Technical Sciences
Academy of Romania
www.jesi.astr.ro

Journal of Engineering Sciences and Innovation
Volume 4, Issue 2 / 2019, pp. 169 - 174

B. Chemical Engineering, Materials Science and Engineering

Received ***5 February 2019*** *Accepted* ***30 May 2019***
Received in revised form ***15 May 2019***

Study and research on obtaining porous materials type Al_3Ti by sintering reactive

GYORGY THALMAIER, SILVIA ŞUTA, NICULINA SECHEL,
IOAN VIDA – SIMITI[*]

Technical University of Cluj-Napoca 103-105 Muncii Ave., 400641 Cluj-Napoca, Romania

Abstract. Aluminum-titanium compounds (Ti_3Al, TiAl and Al_3Ti) were identified as materials for high temperature applications in areas like automotive industry, materials for aircrafts or filter materials used at high temperatures and in corrosive environments. From these compounds, Al_3Ti has a low density and good resistance to the oxidation especially in air. This compound was obtained in the form of porous material by reactive sintering starting from elementar powders. The powder mixture was pressed at 500 MPa, and, the sintering took place at a temperature of 700 ° C for 30 minutes under vacuum. Following the reaction only the Al_3Ti compound was formed. The obtained samples had a pore size distribution in the 3-100 µm range.

Keywords: sintering reactive, intermetallic compound, porous material.

1. Introduction

Aluminum - titanium alloys and intermetallic compounds have been investigated extensively as a material for high temperature applications for more than two decades [1, 2]. Their outstanding properties such as low density, high melting temperature, good oxidation resistance at high temperatures, high specific strength and high specific stiffness are still attracting for the researchers [3].

The equilibrium diagram of the system Al –Ti containing different intermetallic compounds, between them the Al_3Ti has the best oxidation resistance combined with the lowest density [4].

It was observed that during sintering, samples obtained by pressing different mixtures of Al and Ti increased their the volumes and prevented densification. This observation can be used to generate the desired porosity in the sintered aluminum –

---

[*] Correspondence address: vida.simiti@stm.utcluj.ro

titanium samples. The increase in porosity is achieved by redistribution of the aluminum melt and titanium in contact and forming a very porous layer of Al_3Ti [5].

H.C. Yi studied the effect of heating on the reaction rate for the formation of the Al-Ti compounds and observed a significant influence of the heating rate on the overall reaction mechanism, and, the density of the product [6]. A fast heating resulted in an increased density and a more homogeneous product. At low heating rate the reaction mechanism involves liquid (Al) and solid (Ti), while at high heating rates the solid reaction mechanisms is active. On the other hand, the maximum temperature generated by the reaction does not significantly dependent on the heating rate or the composition [7].

F. Yang et al. presented the influence of the porous structure on the gas permeability of some Nb containing AlTi compound usable in microfiltration [8].

Different researches on the reactions that take place during the during the reactive synthesis are well documented in the paper of H.C. Yi [9].

Our group presented also different papers on the manufacturing of different porous materials, like Ni superalloy metallic foams using expanded polystyrene as sacrificial space holder [10] and asymmetric membranes for microfiltration obtained by sedimentation of metallic or ceramic particles [11].

This paper focuses on the study and characterization of porous Al_3Ti obtained by reactive sintering. Characterization of the resulting material was accomplished by scanning electron microscopy (SEM), X-ray diffraction (XRD) and mercury porosimetry.

2. Experimental

In order to obtain the porous material, we used stoichiometric quantities of commercially available titanium powder (purity 99.5%, particle size distribution 150-250 μm) and Al powder (purity 99%, particle size distribution 0.3 - 300 μm). After homogenization the mixture was pressed in a steel die with a diameter of 6 mm, at a pressure of 500 MPa. After pressing the samples were polished to reduce burrs that formed during forming and then were measured and weighed to determine the porosity of the green samples.

