INFLUENCE OF DEFORMATION ON THE STRUCTURE AND MECHANICAL PROPERTIES OF A TITANIUM-BASED ALLOY OBTAINED BY SELF-PROPAGATING HIGH TEMPERATURE SYNTHESIS

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Abstract

A fine-grained alloy with a Ti-48Al-2Nb-2Cr (in atom pct) composition was prepared by Self-propagating High temperature Synthesis (SHS). The relationship among different compression ratio, microstructure, porosity, density, mechanical properties and residual stresses of the Ti-48Al-2Nb-2Cr alloy was studied by scanning electron microscopy, X-ray diffraction, neutron diffraction and mechanical testing. The results show that the morphology of the SHS-synthesized alloy is regular with the size of 4−25 μm. The main γ-TiAl phase and the α_2-Ti_3Al and $TiAl_3$ phases were observed in the samples. The cell parameters of the γ-TiAl crystal lattice after SHS were determined. It was observed that during compression the processes of hardening as well as those of increasing residual stresses take place. The residual strains and stresses in bulk of the material were calculated.

Keywords: Self-propagating high temperature synthesis, Ti-48Al-2Nb-2Cr, titanium aluminides, neutron diffraction, mechanical properties

1. INTRODUCTION

Intermetallic compounds (intermetallics) are a unique class of materials which preserve the ordered structure and high values of strength properties and the modulus of elasticity until the melting point and which hold an intermediate position between metals and ceramic following their technological and operational properties [1]. Intermetallics and alloys on their basis are used in power engineering, aerospace industry and medicine. An additional advantage of alloys based on Ti-Al compounds is their low density. Among the outstanding casting alloys based on titanium aluminides with promising mechanical properties is the Ti-48Al-2Nb-2Cr (at.%) alloy. However, like most alloys based on intermetallics, its disadvantage is low plastic properties at room temperature.

Wide use of materials based on titanium aluminides is limited by the lack of a reliable industrial technology for obtaining homogeneous alloys of a given composition. The difficulties are caused by the difference in the melting temperatures of the elements (1668 °C vs. 660 °C) and the difference in the densities (4.5 g/cm^3 vs. 2.7 g/cm^3) of the main components of the alloy (Ti vs. Al, respectively) [2]. Therefore, the manufacture of finished products with the required properties on the basis of titanium aluminides is a complex, multi-stage and expensive process including up to ten technological operations [3].

In recent years, the technology of Self-propagating High Temperature Synthesis (SHS) has been used in the manufacture of intermetallics. First demonstrated in 1967 [4], the method is based on the use of the internal chemical energy of original agent powders released in the formation of solid reaction products. The SHS

technique reduces the production time, and thus costs and energy, compared to the traditional melting process [1].

The aim of the present study was to investigate the microstructure, phase composition and mechanical properties of the SHS-synthesized alloy Ti-48Al-2Nb-2Cr (at. %) and to evaluate the residual strains and stresses in bulk of the material after compressive deformation at room temperature for its possible industrial use.

2. EXPERIMENTAL

The Ti-48Al-2Nb-2Cr (at.%) titanium alloy under investigation was obtained by the Self-propagating High temperature Synthesis (SHS) method. Ti, Al, Nb and Cr metallic powders were mixed, the resulting mixture was compacted to form cylinders 8 mm in diameter and 4 mm high. After compacting at room temperature, the samples were placed in an oven and heated to the auto-ignition point. The surface was heated by a laser beam (CO_2) 6 mm in diameter and with a capacity of 60 W. The experiment was conducted in a vacuum chamber under an argon atmosphere at a pressure of approximately $1.5\pm0.5\cdot10^2$ Pa. he SHS reaction temperature was ca. 1400 °C. The reaction proceeded with the participation of molten aluminum, as indicated by a delay at 645 °C on the thermogram (not shown). It should be noted that after the SHS reaction the samples were not additionally pressed and heat treated.

The chemical composition of the experimental material, strain parameters and resulting mechanical properties are given in **Table 1**. The stress state in the obtained alloy was induced by the uniaxial compression of the samples using a mechanical testing machine with a true strain ε = 0.10; 0.15; 0.20 at room temperature. Compression at ε = 0.15 was carried out at two strain rates - ε = 0.001 s^{-1} and ε = 1 s^{-1}.

