

# Fast Production of $TiAl_3$ Intermetallic Matrix Composites

\*<sup>1</sup>Tuba Yener and <sup>1</sup>Sakin Zeytin

\*<sup>1</sup>Faculty of Engineering, Department of Metallurgy and Materials Engineering Sakarya University, Turkey  
e-mail:tcerezci@sakarya.edu.tr

## Abstract

This paper aims to provide a new approach to produce intermetallic matrix composites in a few seconds using electric current activated sintering (ECAS) methods. In conventional techniques, a lot of heat is wasted as the whole volume of space is heated and the compact indirectly receives heat from the hot environment. In this study, it was investigated the fabrication of in situ metallic intermetallic Ti- $TiAl_3$  composites from powder mixture containing 50 wt % titanium-50 wt % aluminum by electric current activated sintering method. Powder mixtures without additive were compressed uniaxially under 170 MPa of pressure and sintered with 2000 A current for 5, 15, 90 seconds in a steel mould. Microstructures of sintered samples were investigated by scanning electron microscopes, phases in samples were analysed by XRD and their hardness was measured by Vickers hardness tester. Scanning electron microscope investigations showed that microstructures of samples consisted of two components: Main component was titanium aluminide and other was metallic titanium. Also there was a trace amount of aluminium oxide in the sintered body. In addition, the average hardness values of samples were about  $HV_{0.05} = 625 \pm 166$ .

**Key words:** Intermetallics, Electric Current Activated Sintering (ECAS), Titanium aluminide

## 1. Introduction

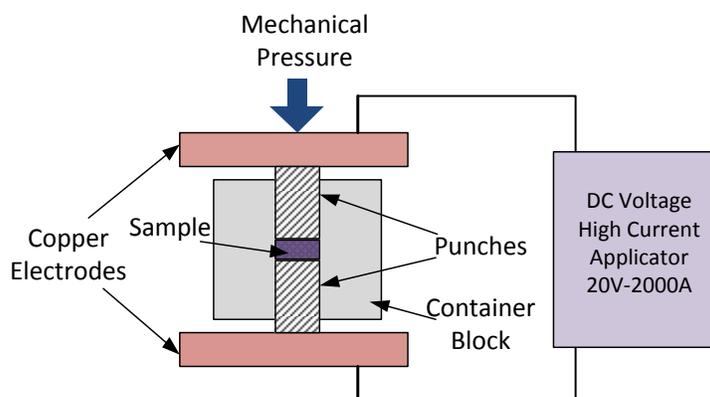
Titanium-based intermetallics are gaining more popularity due to their excellent properties. The combination of light weight and high strength makes them attractive for aerospace and automotive industries. [1, 2]. Especially, the stoichiometric compounds  $TiAl_3$  have attracted extensive attention of many researchers because of their excellent properties, such as the superior specific stiffness, high melting point, good oxidation resistance at high temperature, low density. However, the application of  $TiAl_3$  has been restricted by its limited room temperature ductility. Microalloying with transition metals, in order to increase the number of crystallographically equivalent slip systems, has not resulted in large improvements in the room temperature ductility. Recently,  $TiAl_3$  has also been considered as a composite material constituent, particularly as the reinforcing phase in an aluminum matrix. These materials have been processed by in situ synthesis methods, such as self-propagating high-temperature synthesis, mechanical alloying followed by hot pressing and reactive liquid phase sintering of elemental Al and Ti [3-5].

However, in conventional techniques, the powder container is typically heated by radiation from the enclosing furnace through external heating elements and convection of inert gases if applicable. Therefore, the sample is heated as a consequence of the heat transfer occurring by conduction from the external surface of the container to the powders. The resulting heating rate is

\*Corresponding author: Address: Faculty of Engineering, Department of Metallurgy and Materials Engineering Sakarya University, 54187, Sakarya TURKEY. E-mail address: tcerezci@sakarya.edu.tr, Phone: +902642955793

then typically slow and the process can last hours. In addition, a lot of heat is wasted as the whole volume of space is heated and the compact indirectly receives heat from the hot environment [6]. On the other hand, ECAS processes are characterized by the efficient use of the heat input, particularly when electrically insulating container is used and the electric current is applied for extremely short duration (down to few hundreds of microseconds) [6, 7].

