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SYNTHESIS AND CHARACTERIZATION OF METALLIC-INTERMETALLIC Ti-TiAl$_3$, Nb-Ti-TiAl$_3$ COMPOSITES PRODUCED WITH ELECTRIC-CURRENT-ACTIVATED SINTERING (ECAS)

SINTEZA IN KARAKTERIZACIJA KOMPOZITOV KOVINA-INTERMETALNA ZLITINA Ti-TiAl$_3$, Nb-Ti-TiAl$_3$, IZDELANIH S SINTRANJEM, AKTIVIRANIM Z ELEKTRIČNIM TOKOM (ECAS)

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In this study, we investigated the fabrication of in-situ metallic-intermetallic Ti-TiAl$_3$ and Nb-Ti-TiAl$_3$ composites from a powder mixture containing w = 40 % titanium and w = 60 % aluminum with (w = 5 % and w = 10 %) and without a metallic-niobium addition. Powder mixtures without a binder were compressed uniaxially under a pressure of 130 MPa and sintered at a current of 2000 A for 20 min in a steel mould using the electric-current-activated sintering method. The microstructures of the sintered samples were investigated with light and scanning electron microscopes, the phases in the samples were analyzed with XRD and their hardness was measured with a Vickers hardness tester. Light and scanning electron microscope (EDS) investigations showed that the microstructures of non-reinforced titanium-aluminum samples consisted of two components: the main component was titanium aluminide and the other one was metallic titanium. Also, there was a trace amount of aluminum oxide in the sintered body. XRD analyses also demonstrated that the main phase in the composite is TiAl$_3$. The XRD analyses of the samples reinforced with w = 5 % and w = 10 % of niobium to enhance the ductility of the body showed that metallic niobium and Ti-TiAl$_3$ remained the main phase. In addition, the average hardness values of the samples of the non-reinforced Ti-TiAl$_3$ composite, w = 5 % Nb-reinforced Ti-TiAl$_3$ composite and w = 10 % Nb-reinforced Ti-TiAl$_3$ composite were measured to be about (759, 669 and 600) HV, respectively.

Keywords: intermetallics, powder metallurgy, electric-current-activated sintering

V tej študiji je bila preiskovana in situ izdelava kompozitov kovina-intermetalna zlitina Ti-TiAl$_3$ in Nb-Ti-TiAl$_3$ iz mešanice kovinskih prahov z w = 40 % titana, w = 60 % aluminija z dodatkom kovinskega niobija (w = 5 % in w = 10 %) ali brez njega. Mešanica prahov brez veziva je bila enoosno stisnjena pri tlaku 130 MPa in sintrana pri toku 2000 A 20 min v jekleni formi z uporabo metode sintranja, aktiviranega z električnim tokom. Mikrostruktura sintranih vzorcev je bila preiskovana s svetlobno in vrstično elektronsko mikroskopijo, faze so bile analizirane z XRD, njihova trdota pa je bila izmerjena z merilnikom trdote po Vickersu. Svetlobni in vrstični elektronski mikroskop (EDS) sta pokazala, da je mikrostruktura neojačanega titanovega aluminida sestavljena iz dveh komponent: glavna komponenta je bila titanov aluminid, druga pa kovinski titan. V sintrani osnovi so bili tudi sledovi aluminijevega oksida. XRD-analize so pokazale, da je glavna sestavina kompozita TiAl$_3$. XRD-analiza vzorca, ojačanega z w = 5 % in w = 10 % niobija za povečanje duktilnosti osnove, je pokazala, da sta kovinski niobij in Ti-TiAl$_3$ glavni fazi. Izmerjene trdote vzorcev Ti-TiAl$_3$-kompozita brez ojačitve in z w = 5 % Nb ter w = 10 % Nb utrjenega Ti-TiAl$_3$-kompozita so bile (759, 669 in 600) HV.

Ključne besede: intermetalne zlitine, prašna metalurgija, sintranje, aktivirano z električnim tokom

1 INTRODUCTION

Titanium aluminides have several potential applications in the aerospace, automotive and turbine-power-generation markets due to their light weight and considerable high-temperature strength.[1–3] To meet the rising demand of the aerospace and automobile industries for light-weight high-temperature structural materials, researches focus on light-weight and high-strength intermetallics. Due to their low density, high specific strength and reasonable oxidation resistance, ordered intermetallic compounds based on aluminides have been developed, for decades, as high-temperature structural materials, such as γ-TiAl and NiAl. Unfortunately, the inherent low-temperature ductility of TiAl alloys at ambient temperature still remains one of the main obstacles for practical applications.[4,5] Especially, Al$_3$Ti has a superior strength, notable oxidation properties and the lowest density [3.3 g/cm^3]. Al$_3$Ti has not been considered as a structural material yet because of its low ductility at ambient temperature.[4–8] Therefore, various ternary and other additives, such as V, Nb, Cr, Mn, W, Ta, Mo, Si, B, C and others, have been used to improve the room-temperature ductility and strength, and to suppress high-temperature oxidation.[9,10]

