (Fig. 4c for S5) it can also be seen a relative increase of the inter-lamellar spacing size of the alloy, comparatively to 1100°C (Fig. 4a). In the case of the sample S6 (TM treated at 1250°C, Fig. 4d) it can be asserted that the structure was transforming

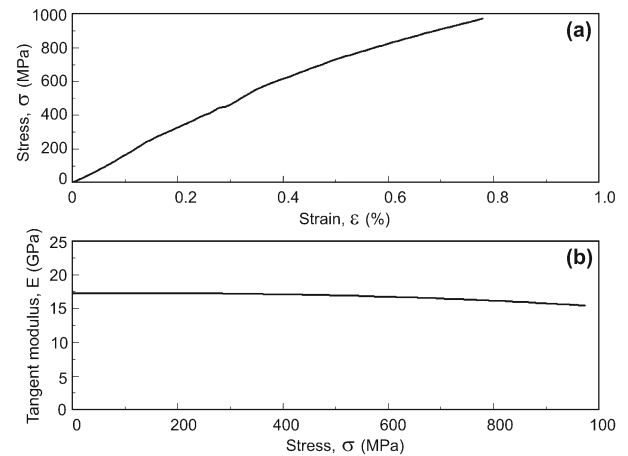


Fig. 9. Variation curve for tangent modulus as a function of applied stress (a) and the strain-stress curve (b) of as-cast Ti-46Al-6.5Nb alloy (alloyed with Cr, Ta, Ni, Si).

from near-lamellae one into a duplex type one, with a refined colonies size about 40–50  $\mu\text{m}$ .

The XRD spectra of the TM treated samples from Figs. 5–7 indicate that the volume fraction of the  $\gamma$  phase tends to decrease as the amount of  $\alpha$  phase increases, from sample S3 to sample S5. This fact, along with the grain deformation (visible from Figs. 4a–c), can explain the transformation into a duplex structure after TM processing at 1250°C, visible from Fig. 4d. During the hot TM deformation, the kinetic energy is stored in the deformed grains. As a result, the micro-segregation in the alloy can be eliminated due to the fast atomic diffusion, when heat treating the alloy in ( $\alpha + \gamma$ ) two-phase temperature range. Thus, when the alloy is heat treated at 1250°C, a duplex structure with a finer grain size is obtained, with no  $\beta$  phase. In future research work, it intends, by means of detailed analysis, to describe more precisely the structural evolution under TM processing, near  $T_\alpha$  temperature.

With regards to the *mechanical characterization* of the studied alloy, Fig. 9b shows the strain-stress

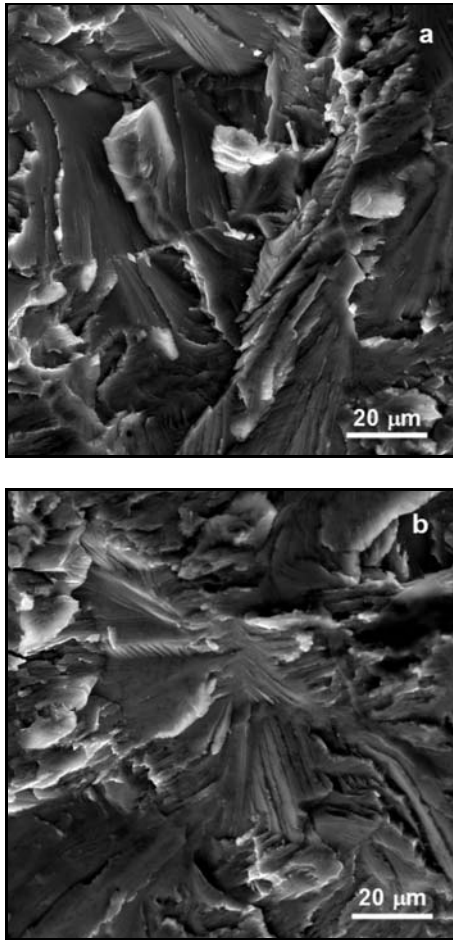


Fig. 10a,b. Two micro-fractographies of Ti-46Al-6.5Nb as-cast alloy (alloyed with Cr, Ta, Ni, Si).

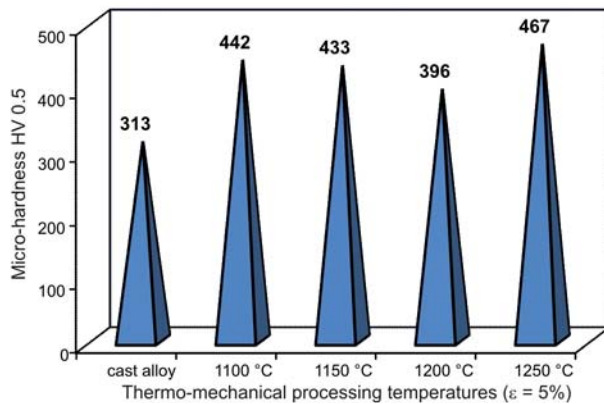


Fig. 11. The micro-hardness values variation for the alloy samples in cast status and after each thermo-mechanical processing variant.

diagram of the as-cast Ti-Al-Nb alloy, obtained after processing the tensile test at room temperature, through which the ultimate tensile strength  $\sigma_{\max} =$

