

In Situ Formation of Ti-TiAl₃ Metallic-Intermetallic Composite by Electric Current Activated Sintering Method

T. YENER*, S.C. OKUMUS, S. ZEYTIN

Sakarya University, Engineering Faculty, Department of Metallurgy and Materials Engineering,
Esentepe Campus, Serdivan, Sakarya, Turkey

In this study we have investigated fabrication of in situ metallic- intermetallic Ti-TiAl₃ composites from powder mixture containing 40 wt % Ti-Al, 50 wt % Ti-Al and 60 wt % Ti-Al by electric current activated sintering method. Powder mixtures without additive were compressed uniaxially under 130 MPa of pressure and sintered 2000 A current for 20 minutes in a steel mould. Microstructures of sintered samples were investigated by optic and scanning electron microscopes, phases in samples were analyzed by XRD and their hardness was measured by Vickers hardness tester. Optic and scanning electron microscopes investigations showed that microstructures of samples were consisting of two components: Main component was titanium aluminide and other was metallic titanium. Besides this there was a trace amount of aluminium oxide in the sintered body. XRD analyses also demonstrated that main phase is TiAl₃. It was determined that as weight percentage of titanium in the mixture was decreasing, also the amount of metallic titanium has decreased in the sintered body. Additionally, average hardness values of samples were about 500 HV

DOI: [10.12693/APhysPolA.127.917](https://doi.org/10.12693/APhysPolA.127.917)

PACS: 81.05.Zx, 81.20.Ev

1. Introduction

Ti-Al intermetallic compounds, especially the stoichiometric compounds like TiAl₃, TiAl(γ) and Ti₃Al(α_2), are a topic of current research owing to their excellent properties, such as relatively high specific stiffness, high melting point and good oxidation resistance at high temperature [1–3]. Among these compounds, TiAl₃ has the lowest density of 3.4 g/cm³, the highest microhardness of 465–670 kg/mm² and the best high temperature oxidation resistance at temperatures up to 1000 °C [1–4]. Therefore, they are being considered as potential alterna-

tive of Ti-based super alloys. Titanium-based alloys find extensive applications in aerospace, chemical, metallurgical, papermaking and medical industries. Such applications require not only excellent mechanical properties, but also high temperature resistance [2]. For harnessing these materials in industrial applications, an extensive characterization of microstructure and mechanical properties as well as oxidation behavior, based on the processing route, is required to ensure a reliable performance [5, 6]. Limited ductility and toughness values are the critical hindrances in the employment of these intermetallics in high temperature structural applications. During the development and research of high temperature structural intermetallics, much attention has been paid to the Ti-Al and Ni-Al systems. However, relatively low plasticity is a major impediment in the employment of these materials [7, 8]. Considerable research is being carried out in the

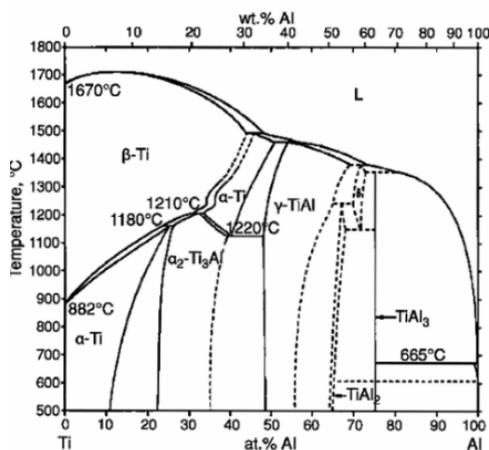


Fig. 1. Ti-Al phase diagram [11].

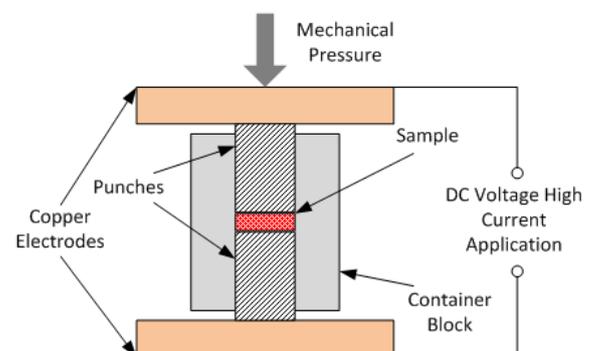


Fig. 2. Schematic representation of Electric Current Assisted Sintering (ECAS) process.

corresponding author; e-mail: tcerezci@sakarya.edu.tr

framework of alloying strategies to transform the brittle non-cubic Ti–Al compounds, such as D022-TiAl₃ into the cubic L12 structure, by using additives [9]. Manufacturing of metallic-intermetallic compounds can be customized, using different proportions of the constituent materials, to get an optimized set of properties and benefits of the apportioned elements such as strength, stiffness and toughness of the intermetallic phases. Particle reinforcement with fibers, rods or layers of ductile elements can be carried out to improve toughness. Laminate interactions can be utilized, through ductile phase reinforcement of brittle materials, to form a zone of bridging elements causing inhibition of crack opening resulting in increased fracture resistance of the composite materials [10]. The goal of the present study is to manufacture titanium-titanium aluminide metallic- intermetallic composites from powder mixture in stoichiometric ratio according to Ti-Al phase diagram as shown in Fig. 1, to investigate the resulting microstructure and the corresponding properties.