The microstructure of the samples was studied by Scanning Electron Microscopy (SEM) using the Philips XL20 microscope. X-ray diffraction (XRD) measurements were carried out with a step scan diffractometer DRON 4-13 using $Mo_{K\alpha}$ radiation ($\lambda_{K\alpha}$ = 0.0711 nm) in the range of angles 10-55º. The X-ray patterns were interpreted according to the data in [5]. The density of the samples before and after the mechanical impact was determined by a standard hydrostatic method based on Archimedes' principle by weighing the samples in air and in water. The hardness measurements of the samples were carried out using a microhardness tester PMT-3 and a Vickers indenter with a tip angle of 136°. The maximum load was chosen as 0.98 N (100 gram). Twenty indentations were performed on each sample. By means of special tables, the obtained values of microhardness HV_{100} were converted into Brinell hardness *HB* which was used to evaluate such mechanical properties as yield strength $\sigma_{0.2}$ according to formula (1):

$$\begin{cases} \sigma_{0.2} = 0.2 \cdot HB, & HB < 1500 \text{ MPa} \\ \sigma_{0.2} = 0.367 \cdot HB - 240, & HB \geq 1500 \text{ MPa} \end{cases} \quad (1)$$

and ultimate tensile strength σ - according to the table of dependence σ on *HB* [6].

Table 1 Chemical composition, deformation parameters and mechanical properties of tested alloy

Content (at.%)				**Conditions of deformation**		**Mechanical properties**				
Ti	Al	Nb	Cr	true strain ε	strain rate $\dot{\varepsilon}$ (s^{-1})	HV_{100} (MPa)	*HB* (MPa)	$\sigma_{0.2}$ (MPa)	σ (MPa)	$\sigma_{0.2}/\sigma$
Base	48 ± 0.5	2 ± 0.2	2 ± 0.2	0	0	2256	2197	566	745	0.760
				0.10	0.001	3181	2976	852	989	0.862
				0.15	0.001	3381	3171	924	1054	0.876
					1.000	3690	3484	1039	1165	0.891
				0.20	0.001	3524	3316	977	1107	0.882

For determining of the residual stresses in the SHS-synthesized alloy Ti-48Al-2Nb-2Cr we chose to perform neutron diffraction measurements at the Laue-Langevin Institute (ILL) SALSA reactor facility in Grenoble. The theoretical details and the used parameters of a neutron diffraction experiment are given in [7]. Briefly, a monochromatic neutron beam of a cross section of 0.6 × 0.6 mm^2 was used for residual strain measurements at room temperature, in a spatially resolved mode. The Bragg peaks (110) and (211) for γ-phase (TiAl) were measured to $2\theta = 45°$ and 83.5°, for deformed and non-deformed samples, respectively. The data of the experiment on neutron diffraction were processed using the LAMP (Large Array Manipulation Program).

3. RESULTS AND DISCUSSION

The microstructure of the samples obtained by SEM is shown in **Figure 1**.