The ECAS process is schematically shown in Figure 1. It simultaneously applies an electric current along with a mechanical pressure in order to consolidate powders or synthesize and simultaneously densify specific products with desired configuration and density. The applied electric current and mechanical load may be constant throughout the sintering cycle or may vary during the selected densification stages. In particular, the current may be adjusted by an automatic controller so that a prescribed temperature cycle is followed. Powders to be consolidated, which can be electrically conducting or insulating, are placed in a container (die, tube, etc.) and heated by applying the electric current. Conducting powders are heated by Joule effect and by heat transfer from the container and electrodes, while the non-conductive powders are heated only through the latter way. Conductive containers can be of graphite, ceramic, or steel [6].



**Figure 1.** Schematic of Electric Current Assisted Sintering (ECAS) process.

This paper aims to provide a new approach to produce metallic intermetallic matrix composites in a few seconds and effect of holding time on the properties of microstructure using electric current activated sintering (ECAS) methods.

## 2. Materials and Method

Powder materials from titanium (99.5 % purity, 35-44  $\mu\text{m}$ ) and aluminum (99.8 % purity, 35-44  $\mu\text{m}$ ) were used as starting materials to manufacture Ti-TiAl<sub>3</sub> metallic intermetallic compound. Ti and Al powders were mixed in stoichiometric ratio corresponding to the Ti-Al phase diagram, Figure 2. After ball milling, 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 Amper for 5, 15, 90 seconds. Process parameters are listed in Table 1.

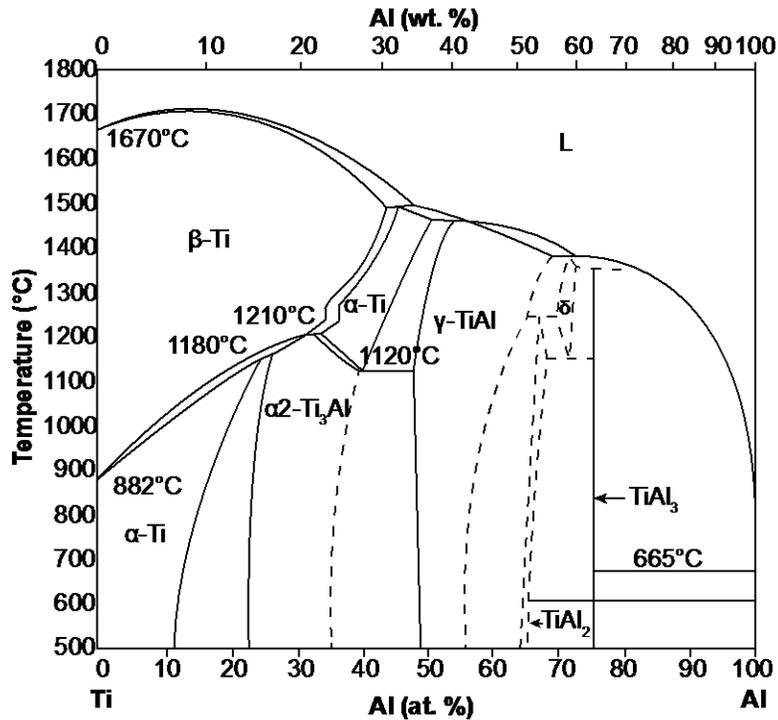


Figure 2. Ti-Al Phase Diagram [8]

Table 1. Process parameters for the samples

Sample code	Wt. %	Current (A)	Voltage (V)	Holding Time (s)
C1	50Ti-50Al	2000	0.9 - 1.2	5
C2	50Ti-50Al	2000	0.9 - 1.2	15
C3	50Ti-50Al	2000	0.9 - 1.2	90

