

Special issue of the 3rd International Conference on Computational and Experimental Science and Engineering (ICCESSEN 2016)

Production and Characterization of Niobium Toughened Ti-TiAl₃ Metallic-Intermetallic Composite

T. YENER* AND S. ZEYTIN

Sakarya University, Metallurgy and Materials Department, Sakarya, Turkey

Ti-TiAl₃ in situ composites with 10 wt.% Nb were successfully prepared from Ti, Al, and Nb metallic powders by powder metallurgy processing technique of electric current activated/assisted sintering. The current and process time used for producing metallic-intermetallic composites were 2000 A and 90 s, respectively. In terms of fracture toughness, effects of addition of ductile niobium phase to Ti-TiAl₃ composites were investigated. According to SEM-EDS and XRD results, the synthesized composites mainly consisted of TiAl₃ matrix and dispersive Nb reinforcing phases, as well as ductile Ti phases. Hardness and fracture toughness values of the samples were measured by Vickers hardness tester under loads of 100 g and 10 kg, respectively. Fracture toughness value of TiAl₃ intermetallic composites was increased with Nb ductile phase addition from $1.69 \pm 0.05 \text{ MPa m}^{1/2}$ to $5.23 \pm 0.3 \text{ MPa m}^{1/2}$.

DOI: [10.12693/APhysPolA.132.941](https://doi.org/10.12693/APhysPolA.132.941)

PACS/topics: 46.50.+a, 72.80.Tm

1. Introduction

Intermetallics are materials which have a sequential order of mixed atomic species of metal, metal or metal-semimetal species with nearly stoichiometric composition, such as for example, Ni₃Al, FeAl, TiAl, MoSi₂. Here, nickel (Ni), iron (Fe), titanium (Ti) and molybdenum (Mo) play the role of metallic component and aluminum (Al) or silicon (Si) of metal/semimetal. In such cases, the metal-to-metal or metal-to-semimetal association is partially metallic and partially covalent (or ionic) in nature. There are also important intermetallic compounds of the metal-metal combinations, which are especially responsible for the atomic size differences that are unregulated for the Laves phases [1–3].

Among the intermetallics, titanium aluminide intermetallic compounds have received considerable attention recently due to their exceptional properties, such as high yield strength at elevated temperatures, improved creep properties, high oxidation and corrosion resistance, low density and good dimensional stability [4–7]. However, the biggest problem with these materials is the lack of ductility at conventional production temperatures [8]. Low room temperature ductility limits the use of intermetallics and leads to their production by costly production methods [6, 8–11].

In this study, we aimed to increase the room temperature ductility by producing metallic reinforced composite with intermetallic matrix. Unlike the previous studies, we have produced Ti-TiAl₃ composite with in situ production technique and reinforced ductility by reinforcing it with Nb. Phases were determined from XRD following the manufacturing process. Composites were analyzed by XRD technique using Cu K_α radiation with

a wavelength of 1.5418 Å [12], in order to determine the phases formed in the body of the composite. Surfaces of materials were analyzed by SEM. The EDS analysis revealed how much of the element there was in the sample (weight percent). The Archimedes principle was used for density determination and the Vickers hardness test for hardness measurements. We have applied a load of 98 N for the fracture toughness measurement and determined the fracture toughness value using the Evans Charles equation.

2. Materials and methods

Powder materials of titanium (99.5% purity, 35–44 μm), aluminum (99.8% purity, 35–44 μm) and niobium (99.5% purity, 35–44 μm) were used as starting materials for manufacturing of Ti-Nb-TiAl₃ metallic intermetallic compound. Ti, Al and Nb powders were mixed for 30 minutes. After ball milling process, powder mixture was cold-pressed before sintering, to form a cylindrical compact in a metallic die under a uniaxial pressure of 200 MPa. Dimensions of the compact were 15 mm diameter and 5 mm thickness. The production of intermetallic compound was performed via electric current activated sintering technique in an open atmosphere at 2000 A, for 90 s. Process parameters are listed in Table I.

TABLE I

Process parameters for the samples.

Sample code	Wt.%	Current [A]	Voltage [V]	Holding time [s]
R (Reference)	38Ti-62Al	2000	0.9–1.2	120
C-10Nb	45Ti-45Al-10Nb	2000	0.9–1.2	90

3. Characterization

The morphologies of the samples and the presence of the phases formed were examined by scanning electron

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

microscopy (SEM-EDS). X-ray diffraction (XRD) analysis, using Cu K_{α} radiation with a wavelength of 1.5418 Å over a 2θ range of 10–80°, was also carried out. The micro-hardness of the test materials was measured using Vickers indentation technique with a load of 0.98 N using Leica WMHT-Mod model Vickers hardness measurement instrument. Evans-Charles equation [13–15] was used for fracture toughness calculations.

$$K_{Ic} = 0.0824 \frac{P}{c^{3/2}}, \quad (1)$$

where K_{Ic} is the fracture toughness, P is the load and c is the crack length. Crack lengths were immediately measured by an optical micrometer attached to the optical microscope. Tests were repeated three times under the same conditions to ensure the reproducibility of the fracture toughness data.