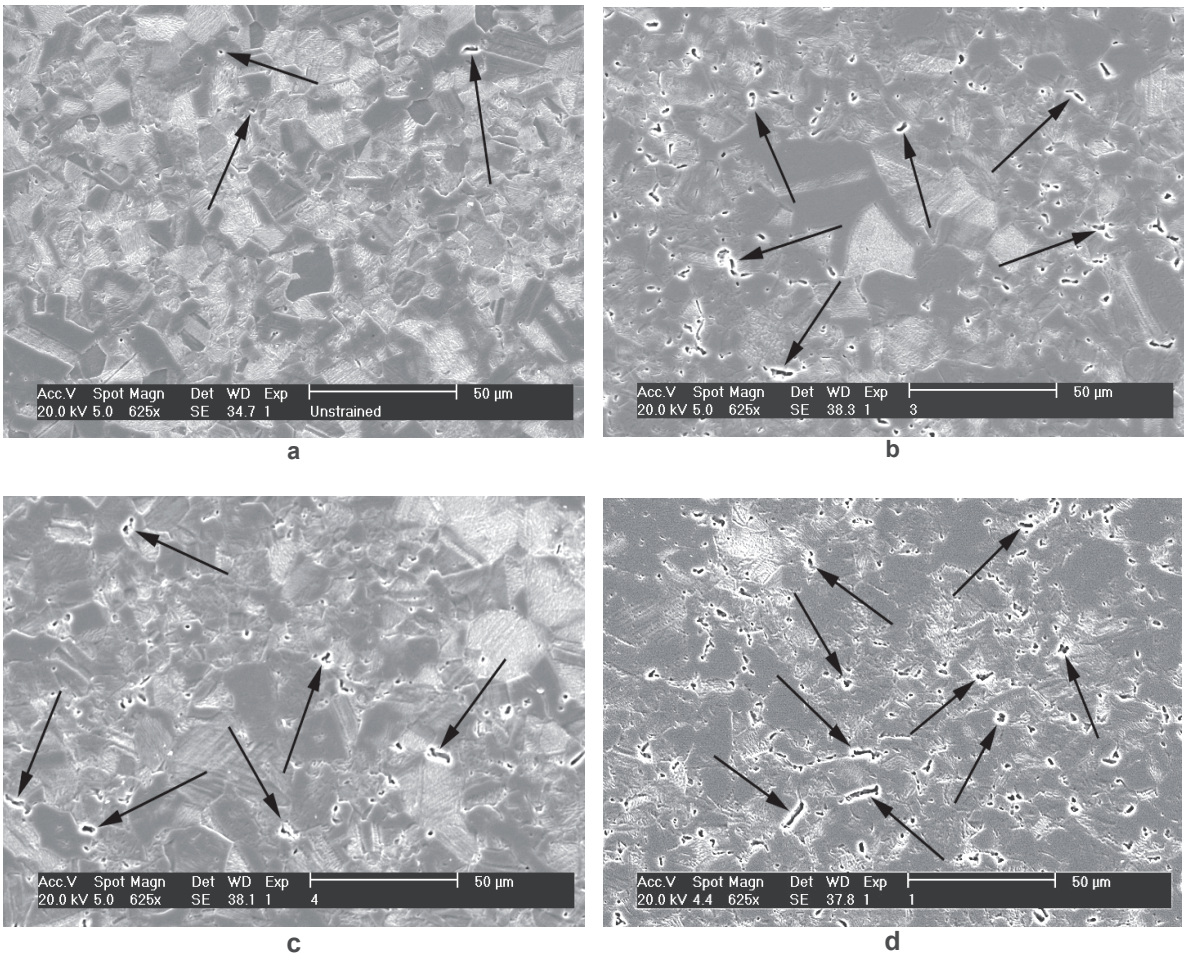

Figure 1 SEM images of the alloy Ti-48Al-2Nb-2Cr in the initial state and after compression: a) initial state; b) $\varepsilon = 0.15$, $\dot{\varepsilon} = 0.001\ s^{-1}$; c) $\varepsilon = 0.15$, $\dot{\varepsilon} = 1\ s^{-1}$; d) $\varepsilon = 0.20$, $\dot{\varepsilon} = 0.001\ s^{-1}$

Both before (see **Figure 1a**) and after (see **Figures 1b-d**) deformation, two structurally distinguishable components are observed, one of them having a homogeneous structure, while the etching of the other reveals an alternation of at least two phases in the form of plates. According to the phase equilibrium diagram of Al-Ti [8] and our XRD measurements (not shown), the main phase in this alloy is the γ-phase (TiAl) with the presence of the α_2 (Ti_3Al) phases and a small amount of $TiAl_3$. Thus, the homogeneous structural component is identified as a γ-phase, and the lamellar component is an eutectoid of the (α_2+γ) composition. In the initial (undeformed) state, the number of structural components of the γ-phase (Q_γ) and the eutectoid $Q_{eut\text{-}d}$ is related as Q_γ:$Q_{eut\text{-}d}$

= 1.3:1. As a result of deformation, this ratio increases with an increase in the true strain ε and at the strain rate $\dot{\varepsilon}$ = 0.001 s^{-1} equals Q_γ:Q_{eut-d} = 1.4:1; 1.8:1; 2.6:1 for ε = 0.10; 0,15; 0.20, respectively. At $\dot{\varepsilon}$ = 1 s^{-1} and ε = 0.15, the value of Q_γ:Q_{eut-d} = 1.5:1. The method of measuring the chord length in the plane of the section which is normal to the direction of compression made it possible to determine that for all samples the grain sizes are in the range 4-25 μm, and their distribution is asymmetric with respect to the average values (7.6, 8.2, 8.5 and 9.9 μm for ε = 0, 0.10, 0.15, 0.20, respectively, and $\dot{\varepsilon}$ = 0.001 s^{-1}) with a shift toward larger grain sizes.