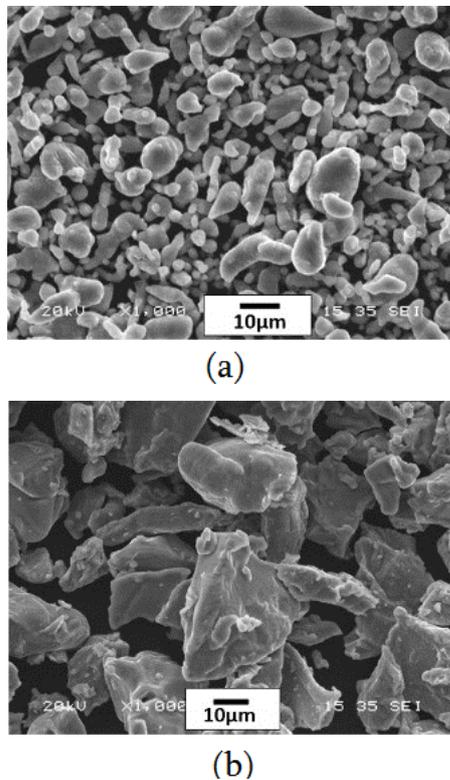


Fig. 3. SEM micrographs of the elemental powders: (a) Al, (b) – Ti powder.

2. Eperimental Procedure

Metallic Ti powders which has a purity of 99.5% and finer than 44 μm , metallic Al which has 99.5% purity, finer than 25 μm were used, as starting powder mixture for the formation of TiAl₃-Ti intermetallic–metallic in situ composite. The powder mixture were prepared in

three different compositions which were 40wt% Ti–Al, 50 wt% Ti– Al and 60wt% Ti–Al. In order to provide contiguity of individual powders at the beginning of the process, samples were compacted in a die with the load of 130 MPa, for 1 minute. 2000–2200 A, 0.9-1.1 Volt DC was applied to steel substrate with a pressure of 55 MPa for 20 minutes via Electric Current Assisted Sintering (ECAS), as shown in Fig. 2. After sintering in normal atmospheric conditions, specimens were unloaded and cooled to room temperature. The morphologies of the samples and the presence of the phases formed were examined by optical microscopy and with scanning electron microscopy (SEM-EDS). Also X-ray diffraction (XRD) analysis us-

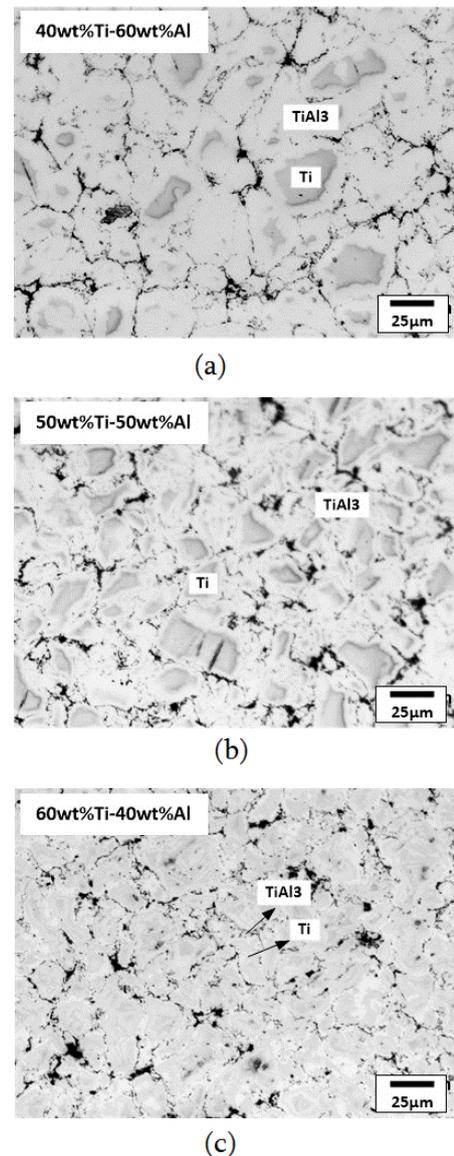
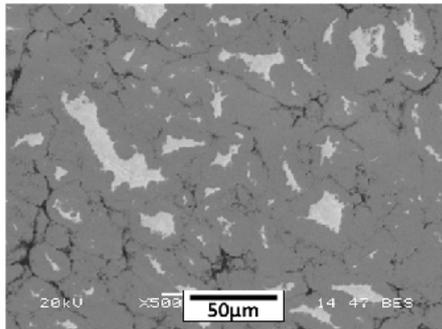