4. Results and discussion

4.1. SEM-EDS analyses

The morphologies of as-received Ti, Al and Nb powders are shown in Fig. 1. The metallic Al powder particles were generally spherical with a diameter of 10 μm in size. Some of the particles are agglomerated, however are finer than 35–40 μm . In contrast, the Ti and Nb powder grains had sharp irregular corners and were finer than 40 μm in size.

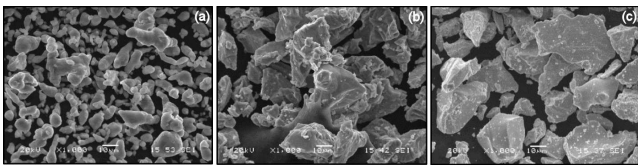


Fig. 1. SEM micrographs of (a) Al, (b) Ti, (c) Nb powders.

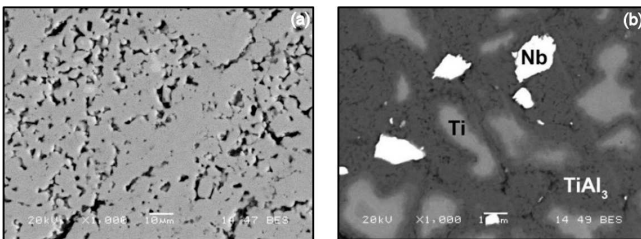


Fig. 2. SEM micrographs of sintered (a) TiAl_3 (R), (b) Ti-Nb- TiAl_3 (C-10Nb).

SEM micrograph of the sintered TiAl_3 sample produced during 120 seconds at 2000 A, using the starting powders of Ti and Al, is shown in Fig. 2a. The image of the Ti-Nb- TiAl_3 sample, which was produced by supplementing 10 wt.% Nb to the reference composition is shown in Fig. 2b. The conversion of TiAl_3 is expected to take 120 seconds to complete, while the ductile phase effect is considered sufficient for the desired titanium phase to remain in the structure for 90 seconds. Nb, the reinforcing phase, remained visible as a bright white phase.

TiAl_3 phase was determined as the predominant phase, while composites were seen as titanium light gray islets. SEM-EDS analyses also support these results (Fig. 3).

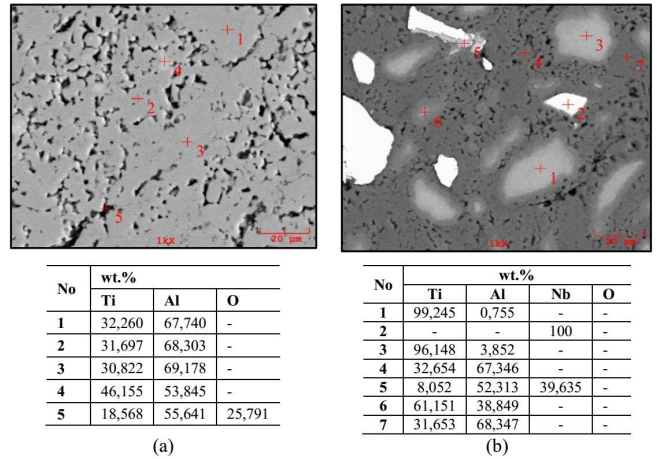


Fig. 3. SEM-EDS analyses of (a) TiAl_3 (R), (b) Ti-Nb- TiAl_3 (C-10Nb).

4.2. X-ray analysis

The main phase in R composites is TiAl_3 , whereas C-10Nb composite contains Nb, Ti and TiAl_3 , as shown in XRD graph in Fig. 4. A small amount of oxygen is also detected from XRD analyses.