### 3. Characterization

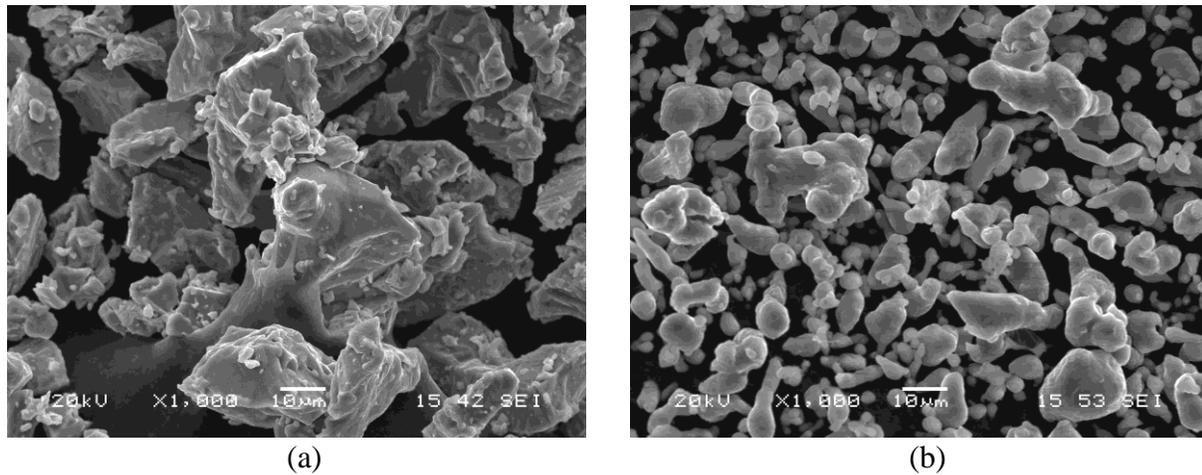
The morphologies of the samples and the presence of the phases formed were examined by scanning electron microscopy (SEM-EDS). Also X-ray diffraction (XRD) analysis using CuK $\alpha$  radiation with a wavelength of 1.5418 Å over a  $2\theta$  range of 10–80° were done. The micro-hardness of the test materials was measured using by a Vickers indentation technique with a load of 0.98 N using Leica WMHT-Mod model Vickers hardness instrument.

### 3. Results and Discussion

#### 3.1. SEM-EDS Analyses

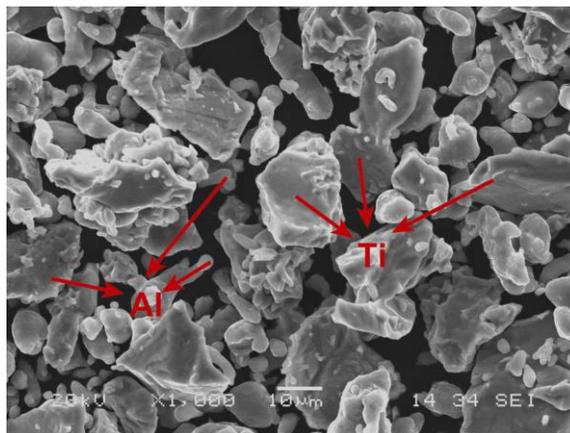
The morphologies of as-received Ti and Al powders are shown in Figure 3. The metallic Al powder particles were generally spherical with a diameter of 10  $\mu\text{m}$  in size. Some of the particles

are agglomerated however finer than 35-40  $\mu\text{m}$ . Otherwise, The Ti powder had sharp irregular corners and was finer than 40  $\mu\text{m}$  in size.