Fig. 4. Optic micrographs of the sintered samples (a) 40wt%Ti–Al, (b) 50wt%Ti–Al, (c) 60wt%Ti–Al.

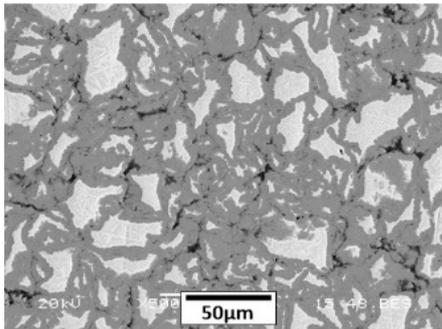
ing CuK α radiation with a wavelength of 1.5418 Å over a 2 θ range of 10–80 were done. The micro-hardness of the test materials was measured using a Vickers indentation technique with a load of 0.98 N using Leica WMHT-Mod model Vickers hardness instrument.

3. Results and Discussion

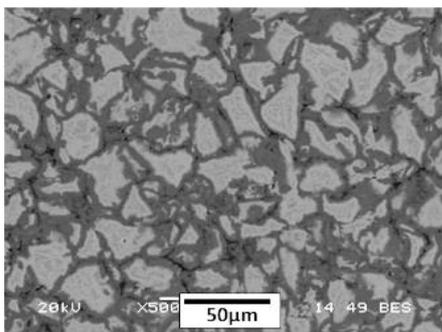
Figure 3 shows the morphologies of as-received Al, and Ti powders. Aluminum powder particles were spherical in general, within the 10–20 μ m diameter range. Whereas Titanium powders has sharp corners and was finer than 40 μ m in size. In ECAS method, an electric current is simultaneously applied with a mechanical pressure to



(a)

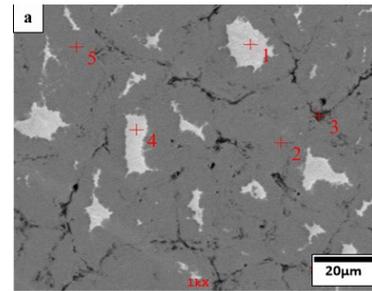


(b)

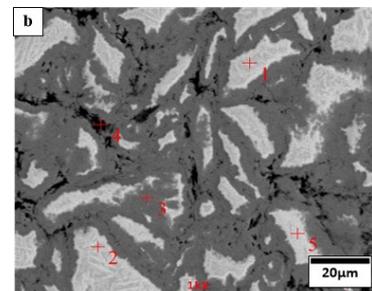


(c)

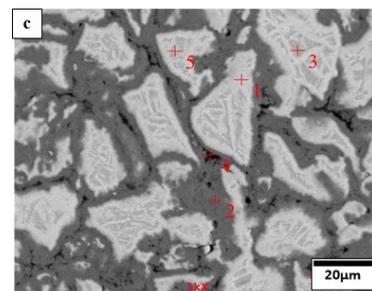
Fig. 5. SEM micrographs of sintered samples (a) 40wt%Ti-Al, (b) 50wt%Ti-Al, (c) 60wt%Ti-Al.



Marks	Elements		
	wt%		
	Ti	Al	O
1	100	-	-
2	30,8	69,1	-
3	15,16	55,5	29,3
4	100	-	-
5	30,8	69,1	-



Marks	Elements		
	wt%		
	Ti	Al	O
1	100	-	-
2	99,6	0,3	-
3	39,7	60,2	-
4	14,5	57,2	28,1
5	100	0	-



Marks	Elements		
	wt%		
	Ti	Al	O
1	100	-	-
2	31,23	68,7	-
3	99,3	0,62	-
4	18,3	57,91	23,7
5	99,8	0,12	-

Fig. 6. SEM-EDS analysis of sintered samples (a) 40 wt% Ti-Al, (b) 50wt % Ti-Al, (c) 60wt% Ti-Al.

consolidate or synthesis and to densify specific products into the desired configuration and density [1, 2].