These were sintered at a temperature of 700 °C with holding time for 30 minutes under vacuum ($P \sim 10^{-5}$ torr). Sintered samples were analyzed by measuring the density by the same method as in the case of the green samples, the formed phases were identified by X-ray diffraction (XRD Schimadzu - 6000, $\lambda = 0.15418$ nm) and the morphology and pores size distribution was measured by scanning electron microscopy (JEOL JSM 5600 - LV) and mercury porosimetry (Pascal140). Particle size distribution of the powders was carried out using the analyzer (Fritsch Analysette 22 - NANOTEC).

3. Results and discussion

After sintering the samples Al_3Ti were subjected to X-ray diffraction and the result is shown in Figure 1. In this we could identify the formation after sintering of the Al_3Ti intermetallic compound, all peaks correspond to it except for one low intensity peak ($2\theta = 35.8$) that could not be identified. This is probably corresponding to an oxide phase, sometimes visible in the SEM images in low quantity and its due to the native oxide affinity of the starting metals.

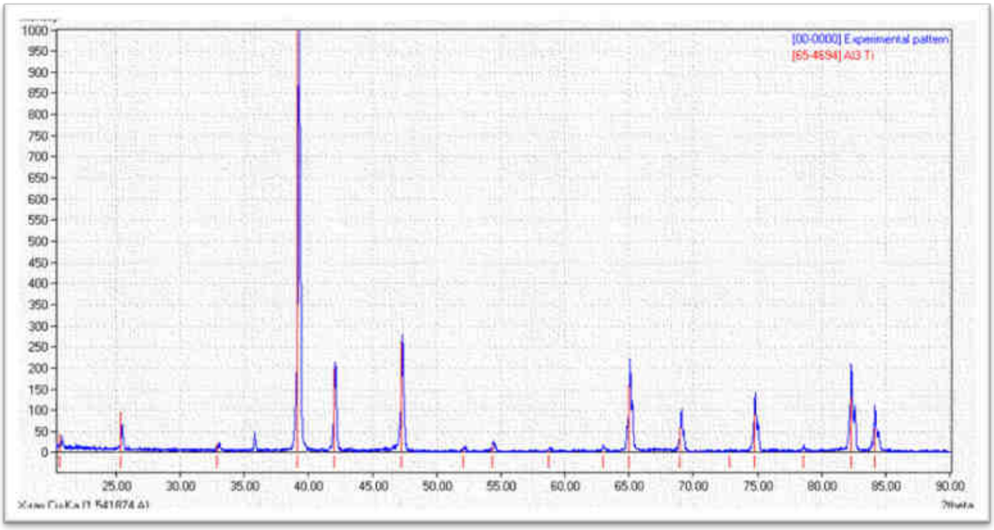

Fig. 1. Diffraction pattern of the obtained sample.

Upon reaching the melting temperature of aluminum covers the titanium particle, and by an exothermic reaction the Al_3Ti intermetallic compound is formed. The compound is lamellar with lengths in the order of 1 µm and thickness of tens of nanometers. During the reaction, aluminum diffuses into the titanium particles, increasing their volume. As a result of the diffusion in place of the aluminum particles be pores are formed. The distribution of aluminum particles directly influences the pore distribution in the obtained material.