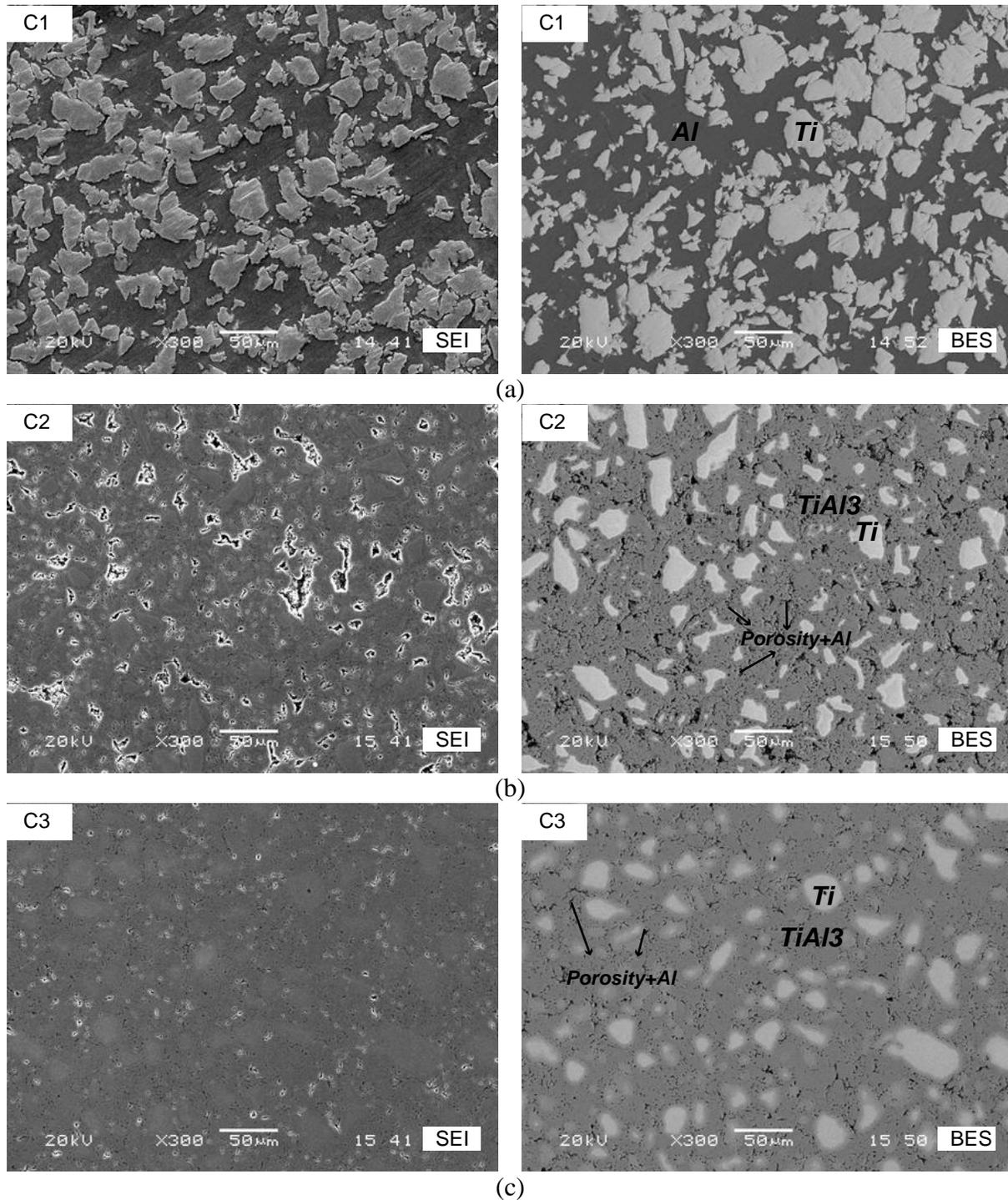
**Figure 3.** SEM micrographs of (a) Ti powder, (b) Al powder

After ball milling process, 250 rpm, for 20 minutes, the powders are mixed and homogeneous distribution of the particles can be seen in Figure 4.



**Figure 4.** SEM micrographs of Ti-Al powders after ball milling process

SEM-EDS analyses (Back Scattered Mode and Secondary Electron Mode) of C1, C2 and C3 intermetallic compounds are shown in Figure 5. The microstructure seen in Figure 5a. shows that: the shorter holding time such as 5 seconds results in separately formed Ti and Al areas. Increasing the holding time as it shown Figure 5b, it starts to form new phases like  $\text{TiAl}_3$ , but these microstructures are still far from a fully dense composition of desired Ti- $\text{TiAl}_3$  compounds. When it comes to Figure 5c, it can be easily realized that the residual aluminum and porosity amount remarkably decreased and nearly full dense microstructure has obtained for 90 seconds holding time in ECAS process.



**Figure 5.** SEM micrographs of C1, C2 and C3 composites (in the left side SEI mode, right side BES mode)

As it can be seen in Figure 6, SEM-EDS analysis were conducted at seven different points. Besides Ti metallic and TiAl<sub>3</sub> 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<sub>3</sub> compound in Ti-Al system Figure 2. This reaction

takes place completely for the Gibbs free energy of forming  $TiAl_3$  is the minimum compared with all the other type of Ti-Al intermetallic compounds.