Metallic-intermetallic composites can be designed for structural use to optimize the unique properties and benefits of the constituent components, resulting in the materials that have the high strength and stiffness of the intermetallic phase and the high toughness of the metal. Intermetallics have been reinforced with the particles, rods or layers of ductile metals in order to increase the toughness. A ductile-phase reinforcement of a brittle material creates a zone of bridging ligaments that restrict the crack opening and growth by generating closure tractions in the crack wake and utilize the work of plastic deformation in the ductile metal phase to increase the fracture resistance of the composite.[11,12]

To produce metallic-reinforced intermetallic-based composites, the pressure-assisted electric-current-activated sintering method is an alternative method. The use of ECAS for consolidating samples not only provides a faster heating time and shorter dwell time but also gives lower sintering temperatures. In this technique, an electric current is applied simultaneously with a mechanical pressure to consolidate or synthesize and to densify specific products into a desired configuration and density.[13]

In this study, we aimed at producing titanium-titanium aluminide (Ti-TiAl$_3$) and niobium-titanium-titanium aluminide (Nb-Ti-TiAl$_3$) metallic-intermetallic composites from a metallic Ti, Al, Nb powder mixture. The scope of this study is to improve the ductility of the composites while optimizing the composites in terms of metallic- and intermetallic-phase composition, enhancing the toughness of the brittle TiAl$_3$ intermetallic phase using metallic niobium and titanium. It is important that the electric-current-activated sintering (ECAS) technique can be used for producing such metallic-intermetallic composites.

2 EXPERIMENTAL PROCEDURE

2.1 Materials and the method

Metallic Ti, Al, Nb powders (99.5 % purity, finer than 44 µm) were used as the starting powder mixture for the formation of a TiAl$_3$ intermetallic-based metallic Ti- and Nb-Ti-reinforced in-situ composite. The powder mixtures were prepared with three different compositions, denoted as shown in **Table 1**.

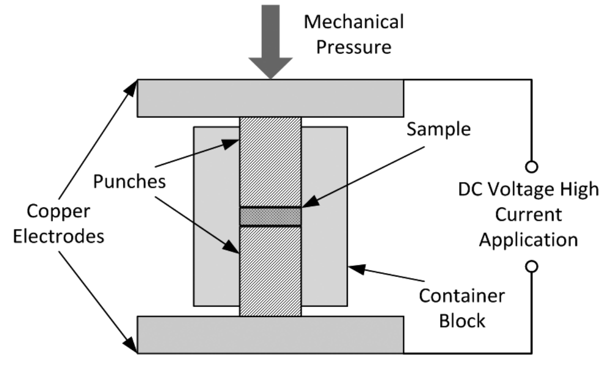


Figure 1: Schematic presentation of the electric-current-assisted sintering (ECAS) process
Slika 1: Shematski prikaz postopka sintranja z električnim tokom (ECAS)

Table 1: Codes for powder mixtures in mass fractions, *w*/%
Tabela 1: Oznake mešanic prahov v masnih deležih, *w*/%

A1	40 % Ti-60 % Al
B1	38 % Ti-57 % Al-5 % Nb
B2	36 % Ti-54 % Al-10 % Nb

In order to provide the contacts between individual powders in the initial period of the process, the A1, B1, B2 samples were pressed with a die with a compression load of 130 MPa for 1 min. After obtaining the samples, direct electric current (2000 A, 0.9–1.1 V) was applied to the steel substrate with a pressure of 55 MPa for 20 min using the electric-current-activated sintering technique in an open-atmosphere ECAS system, as shown in **Figure 1**. After the sintering, the specimens were unloaded and cooled to room temperature.

2.2 Characterization

The morphologies of the samples and the presence of the phases formed were examined with light microscopy and scanning electron microscopy (SEM-EDS). Also, an X-ray diffraction (XRD) analysis using the Cu K_α radiation with a wavelength of 1.5418 A over a 2θ range of 10–80° was done. The micro-hardness of the test