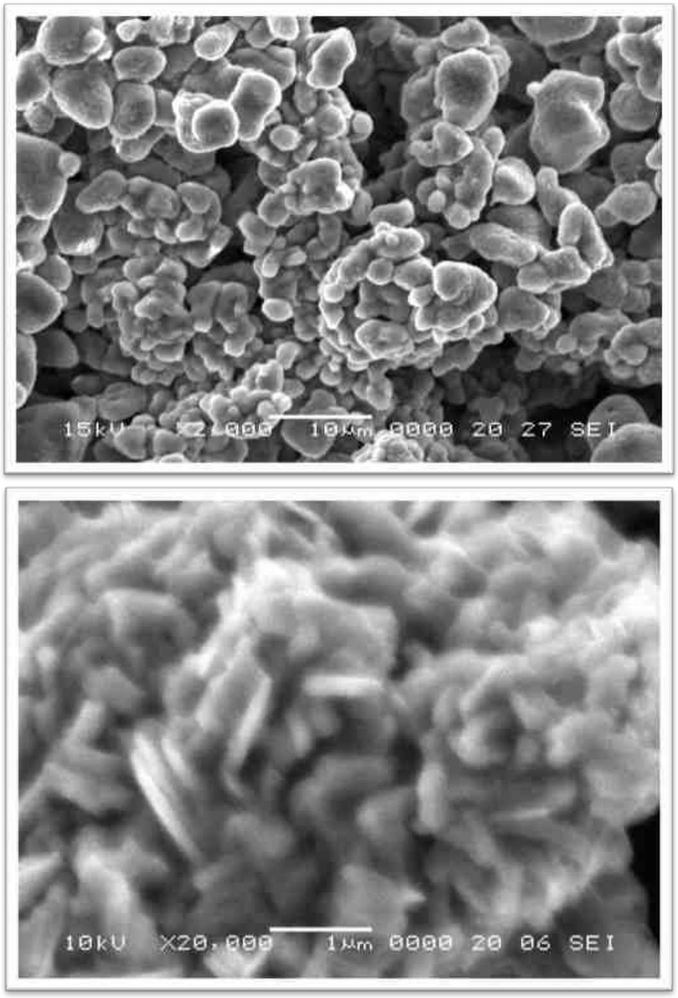

Fig. 2. SEM images of Al_3Ti alloy.

The figure 2 presents the shape and pore size of Al_3Ti alloy after sintering as evidenced by scanning electron microscopy. Figure 2a presents an overview of the structure obtained. One can observe two types of pores. Large ones, which originate from the diffusion of the aluminum in the titanium particles. Their size depends on the size of the starting Al powders, which in this case was quite high in order to increase the porosity. Fan Yang et al [8] observed the decrease in pore size with the decrease in size of the powder used, but they obtained only moderate porosity (~ 55%). The second type of pores present are small ones appeared during the formation of intermetallic compounds by fragmentation of the Al_3Ti by the molten aluminum

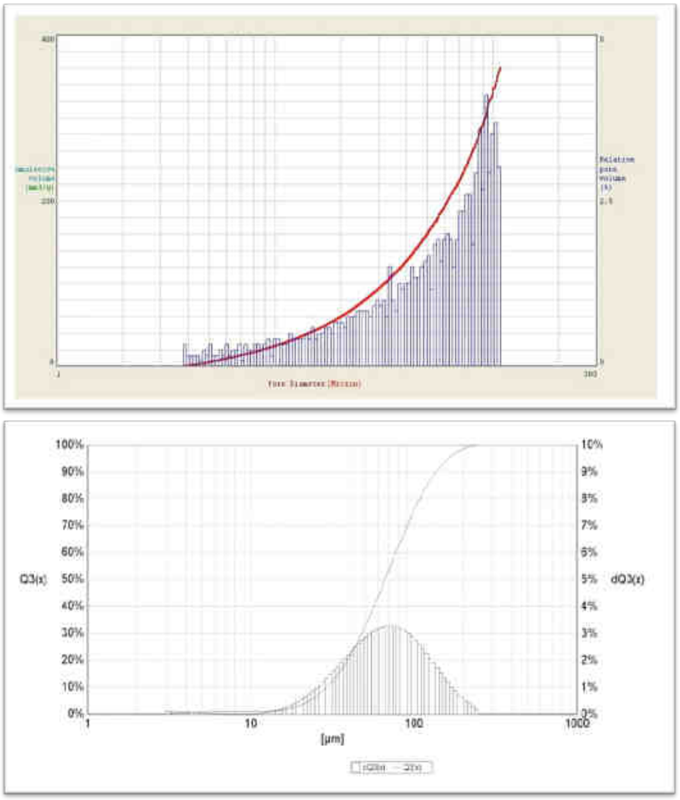

Fig. 3. The pore distribution curve and the particle size distribution of the Al powder used.

After sintering an increase of the sample size was also observed. The diameter increased from 6 to 9.5 mm and a height of 4.6 to 5.7 mm corresponding to an increase of 158% and 124% of the linear dimensions. During this step the porosity increased from 24.3% (after pressing) to 75.6% (after sintering). Y. Liang et al [12] identified two stages in which trapped the gases inside the compact produces increase in the samples volume. In the temperature range of 600-640 °C, part of the residual hydrogen present in the titanium particles is released and together with the air existing in the pores, is heated by the exothermic reaction and causes expansion of the sample. The second phase occurs after the melting of aluminum, when further hydrogen is released. This reduces the oxide layer on the powder particles and enhances the reaction. The amount of the reaction heat release is increased, and the residual gas in the pores additionally increases its volume.