In the initial (undeformed) state, there are material discontinuities (indicated by arrows in **Figure 1a**), the share of which increases with an increase in the strain ε (compare **Figures 1b and d**). The experimentally determined values of the pore volume fractions V in their dependence on ε are well approximated by the regression equation of the form: $V = 32.4 \cdot \varepsilon^2 + 1.7 \cdot \varepsilon + 1.3$. Under the conditions of this experiment, the dependence of V on the strain rate $\dot{\varepsilon}$ was not revealed (cf. **Figures 1b and c**).

The hydrostatic weighing method established that in the experimental range of true strains ε the alloy density ρ decreases linearly depending on ε (for $\dot{\varepsilon}$ = 0.001 c^{-1}) in accordance with the expression of the form $\rho = 4.040 - 0.787 \cdot \varepsilon$, g/cm^3. The values of the density ρ at ε = 0.15 are the same for both strain rates $\dot{\varepsilon}$ (0.001 and 1 s^{-1}). It is obvious that a decrease in the material density is connected with the formation of discontinuities during deformation.

The XRD method ($Mo_{K\alpha}$ radiation, $\lambda_{K\alpha}$ = 0.0711 nm) made it possible to reveal that the main phase in the initial state is the γ-TiAl phase with a tetragonal crystal lattice, as well as the α_2-Ti_3Al and $TiAl_3$ phases (not shown). The calculated γ-TiAl phase lattice parameters are a = 0.4009 nm, c = 0.4051 nm. Although the presence of the $TiAl_3$ phase requires a higher Al content, because the range of its homogeneity starts from ca. 65 % Al (at.) [8], many researchers (see, e.g., [9]) detect its presence by the XRD method in alloys with the Al content ≤ 50% (at.). The samples after deformation reveal an increase in the peak intensity corresponding to the γ-phase, some peaks broaden and their intensity changes, the background level increases, which indicates an increase in internal stresses.

The calculation results for hardness according to the *HB* scale, the yield stress $\sigma_{0.2}$, and the ultimate tensile strength σ, which were determined from the experimentally determined values of microhardness HV_{100}, are given in **Table 1**. The dependence of hardness *HB* on the value of the true strain ε can be well approximated by a second order polynomial: $HB = 2060 + 11400 \cdot \varepsilon - 25400 \cdot \varepsilon^2$, MPa. Differentiating the obtained dependence *HB(ε)* by ε, we obtain: $\dot{HB} = -50800 \cdot \varepsilon + 11400$. Thus, the increase in hardness ΔHB by a single change in deformation $\Delta\varepsilon$ ($\Delta HB/\Delta\varepsilon$) decreases with an increase in the external impact ε, and the material hardening reaches its maximum at $\varepsilon \approx 0.22$. The comparison of the values of hardness *HB* equal to 3,171 MPa at $\dot{\varepsilon}$ = 0.001 s^{-1} and 3,484 MPa at $\dot{\varepsilon}$ = 1 s^{-1} in **Table 1** show that higher compression rates $\dot{\varepsilon}$ initiate greater hardening of the alloy.

The calculated value of the $\sigma_{0.2}/\sigma$ parameter equal to 0.76 MPa for a sample in its initial state increases with an increase in the true strain ε and also depends on the strain rate $\dot{\varepsilon}$. Thus, the maximum value $\sigma_{0.2}/\sigma$ = 0.891 was observed in a sample deformed at ε = 0.15 and $\dot{\varepsilon}$ = 1 s^{-1}.

