Proceedings of the 4th International Congress APMAS2014, April 24-27, 2014, Fethiye, Turkey

Microstructural Characterization and Thermal Properties of Aluminium Titanate/Talc Ceramic Matrix Composites

E. Ozsoy*, U. Onen, T. Boyraz
Cumhuriyet University, Metallurgical and Materials Engineering, Sivas, Turkiye

Talc ($3MgO{\cdot}4SiO_2{\cdot}H_2O$) has excellent mechanical properties, shock and abrasion resistance, good electrical and thermal shock resistance, softness, isolation, chemical activity, heat resistance and oil absorption properties. Aluminium titanate (Al_2TiO_5) exhibit extremely good thermal shock resistance and low thermal conductivity coupled with good chemical resistance in molten metals. In the present work, Aluminium titanate/talc ceramics composites with different percentages of Al_2TiO_5 was prepared using powder metallurgy techniques. The microstructural, mechanical and thermal properties were characterized using XRD, SEM, dilatometer and hardness. Thermal shock resistance behaviour under water quenching of the as-prepared ceramics was also evaluated. Results, revealed that the addition of aluminium titanate to talc matrix improves the properties of the aluminium titanate/talc ceramics.

DOI: 10.12693/APhysPolA.127.1136
PACS: 81.05.Mh.

1. Introduction

Talc, $Mg_3Si_4O_{10}(OH)_2$, is a major hydrous metamorphic mineral. It is well known as the softest mineral, with a rating of 1 on Mohs scale of hardness. Owing to its unique mechanical and chemical qualities, it has been frequently used as an industrial material, such as a lubricant, a filler, and a pressure-transmission material in high-pressure experiments [1]. Also talc is widely used as a fine powder in several industrial products such as paper, paints, rubbers, polymers, ceramics, putties, etc. because of its inertness, whiteness, low thermal and electrical conductivity and adsorption capacity of organic substances. Talc is also used as filler in composites in order to improve the mechanical characteristics of the compound by enhancing the nucleation of the polymer and the dimensional stability of the end product. Recently, attention was paid to evaluate the possible changes of its main properties during processing because specific treatments such as grinding could modify intrinsic characteristics. The main effects described in the literature regard a progressive structural disorder and subsequent amorphization of the material leading to several changes in its physical and thermal properties [2].

Aluminium titanate ceramics (Al_2TiO_5) is a synthetic ceramic material of potential interest for many structural applications, owing to its high melting point, low thermal conductivity and excellent thermal shock resistance. However, a critical feature, which greatly limits the mechanical properties of polycrystalline Al_2TiO_5, is considerable inter granular micro cracking, which occurs due to the high thermal anisotropy of individual grains [3]. Aluminium titanate (Al_2TiO_5), generally obtained by a solid-state reaction between Al_2O_3 and TiO_2, has a very low thermal expansion coefficient (0.2×10^{-6} to 10^{-6}) low thermal conductivity (0.9–1.5 $W\,m^{-1}\,K^{-1}$), and a high thermal shock resistance. These properties make it most suitable to be used in components of internal combustion engines, as insulating coating, electronical and high temperature electrical part components, molten glass and metal thermometer components [4–6].

In this study; binary talc – aluminium titanate composites at various compositions were obtained and microstructural, physical, mechanical and thermal properties were characterized.

2. Experimental

In this study, a pure talc sample was taken from Sivas-Turkey. Talc that was used in experimental work is the carbonated type with calcite, dolomite and some copper minerals (cuprite, tenorite, native Cu) in minor quantities as gangue minerals. The ore consists of mainly 63.75% SiO_2 and 29.55% MgO and the sample was pure enough (more than 94% talc) for determinate experimental studies. Talc was calcined at 1150 °C in air for 2 h. After weighting of Al_2O_3 and TiO_2 in a mole ratio of (1:1) of aluminium titanate, the 1:1 molar ratio mixtures were dispersed in acetone and wet-milled with alumina balls of 10 mm Ø for 3 h. Al_2TiO_5 ceramics were synthesized by reaction sintering of an equimolar mixture of Al_2O_3 and TiO_2 powders. The powders were first homogenized by wet ball milling. The resulting mixtures were then sintered at 1600 °C for aluminium titanate (AT) in air for 2 h.

