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Enhanced properties of alumina-aluminium titanate composites obtained by spark plasma reaction-sintering of slip cast green bodies

Amparo Borrell*[1], María Dolores Salvador[1], Victoria G. Rocha[2], Adolfo Fernández[2,3], Tamara Molina[4], Rodrigo Moreno[4]

[1]Instituto de Tecnología de Materiales (ITM), Universidad Politécnica de Valencia (UPV), Camino de Vera s/n, 46022 Valencia, Spain

[2]ITMA Materials Technology, Parque Tecnológico de Asturias, 33428 Llanera (Asturias), Spain

[3]Centro de Investigación en Nanomateriales y Nanotecnología (CSIC-UO-PA), Parque Tecnológico de Asturias, 33428 Llanera, Spain

[4]Instituto de Cerámica y Vidrio (ICV), Consejo Superior de Investigaciones Científicas (CSIC), E-28049 Madrid, Spain

*Corresponding author. Address: Instituto de Tecnología de Materiales (ITM), Universidad Politécnica de Valencia (UPV), Camino de Vera s/n, 46022 Valencia, Spain. Tel.: +34 963 877 007; Fax: +34 963 877 629. E-mail address: aborrell@upvnet.upv.es (A. Borrell)

ABSTRACT

Full dense Alumina+40vol.% aluminium titanate composites were obtained by colloidal filtration and fast reaction-sintering of alumina/titania green bodies by spark plasma sintering at low temperatures (1250-1400 ºC). The composites obtained had near-to-theoretical density (>99%) with a bimodal grain size

distribution. Phase development analysis demonstrated that aluminium titanate has already formed at 1300 ºC. The mechanical properties such as Vickers hardness, flexural strength and fracture toughness of bulk composites are significantly higher than those reported elsewhere, e.g. the composite sintered at 1350 ºC show values of about 24 GPa, 424 MPa and 5.4 MPa $m^{1/2}$, respectively. The improved mechanical properties of these composites are attributed to the enhanced densification and the finer and more uniform nanostructure achieved by non-conventional fast sintering of slip-cast dense green compacts.

Keywords: A. Particle-reinforcement; B. Mechanical properties; E. Slip casting; Spark plasma sintering

1. Introduction

Aluminium titanate (AT, Al_2TiO_5) is a ceramic oxide isostructural to the mineral pseudobrookite characterized by low thermal expansion coefficient, low thermal conductivity, and excellent thermal shock resistance [1-3]. These properties make it to be a promising candidate material for service conditions involving thermal shock resistance and good thermal insulation, such as thermal processing technology, metallurgy, glass industry, automotive industry, refractory, engine components and thermal barriers, etc. [1,3,4]. Also, AT is a suitable additive as a second phase that can improve the thermal and mechanical properties of ceramic matrix composites [5]. For example, some researchers have shown that the addition of AT improves the fracture toughness of Al_2O_3 (A) due to the enhancement of the local residual stresses

induced by the large mismatch of thermal expansion coefficient between A and AT [5-8]. It was also observed that the incorporation of Al_2TiO_5 in an Al_2O_3 matrix resulted in better flaw-tolerance properties [6,7] while maintaining the structural integrity. Al_2O_3/Al_2TiO_5 (A/AT) ceramic composites, which combine the good mechanical properties of Al_2O_3 with the high thermal shock resistance of Al_2TiO_5 and display functional as well as structural properties, have found applications such as thermal barrier coating, exhaust filter components for diesel engines and high-temperature ceramic substrates [6,8].

The mechanical strength of Al_2TiO_5 ceramics also increases as the grain size decreases [9]. The properties of Al_2O_3-based ceramics strongly depend on the final microstructure, which is undoubtedly attached with the characteristics (size, shape, chemistry, etc.) of the starting powders [10]. In general, Al_2TiO_5 is formed by the solid-state reaction between Al_2O_3 and TiO_2 (T), which is thermodynamically possible above the eutectoid temperature 1280 ºC [11]. However, the microstructures obtained by reaction-sintering usually display low as-fired densities, abnormal grain growth, and even unreacted titania particles, owing to the special characteristics of the alumina-titania reaction process [12,13]. Previous studies have shown that colloidal processing allows dense, homogeneous A/AT materials to be obtained by reaction sintering of green bodies produced by slip casting of suspensions with controlled mixtures of alumina and titania. However, high temperatures (>1450 ºC) and long processing times (>18h) are required to obtain such dense materials (~97%) with good mechanical properties (230 MPa) by conventional sintering process [5,14,15]. In this sense, the selection of suitable raw materials and adequate green processing techniques is essential to obtain homogeneous, defect-free

materials but also the correct choice of the sintering method is crucial to obtain A/AT composites with improved properties. Spark plasma sintering (SPS) technique is a promising method which can work at heating rates of hundreds degrees per minute, reaching high temperatures in a short time, and producing dense materials [16]. These features allow the achievement of microstructures unattainable by other sintering methods and, therefore, obtain mechanical properties superior to those obtained using conventional technique.