The calculated data of mechanical characteristics show that in deformation at room temperature, the hardness and the $\sigma_{0,2}/\sigma$ parameter increase, which indicates an increase in the brittleness of the alloy and a decrease in its capacity for subsequent forming. Among the determining factors contributing to such behavior of the material is the presence of strong residual stresses in the metal deformed after SHS. They were evaluated using the neutron diffraction method with the help of which the angles of diffraction maximums formed by the family of planes (211), $2\theta_{(211)}$, and (110), $2\theta_{(110)}$ were determined in initial and deformed samples. γ-TiAl has an AuCu-type structure, therefore, Ti and Al atoms alternate in the (110) direction. The experimental data and the hypotheses on the tendency of residual strains to their spontaneous minimization served as the basis for

determining the parameters of an elementary tetragonal γ-TiAl cell: *a* = 0.3999 nm, *c* = 0.4199 nm. The value of the residual strain ε^{res} in the principal (axial and radial) directions was calculated in accordance with the equation $\varepsilon^{res} = \left(d^{110} - d_0^{110}\right) / d_0^{110}$, where d^{110} - the measured lattice parameter; d_0^{110} - the "stress free" lattice parameter; residual stresses σ^{res} - in accordance with the generalized Hooke's law for an isotropic body [7]. The calculation results are given in **Table 2**.

Table 2 The values of residual strains ε^{res} and stresses σ^{res} in the family of planes (110) in the axial and radial directions of compression

Conditions of deformation		Direction of compression			
true strain ε	strain rate ε̇ (s^{-1})	axial		radial	
		$\varepsilon_{axial}^{res}$ (%)	σ_{axial}^{res} (MPa)	$\varepsilon_{radial}^{res}$ (%)	σ_{radial}^{res} (MPa)
0.10	0.001	-0.37	-319	0.34	626
0.15	0.001	-0.36	-122	0.49	1008
	1.000	-0.36	-532	0.13	127
0.20	0.001	-0.36	-5	0.60	1279

As shown in **Table 2**, in the experimental range of true strains ε, the residual strains $\varepsilon_{axial}^{res}$ in the direction of compression do not depend on the value ε and are ~ -0.36%. Thus, already at the minimum value ε = 0.10, the approach of atomic planes (110) is maximum. The residual strains in the plane $\varepsilon_{radial}^{res}$ which is normal to the direction of compression are proportional to ε. The calculated values of the residual stresses are significant and in all the tests exceed the values of the Ti-48Al-2Nb-2Cr alloy ultimate tensile strength given in [10].

Consequently, with an increase in the value of the true strain ε, there is an increase in the amount of the γ-phase and in the average grain size in the sample's plane which is normal to the direction of compression. Along with this, new discontinuities are formed and the material density decreases. The latter can be explained by the lack of sufficient accommodation of deformation on grain boundaries (a small number of slip and twinning systems) at room temperature [11] or by significant compressive stresses exceeding the ultimate strength, which is revealed by the neutron diffraction method.

Thus, the main strategy for the industrial use of the Ti 48Al-2Nb-2Cr alloy obtained by the SHS method is post-processing which leads to a decrease in brittleness and an increase in material ductility (e.g., hot deformation), as well as to lower residual stresses in the material (e.g., ion implantation [12], high-intensity pulse electric current treatment [13], etc.).

4. CONCLUSION

Self-propagating high temperature synthesis (SHS) is a useful technique to produce multicomponent titanium-based alloys, such as Ti-48Al-2Nb-2Cr (in atom pct). The obtained alloy is characterized by a duplex structure consisting of rounded grains of the γ-phase (TiAl) and grains consisting of alternating α_2-phase (Ti_3Al) and γ-phase plates. With the value of the true strain ε increasing, there is an increase in the amount of the γ-phase and in the average grain size in the sample's plane normal to the direction of compression, as well as the formation of new discontinuities and a decrease in the density of the material. During compression, the material hardens, which is accompanied by an increase in hardness, while the increase in hardness Δ*HB* by a single change in the strain ε (Δ*HB*/Δε) decreases with an increase in the true strain ε and the material hardening reaching its a maximum at ε ≈ 0.22. The alloy under investigation in its initial state has a relatively high value

of the ratio $\sigma_{0.2}/\sigma$ = 0.76, the latter increasing together with an increase in the true strain ε, which indicates a low ability to forming in the cold state without cracking. In order to decrease very high residual stresses in bulk of the material and to increase the workability of the Ti-48Al-2Nb-2Cr alloy obtained by the SHS method, it is necessary to perform hot deformation or special additional processing.

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