Then, aluminium titanate (AT) was added in weight proportions of 0, 10, 20 wt.% to the TAT mixture (hereafter these mixtures are denoted TAT0, TAT10 and TAT20 respectively). Each lot was again wet mixed according to the procedure described above. Then, mixtures we pressed at 100 MPa into 23 mm dimension spec-

*corresponding author; e-mail: ezgi_ozsoy_06@hotmail.com

imens. The compacts were sintered in air using a heating rate of 5 °C min^{-1} soaked at 1300 °C. Bulk density, porosity and water absorption of the sintered samples were measured. Micro hardness (Shimadzu, HMV) was measured on the polished surface of the samples at room temperature. At least six individual tests with a peak load of 2000 g and a loading time of 15 second were performed for each set of composites. Then, the microstructural characterization of the sintered samples was carried out using scanning electron microscopy (Leo 440). The morphological parameters of the various phases were characterized by using a semiautomatic image analyser, EDX and the formed phases were analysed by X-ray powder diffractometer (Rigaku, Dmax, IIIC) using Cu K_α radiation. Thermal expansion coefficients were measurement by using dilatometer (Anter, USA).

3. Results and discussion

Table I shows physical properties of the sintered TAT ceramics obtained for the different mixtures prepared. Both the bulk and true densities of the composites decreased with increasing aluminium titanate content. Note that the theoretical density of calcined talc is 3.047 g cm^{-3} whereas the density of Al_2TiO_5 is 3.20 g cm^{-3} [4, 5]. Also evident in Table I is that the porosity and water absorption decreased with Al_2TiO_5 content (from 0 to 20 wt.%) and sintering temperature.

Physical properties of samples sintered at 1300 °C. TABLE I

	ρ_{bulk} (g/cm^3)	ρ_{true} (gr/cm^3)	$\rho_{relative}$ (%)	Porosity (%)	w.abs. (%)
TAT0	2.018	3.047	66.21	33.79	15.22
TAT10	2.219	3.110	71.35	28.65	6.69
TAT20	2.473	3.175	77.92	22.08	1.01

Figure 1 shows representative SEM micrographs of the surface of the sintered TAT0, TAT10 and TAT20 samples. It can be seen that TAT20 appears to be denser and to have fewer pores than the sample TAT10 and TAT10 appears to be denser and to have fewer pores than the sample TAT0. Aluminum titanate is uniformly dispersed in the matrix. In as-received samples, a denser structure is observed with the increasing aluminium titanate content. The addition of aluminium titanate also influences the grain morphology, as is observed in the microstructures of the composites. The grain size is growing as the amount of aluminium titanate increases. EDX shows that microstructure of TAT20 sample used for phase analysis.

Figure 1d shows that microstructure of TAT20 sample used for phase analysis with EDX. Analyses revealed that the matrix consists of forsterite, enstatite and aluminum titanate phases: the areas A and B demonstrated forsterite, enstatite and aluminum titanate, which consist of Mg, Al, Si, O and Ti.