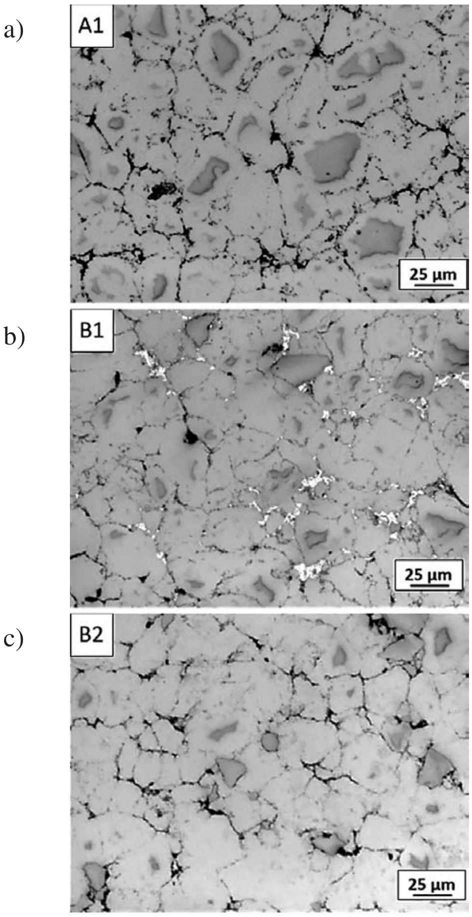


Figure 2: Light micrographs of the sintered: a) A1, b) B1, c) B2 samples
Slika 2: Mikrostruktura sintranih vzorcev: a) A1, b) B1 in c) B2

materials was measured with the Vickers indentation technique with a load of 0.98 N using a Leica WMHT-Mod model Vickers hardness instrument.

3 RESULTS AND DISCUSSION

3.1 Microscopic analysis

Light micrographs of the samples are shown in **Figure 2**. On the A1 micrograph, we can see three main areas. One of them, the darker area shaped as a small island, is titanium; the main phase is the TiAl$_3$ intermetallic phase. There is also a small amount of pores in the structure. The pores include the retained aluminum and a trace amount of oxygen. Besides this micrograph, B1 and B2 have the same appearance. We cannot see the Nb phase on these light micrographs, because the metallic Nb phase is lighter than the other phases. However, we can detect the Nb phase from the SEM-BES analyses as shown in **Figure 3**.

3.2 SEM-EDS analyses

The microstructures of Ti and TiAl$_3$ metallic-intermetallic composites (taken from the back-scattered mode of SEM) are shown in **Figure 3a**. As shown in this figure, darker areas are the intermetallic phases and the white reinforced phase consists of titanium metallic

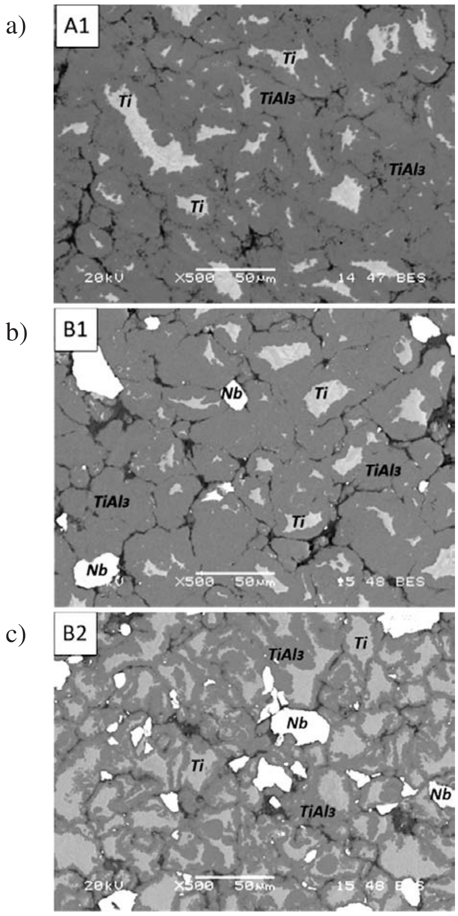
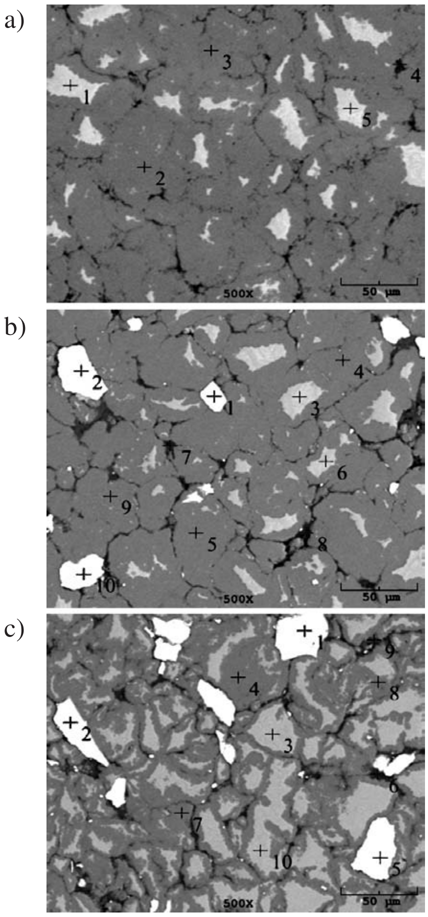