943.49 MPa has been determined, as well as the elongation to fracture of the alloy  $\varepsilon_f = 0.78\%$ .

The analysis of the modulus of elasticity of the as-cast Ti-Al-Nb alloy has been also effectuated, using the Ramberg-Osgood model [26]. The Ramberg-Osgood equation is the following [26]:

$$\varepsilon = \frac{\sigma}{E_0} + 0.002 \left( \frac{\sigma}{\sigma_{0.2}} \right)^n, \quad (1)$$

where  $\varepsilon$  is the strain,  $\sigma$  is the stress,  $\sigma_{0.2}$  is the 0.2 % proof stress,  $E_0$  is the initial elastic modulus and  $n$  is the Ramberg-Osgood parameter. The  $n$  parameter can be obtained by using 0.01 and 0.2 % proof stress, using the following equation [26]:

$$n = \frac{\ln 20}{\ln \left( \frac{\sigma_{0.2}}{\sigma_{0.01}} \right)}. \quad (2)$$

The tangent modulus  $E_t$  is defined as the slope of the stress-strain curve at each stress value. The tangent modulus can be calculated using the following equation [26]:

$$E_t = \frac{E_0 \sigma_{0.2}}{\sigma_{0.2} + 0.002nE_0 \left( \frac{\sigma}{\sigma_{0.2}} \right)^{n-1}}. \quad (3)$$

Figure 9a indicates the variation curve for tangent modulus as a function of stress variation, determined on the basis of stress-strain diagram. The value of the obtained modulus of elasticity is 17.29 GPa.

Figure 10 represents two micro-fractographies, obtained by SEM microscopy, for the alloy in cast condition. The images with the same magnitude are from two different view fields. They represent a trans-granular fracture mode of the studied alloy. The fine lamellar morphology of the  $(\alpha_2 + \gamma)$  formations can be observed, corresponding to two-phase near-gamma structure, asserted and discussed above.

Figure 11 represents the evolution of the micro-hardness values of the studied alloy, after each thermo-mechanical processing variant, compared to the micro-hardness value of the as-cast initial sample. From Fig. 11 it can be asserted that the micro-hardness values are in concordance with micro-structural evolution of the alloy, discussed above by virtue of SEM and XRD images. Starting from as-cast sample, with  $HV_{0.05} = 313$ , Fig. 11 indicates an increase of the micro-hardness, to  $HV_{0.05} = 442$ , for the TM processed sample at 1100 °C, due to the plastic deformed structure and to the increased volume fraction of  $\gamma$  phase, signalized by XRD spectra from Fig. 5. Further, for the samples TM processed at 1150 °C and

respectively at 1200°C, the micro-hardness values are slightly decreasing to HV0.05 = 433, and respectively to HV0.05 = 396. This fact is in accordance with the decreasing of the  $\gamma$  phase volume fraction, the inverse increasing of the  $\alpha_2$  phase volume fraction, and also with the increasing of the inter-lamellar spacing, signalized from Figs. 4b,c. As concerning the sample S6, TM processed at 1250°C, the micro-hardness is anew increasing to HV0.05 = 467, due to structural transformation in duplex structure, harder than near-lamellar one [27]. There must be considered future research works for studying the evolution of other mechanical properties of the TM processed samples, like ductility or tensile strength. For this stage, the presented results underline that the new proposed TiAl based alloy can be TM processed around 1200°C, starting from an optimized as-cast structure, with promising results for various applicable schemes of TM processes.

#### 4. Conclusions

The aim of present work was to obtain a high-quality as-cast TiAl based alloy, with a new chemical composition, capable of a good plastic behavior during subsequent TM processing scheme.

The obtained alloy in cast condition, with specific two-phase structure and near-lamellar morphology, has a relatively chemical homogeneity, fine-grained structure ( $d \sim 30 \mu\text{m}$ ) and no axial texture. Thus, a refinement of colony size of new proposed TiAl-based alloy was achieved through alloying.

The mechanical properties determination for the as-cast specimen has revealed the ultimate tensile strength  $\sigma_{\text{max}} = 943.49 \text{ MPa}$ , the elongation to fracture of the alloy  $\varepsilon_f = 0.78 \%$ , and modulus of elasticity of 17.29 GPa, which represent promising values for a high-quality cast alloy.

For the TM processed specimens, the micro-structural investigations indicate an interesting evolution of the structural features: as the heating temperature increases from 1100 to 1200°C, the structure remains the same as as-cast initial one (two-phase near-gamma, with near-lamellae morphology), but with a gradual grain flattening on deformation direction. At 1150 and 1200°C, a relative increase of the inter-lamellar spacing size of the alloy can be also seen, comparatively to 1100°C. In the case of the sample TM-treated at 1250°C, it can be asserted that the structure is transforming from near-lamellae one into a duplex type one, with a refined colonies size of about 40–50  $\mu\text{m}$ .

As concerning the micro-hardness measurements for the TM processed specimens, they have revealed a proper concordance between HV values and micro-structural evolution of the samples, being argued in

detail and analyzed after each TM processing step, according to SEM and XRD spectra corresponding images. For this stage, the presented results underline that the new proposed TiAl based alloy can be TM processed around 1200°C, starting from an optimized as-cast structure.

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