Mercury porosimetry is frequently used to characterize porous materials, giving evaluation parameters such as porosity and pore size distribution, as well as their density. Figure 3 shows the pore distribution of the compound represented both Al_3Ti and distribution curve of Al powders. One can observe a continuous distribution of pores. Although at first glance the number of small pores is small, must take into account that the graph is shown the variation of the total volume of pores of certain size. From SEM images it is evident that the number of small pores is large enough and by using optimized process parameters one can obtain porous materials for use in microfiltration.

4. Conclusions

We obtained porous Al_3Ti samples by reactive sintering of aluminum to titanium powders. The porosity obtained is high, ~ 75% and the pore size distribution is a continuous one in the 3-100 µm range. The time necessary for completion of the reaction is relatively low.

Two types of pores were observed. Some are present due to diffusion of aluminum into the titanium particles and the second type that occurs due to fragmentation Al_3Ti layer on the surface of titanium particles by molten aluminum.

After the reaction intermetallic compound has a lamellar shape, small and has a sufficient strength to be used as filtering material.

References

[1]. Clemens H., Kestler H., *Processing and applications of intermetallic γ – TiAl – based alloys*, Adv Eng Mat, **2**, 2000; p. 551- 560

[2]. Zaghal S, Thomas M, Naka S, Finel A, Couret A., *Phase transformations in TiAl based alloys*, Acta Mater, **53**, 2005, p. 2653-2664.

[3]. Wu Y, Hwang Sk., *Microstructural refinement and improvement of mechanical properties and, oxidation resistance in EPM TiAl – based intermetallics with yttrium addition*, Acta Materiallia, **50**, 2002p. 1479-1493.

[4]. Mehra P.D., *Effect of mechanical milling and current intensity on the electrically activated combustion synthesis of titanium aluminides*, PhD Thesis, San Diego State University, 2013

[5]. Kupp D., Claar D., Flemming K., Waag U., Goehler H., *Novel Techniques for the fabrication of titanium aluminide thermal protection systems (TPS) for aerospace applications*, Adv Powd Metall Particulate Mat., **2**, 2002, p. 133-139.

[6]. Yi H.C., Petric A, Moore JJ., *Effect of heating rate on the combustion synthesis of Ti – Al intermetallic*, J. J. of Mater Sci, **27**, 1992, p. 6797-6806.

[7]. Sujata, M., Bhargava S., Sangal S., *On the Formation of $TiAl_3$ during Reaction between Solid Ti and Liquid Al compounds*, J. of Mater. Sci. Lett., **16**, 1997, p. 1175-1178.

[8]. Yang F., Zhang L., Lin J., Liang Y., Shang S., Liu Z. K., *Pore structure and gas permeability of high Nb – containing TiAl porous alloys by elemental powder metallurgy for microfiltration application*, Intermetall., **33**, 2013, p. 2-7.

[9] Fu E.K.Y., McShane H.B., *Reaction synthesis of titanium- aluminides*, J. of Mat. Sci., **3**, 2001, p. 5537 – 5542.

[10]. Vida-Simiti I., Thalmaier Gy., Sechel N., *Research on elaboration of metallic foams based on Ni alloy powders*, J. of Eng. Sci. and Innov., **2**, 2017, p. 70-76.

[11]. Thalmaier Gy., Sechel N., Moldovan V., Vida-Simiti I., *Gradual Porous Membranes Obtained by Powder Sedimentation*, J. of Eng. Sci. and Innov., **3**, 2018, p. 39-50.

[12]. Liang Y., Yang F., Zhang L., Lin J., Shang S., Z. K. Liu, *Reaction behavior and pore formation mechanism of TiAl- Nb porous alloys prepared by elemental powder metallurgy*, Intermetall., **44**, 2014, p 1-7.