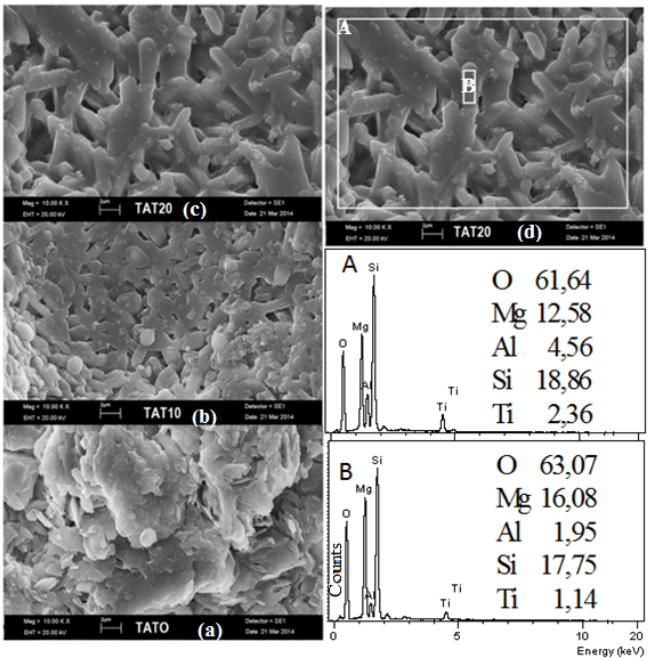

Fig. 1. SEM photomicrographs of (a) TAT0, (b) TAT10, (c) TAT20 and (d) EDX images analyses of TAT20 samples.

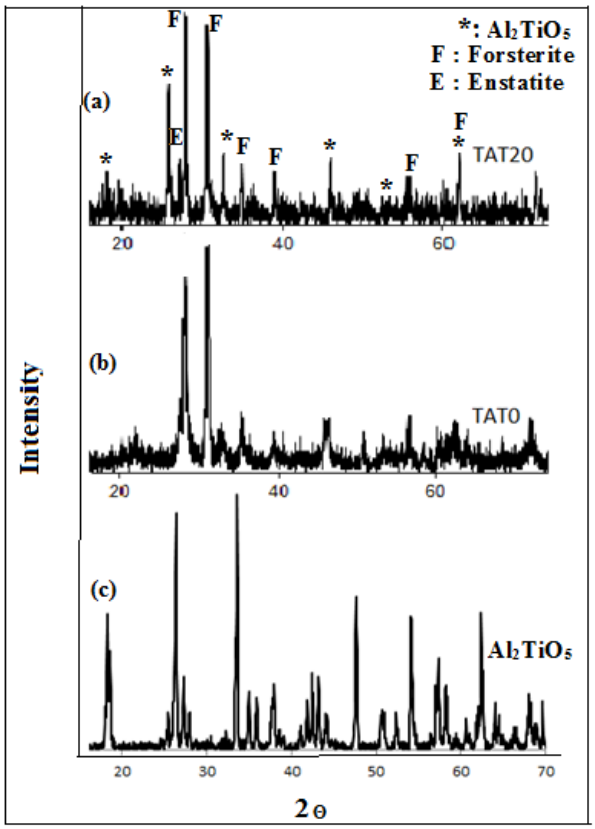

Fig. 2. X-ray diffraction patterns between 10° and 70° of samples ((a)TAT20, (b) TAT0, (c) aluminium titanate). Phases are identified by the following symbols:(E: enstatite, F: forsterite and ∗: aluminium titanate).

The X-ray diffraction patterns of the sintered samples are shown in Fig. 2. XRD analysis of the as-sintered samples revealed that main phases are forsterite (Mg_2SiO_4), enstatite ($MgSiO_3$) and aluminum titanate (Al_2TiO_5) [7].

The thermal expansion behavior of TAT0 and TAT10 ceramics is shown in Fig. 3. TAT0 has a coefficient of thermal expansion (CTE) of 2.91×10^{-6} °C^{-1} for talc in the range 25–1000 °C [6]. Al_2TiO_5 has a coefficient of thermal expansion (CTE) of 1×10^{-6} °C^{-1}. Addition of AT is observed to decrease the CTE of samples. Samples TAT0 and TAT10 have CTE of 2.91×10^{-6} °C^{-1} and 2.79×10^{-6} °C^{-1}, respectively, in the range of 25–1000 °C. It is well known that a low CTE is one of the requirements for improving thermal shock resistance at elevated temperatures.