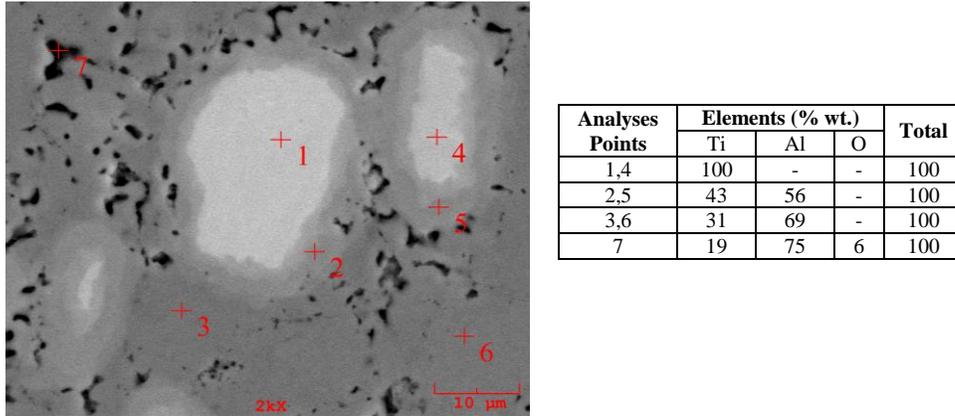


Figure 6 EDS Analysis of C3 composites

### 3.2. X-Ray Analysis

The main phases in C3 composites are Ti and  $TiAl_3$ , as exhibited in XRD graph, can be seen in Figure 7. A slight amount of oxygen detected in the SEM-EDS analysis of samples however it is not detected from XRD Analyses.

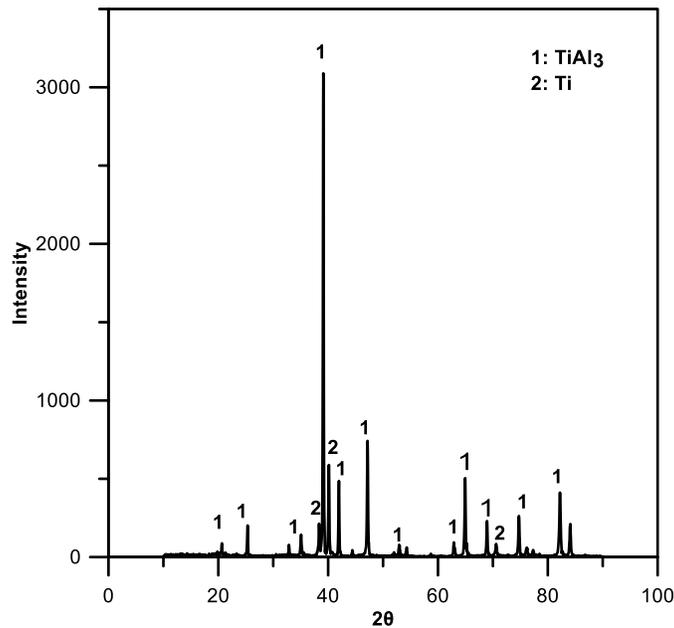


Figure 7. XRD Analyses of C3 samples

### 3.3. Hardness

The hardness values of samples were C1, C2, C3; 625±65 HV, 580±24, 115±25 respectively. Increasing with the intermetallic phase the hardness of the composites also increased from 115 HV to 625 HV. Because of the metallic Ti and Al phases in C1 (absence of intermetallic phase), the hardness values were obtained very low.

### Conclusions

- Ti-TiAl<sub>3</sub> in-situ composites were manufactured successfully by one-step electric current activated/assisted sintering (ECAS) method in 90 seconds in a steel mold without using any inert gas or vacuum medium.
- The presence of Ti and TiAl<sub>3</sub> phases were verified by XRD and SEM-EDS analysis.
- Produced composites in 90 seconds, have remarkable high hardness values as much as 625HV. Hardness of the composites were decreased with reducing with holding time, since ductile absence of intermetallic phase.

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