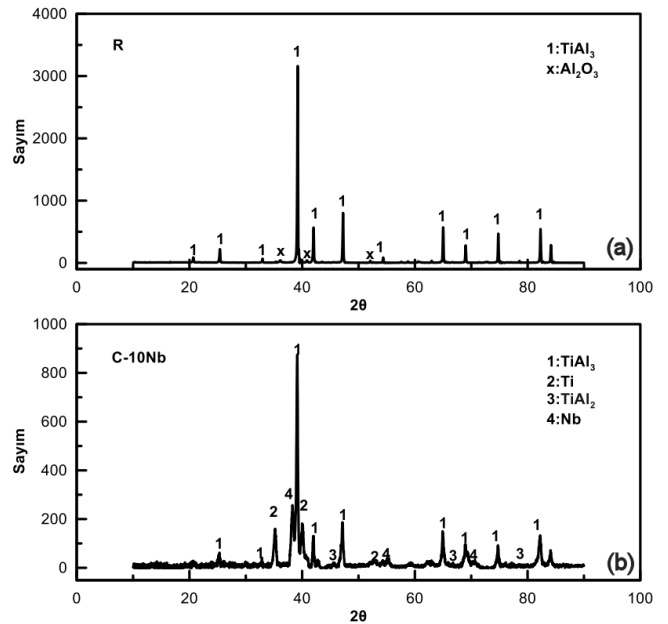


Fig. 4. XRD analyses of (a) TiAl_3 (R), (b) Ti-Nb- TiAl_3 (C-10Nb).

4.3. Hardness and fracture toughness

The hardness values of samples were 460 ± 25 and 380 ± 45 HV for R and C-10Nb respectively. With the increasing metallic phase the hardness of the composites has decreased from 460 HV to 380 HV (Table II).

This decrease in hardness is acceptable. When it comes to fracture toughness values of the samples an increment from 2.15 to 5.23 MPa m^{1/2} can be seen. A two times increase in fracture toughness with niobium reinforcement has been obtained.

TABLE II

Hardness and fracture toughness values of R and C-10Nb samples.

Sample	Hardness [HV]	Fracture toughness [MPa m ^{1/2}]
R	460 ± 25	2.15 ± 0.05
C-10Nb	380 ± 45	5.23 ± 0.3

5. Conclusions

- Nb-Ti-TiAl₃ in-situ composites were manufactured successfully by one-step electric current activated/assisted sintering method during 90 seconds in a steel mould without using any inert gas or vacuum medium.
- The presence of Ti, Nb and TiAl₃ phases were verified by XRD and SEM-EDS analysis.
- TiAl₃ intermetallic sample, produced during 120 seconds, has remarkably high hardness values, as much as 460 HV, whereas ductile-phase-reinforced Nb-Ti-TiAl₃ composite has hardness value of 380 HV.
- A two times increase in the fracture toughness values has been obtained with niobium reinforcement.

Acknowledgments

The authors thank Prof. Dr. Cuma Bindal and expert Fuat Kayis for performing XRD and SEM-EDS studies and special appreciation is expressed to technicians Ersan Demir and Erkut Tas of Sakarya University for assisting with experimental studies.

References

- [1] D. Morris, M. Munoz-Morris, *Revista de Metalurgia* **41**, 498 (2005).
- [2] N. Ergin, O. Ozdemir, *Acta Phys. Pol. A* **127**, 1103 (2015).
- [3] N. Ergin, G. Yoruk, O. Ozdemir, *Acta Phys. Pol. A* **123**, 245 (2013).
- [4] R. Reddy, A. Yahya, L. Brewer, *J. Alloys Comp.* **321**, 223 (2001).
- [5] J. Sienkiewicz, S. Kuroda, R.M. Molak, H. Murakami, H. Araki, S. Takamori, K.J. Kurzydłowski, *Intermetallics* **49**, 57 (2014).
- [6] S. Vaucher, M. Stir, K. Ishizaki, J.M. Catala-Civera, R. Nicula, *Thermochim. Acta* **522**, 151 (2011).
- [7] T. Wang, J. Zhang, *Mater. Chem. Phys.* **99**, 20 (2006).
- [8] M. Mirjalili, M. Soltanieh, K. Matsuura, M. Ohno, *Intermetallics* **32**, 297 (2013).
- [9] T. Marr, J. Freudenberger, A. Kauffmann, J. Romberg, I. Okulov, R. Petters, J. Scharnweber, A. Eschke, C.-G. Oertel, U. Kühn, J. Eckert, W. Skrotzki, L. Schultz, *Metals* **3**, 188 (2013).
- [10] P.-Y. Wang, H.-J. Li, L.-H. Qi, X.-H. Zeng, H.-S. Zuo, *Prog. Nat. Sci.: Mater. Int.* **21**, 153 (2011).
- [11] T. Yener, S.C. Yener, S. Zeytin, *Mater. Tehnol.* **50**, 899 (2016).
- [12] T. Yener, I. Altinsoy, S.C. Yener, G.F. Celebi Efe, I. Ozbek, C. Bindal, *Acta Phys. Pol. A* **127**, 1045 (2015).
- [13] E. Ercenk, U. Sen, S. Yilmaz, *Surf. Coat. Technol.* **232**, 703 (2013).
- [14] E. Ercenk, U. Sen, S. Yilmaz, *Tribol. Int.* **52**, 94 (2012).
- [15] A.S. Demirkiran, S. Yilmaz, U. Sen, *J. Ceram. Proc. Res.* **14**, 51 (2013).