Optical micrographs of samples are shown in Fig. 4. It can be seen three distinct area from the micrographs. One of these areas was titanium, like intense color small islands. Matrix, the main phase was $TiAl_3$. Also there was a small amount of pores, seen from the microstructure. The higher the aluminum content, the higher the intermetallic phase $TiAl_3$, can be figured out from the micrographs.

The microstructures of Ti- $TiAl_3$ metallic-intermetallic composites are also depicted in SEM micrographs (Fig. 5) taken in BSE mode.

As it can be seen in Fig. 6, SEM-EDS analysis were conducted at five points on average, on each sample for all compositions. Besides Ti metallic and $TiAl_3$ intermetallic phases, black line areas also contain some oxygen, titanium and aluminium in the composite. Intermetallic phase has a composition of 69 wt% Al and 31wt% Ti which corresponds to $TiAl_3$ compound in Ti-Al system (Fig. 1).

The main phases in all composites are Ti and $TiAl_3$, as exhibited in XRD graph, can be seen in Fig. 7. A slight amount of oxygen detected in the XRD analysis of samples probably arisen from the reaction of aluminum powders with oxygen in open atmosphere.

The hardness values of samples are 605 HV, 580HV, 450 HV for wt40%Ti-Al, wt50%Ti-Al, wt60%Ti-Al composites, respectively. Hardness of intermetallic composite decreased from 607HV to approximately 450HV as the titanium content increased from 40% to 60% in weight.

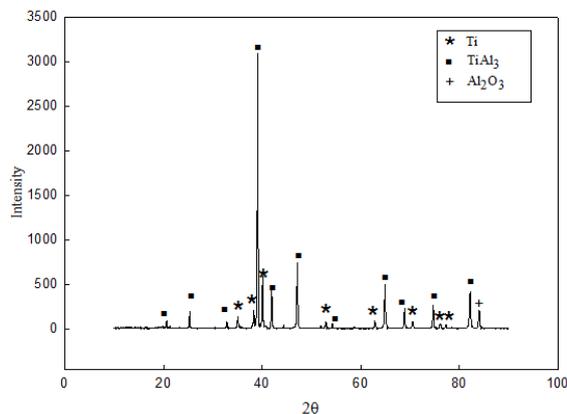


Fig. 7. XRD pattern of sintered 40 wt%Ti-Al sample.

4. Conclusion

Ti- $TiAl_3$ in-situ composites were manufactured successfully by one-step electric current activated/assisted sintering (ECAS) method in 20 minutes in a steel mold without using any inert gas or vacuum medium. The presence of Ti and $TiAl_3$ phases were verified by XRD

and SEM-EDS analysis. All of the composites produced, have remarkable high hardness values as much as 605 HV. Hardness of the composites was decreased with increasing amount of Ti, since ductile titanium phase raised.

Acknowledgments

The authors kindly thank to Research Assistant Shafaqat Siddique from Dortmund Technical University for his great contributions. This work was supported by Sakarya University Scientific Research Foundation (Project number: 2013-01-08-034)

References

- [1] P.Y. Wang, H.J. Li, L.H. Qi, X.H. Zeng, H.S. Zuo, *Progress in Natural Science: Materials International* **21**, 153 (2011).
- [2] K. Zhang, Z. Li, W. Gao, *Materials Letters* **57**, 834 (2002).
- [3] Y.B. Sun, Y.Q. Zhao, D. Zhang, C.Y. Liu, H.Y. Diao, C.L. Ma, *Transactions of Nonferrous Metals Society of China* **21**, 1722 (2011).
- [4] T. Matsubara, T. Shibutani, K. Uenishi, K.F. Kobayashi, *Materials Science and Engineering: A* **329**, 84 (2002).
- [5] J. Geng, G. Gantner, P. Oelhafen, P. Datta, *Applied Surface Science* **158**, 64 (2000).
- [6] S. Vaucher, M. Stir, K. Ishizaki, J.M. Catala-Civera, R. Nicula, *Thermochimica Acta* **522**, 151 (2011).
- [7] H. Xu, L. Meng, J. Xu, Y. Li, X. Zhao, *Intermetallics* **15**, 778 (2007).
- [8] R. Reddy, A. Yahya, L. Brewer, *Journal of alloys and compounds* **321**, 223 (2001).
- [9] Y. Duan, Y. Sun, L. Lu, *Computational Materials Science* **68**, 229 (2013).
- [10] L. Peng, H. Li, J. Wang, *Materials Science and Engineering: A* **406**, 309 (2005).
- [11] Z.C. Schuster, M. Palm, *Basic and Applied Research: Section 1* **27**, 255 (2006).
- [12] R. Orru, R. Licheri, A.M. Locci, A. Cincotti, G. Cao, *Materials Science and Engineering: R: Reports* **63**, 127 (2009).