Figure 3: SEM micrographs of the sintered: a) A1, b) B1, c) B2 samples
Slika 3: SEM-posnetki sintranih vzorcev: a) A1, b) B1 in c) B2



a)

A1 Marks	Elements		
	w/%		
	Ti	Al	O
1	100	–	–
2	31	69	–
3	32	68	–
4	13	61	26
5	100	–	–

b)

B1 Marks	Elements			
	w/%			
	Ti	Al	O	Nb
1,2,10	–	–	–	100
3,6	100	–	–	–
4,5,9	31	69	–	–
7	25	67	8	–
8	17	55	28	–

c)

B2 Marks	Elements			
	w/%			
	Ti	Al	O	Nb
1,2,5	–	–	–	100
3,10	100	–	–	–
4,7,8	32	69	–	–
6	27	65	8	–
9	20	65	15	

Figure 4: SEM-EDS micrographs of the sintered: a) A1, b) B1, c) B2 samples
Slika 4: SEM-posnetki in EDS-analize sintranih vzorcev: a) A1, b) B1 in c) B2

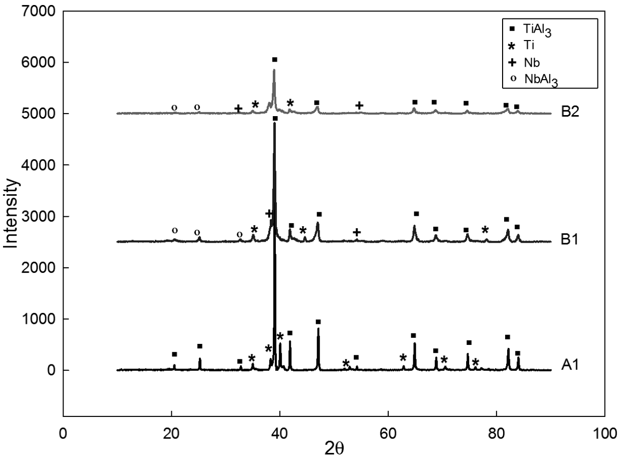


Figure 5: XRD pattern of the sintered A1, B1 and B2 samples
Slika 5: XRD-posnetki sintranih vzorcev A1, B1 in B2

phases. It can also be seen that the composite includes many small pores. And some pores are present among the grain boundaries. In the case of **Figures 3b** and **3c** we can easily notice that the Nb phase is the lightest in colour.

As it can be seen in **Figure 4,** the SEM-EDS analysis was conducted at different points on each sample of the A1, B1 and B2 compositions. In the case of A1, besides the Ti metallic and $TiAl_3$ intermetallic phase, black line areas also contain some oxygen, titanium and aluminum. The intermetallic phase has a composition of $w = 69$ % Al and $w = 31$ % Ti which corresponds to the $TiAl_3$ compound in the Ti-Al system, whereas the B1 and B2 composites contain Ti, $TiAl_3$ and Nb phases and a small amount of oxygen as expected.

3.3 XRD analyses

As seen in **Figure 5,** the main phases in the composites are Ti and $TiAl_3$. In the A1 sample there is no other phase in addition to Ti and $TiAl_3$. On the other hand, in the case of B1 and B2, as expected, a small amount of the Nb phase is detected. Besides, it is observed that the main $TiAl_3$ phase decreased when compared with the A1 XRD pattern. Also, it is detected that a new aluminide ($NbAl_3$) formed with an addition of Nb to the composite.

3.4 Hardness

The hardness values of the samples for A1, B1, B2 are (759, 669 and 600) HV, respectively. As a result of forming and raising the amount of ductile niobium phase from $w = 5$ % to $w = 10$ %, the hardness of the metallic-intermetallic compound decreased from 669 HV to approximately 600 HV due to a decreasing amount of the intermetallic $TiAl_3$ phase. Different hardness values for different production conditions indicate a strong effect of microstructural variations on the mechanical behavior.[10] The decrease in the hardness values of the composites can be easily attributed to forming and increasing the ductile niobium-phase concentration.

4 CONCLUSIONS

The following results can be derived from the present study:

- Ti-$TiAl_3$ and Nb-Ti-$TiAl_3$ in-situ composites were manufactured successfully applying the one-step electric-current-activated/assisted sintering method for 20 min in a steel mould without using any inert medium (gas or vacuum).
- The presence of the Ti and $TiAl_3$ phases were verified with an XRD and SEM-EDS analysis.
- All the composites produced have remarkably high hardness values such as 759 HV.
- The hardness of the composites decreased from 759 HV to 600 HV with a formation of the $w = 10$ % Nb ductile phase.

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