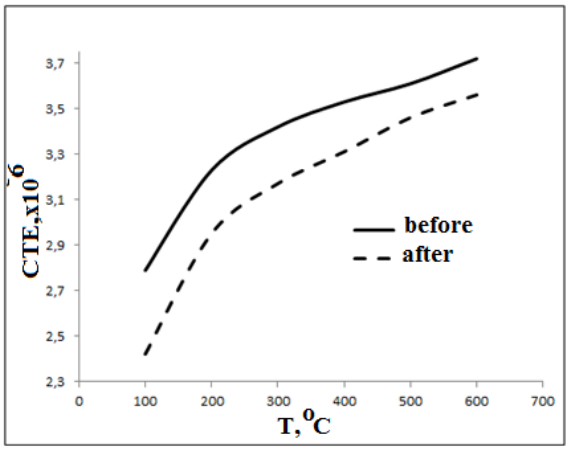

Fig. 3. Average coefficient of thermal expansion plots of TAT10 ceramics.

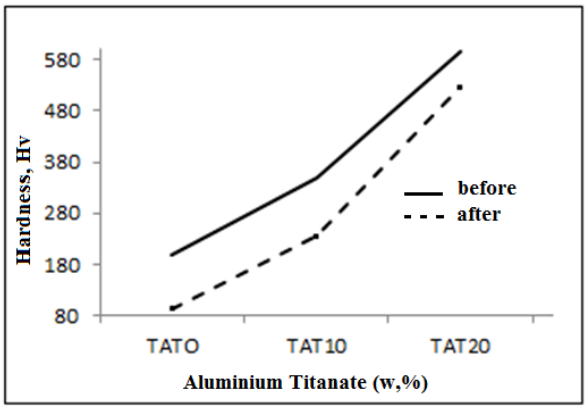

Fig. 4. Vickers micro hardness TAT0, TAT10 and TAT20 samples before and after thermal shock testing.

Figure 4 shows micro hardness of TAT0 and TAT10 samples before thermal shock testing and after thermal shock testing. Micro hardness (Shimadzu, HMV) was measured on the polished surface of the samples at room temperature. At least six individual tests with a peak load of 2000 g and a loading time of 20 s were performed for each set of composites. After thermal shock testing, micro hardness was measured again. The micro hardness increases with increasing AT content. In the case ofthe addition of 20 wt.% AT, the hardness has the biggest value (596 Hv). Therefore, increase of AT content can be beneficial to the mechanical properties, like hardness.

4. Conclusions

Aluminium titanate/talc ceramics with different percentages of Al_2TiO_5 was prepared, and its microstructural and thermal properties were characterized. A denser structure with fewer pores was observed with the increasing aluminium titanate content. The addition of aluminium titanate also influences the grain morphology as is observed in the microstructures of the composites. The grain size is growing as the amount of aluminium titanate increases. Talc-AT ceramic composites show an increased hardness with increasing AT content. Sample TAT0 has a hardness of 200 Hv, which increases to 596 Hv, on addition of 20 wt.% AT. XRD analysis of the sintered samples revealed that main phases are spinel and aluminium titanate (Al_2TiO_5). The CTE of the spinel decreased as AT was added in it. Samples TAT0 and TAT10 have CTE of 2.79×10^{-6} °C^{-1} and 2.42×10^{-6} °C^{-1}, respectively, in the range 25–1000 °C. It is well known that a low CTE is one of the requirements for improving thermal shock resistance at elevated temperatures.

References

[1] A. Yoneda, M. Yonehara, M. Osako, *Phys. Earth Planet. In.* **190-191**, 10 (2012).

[2] F. Dellisanti, G. Valdrè, M. Mondonico, *Appl. Clay Sci.* **42**, 398 (2009).

[3] H.A.J. Thomas, R. Stevens, *Br. Ceram. Trans. J.* **88**, 184 (1987).

[4] H.R. Rezaie, R. Naghizadeh, N. Farrokhnia, S. Arabi, M. Sobhani, *Ceram. Int.* **35**, 679 (2009).

[5] E. Çitak, T. Boyraz, *Acta Phys. Pol. A* **125**, 465 (2014).

[6] U. Önen, T. Boyraz, *Acta Phys. Pol. A* **125**, 488 (2014).

[7] E. Furlani , E. Aneggi, S. Maschio, *Materials* **7**, 4773 (2014).