In the present investigation we report, for the first time, the reaction-sintering by fast SPS technique of green bodies prepared by colloidal filtration of alumina and titania powders for the fabrication of in situ of Al_2O_3/Al_2TiO_5 ceramic composite.

The possibility of obtaining completely reacted and dense A/AT nanostructured composites at very low temperature by reaction-sintering is studied in detail. The microstructure and mechanical properties, such as flexural strength, Vickers hardness and fracture toughness of the obtained ceramic composite are studied and compared with those reported in the available literature for reaction sintered composites obtained by conventional sintering.

2. Experimental procedure

The following commercial powders were used as starting materials: 1) a submicron-sized, high purity α-Al_2O_3 (Condea-Ceralox HPA-0.5, Sasol, USA) with a mean particle size of 0.35 μm and specific surface area of 9.5 $m^2\ g^{-1}$; and 2) a nanosized TiO_2 powder (Aeroxide® P25, Degussa-Evonik, Germany) with an average primary particle size of 40 nm, a specific surface area of 50 $m^2\ g^{-1}$, and a relative ratio of anatase:rutile phases of 3:1 [17]. Mixtures of submicronic

alumina (A) and nanometric titania (T) were always prepared at a relative weight ratio of 87:13. The colloidal stability and the rheological behaviour of concentrated suspensions of nanosized titania powders have been studied elsewhere [18,19]. Suspensions were prepared to a solids loading of 40 vol.% using a commercial polyacrylic acid-based polyelectrolyte (DURAMAX^{TM} D-3005, Rohm & Haas, USA) as a deflocculant. The suspensions were prepared by first adding the PAA required to disperse the nanosized titania particles (4 wt.% PAA in relation to the titania content); the titania nanopowder was subsequently added and homogenized by sonication for 1 min. The alumina powder was then added after the relative amount of PAA needed to disperse the submicronic alumina particles (0.3 wt.% PAA in relation to the alumina content). The mixture was then maintained for 15 min under mechanical stirring. To improve the dispersion state, the suspensions were sonicated for 3 min using an ultrasound (US) probe (UP 400S, Dr Hielscher GmbH, Germany). The optimized suspensions were slip cast in plaster moulds to obtain discs with 20 mm in diameter and 5-8 mm in height.

These samples were introduced into a 20-mm-diameter graphite die and sintered using an SPS apparatus HP D25/1 (FCT Systeme GmbH, Rauenstein, Germany) at temperatures from 1250-1400 ºC and 80 MPa of pressure to obtain fully sintered bulk materials. The tests were carried out under vacuum at a heating rate of 100 ºC min^{-1} with a 1 min dwelling time at the maximum temperature.

The densities of the green and sintered compacts were determined by the Archimedes method (ISO-3369) using mercury and distilled water, respectively. Theoretical densities (TD) were calculated using values of 3.99 g cm^{-3} for

alumina [20], 3.89 g cm^{-3} for TiO_2 (anatase) [21], 4.25 g cm^{-3} for TiO_2 (rutile) [22], and 3.70 g cm^{-3} for aluminium titanate [23]. The crystalline phases of the bulk ceramic composites were determined by X-ray diffraction (XRD, D8 Advance, Bruker, Germany). The measurements were performed in the 15º-70º range and the step size and time of reading were 0.02º and 0.3 s, respectively. Vickers hardness and fracture toughness assessments were carried out using the indentation method. Sintered samples were longitudinally cut in half cylinders with a diamond saw. The samples were previously polished (Struers, model RotoPol-31) with diamond to 1 μm roughness. The hardness of the materials was determined using the indentation technique (Buehler, model Micromet 5103) with a conventional diamond pyramid indenter. The diagonals of each indentation were measured using an optical microscope. Measuring conditions for the Vickers hardness, *Hv*, were an applying load of 5 N for 10 s using 10 indents for each composite and the standard specification ASTM E92-72. The value of *Hv* is the relationship between applied load *P* and the surface area of the diagonals of indentation [24]. To estimate the indentation fracture toughness K_{IC}, 306 N Vickers indentations were performed on the surface of the samples, inducing Palmqvist cracks, from which the indentation fracture toughness was obtained by the method of Niihara [25]. The flexural strength was measured using biaxial testing using the equations of Kirstein and Woolley [26], Vitman and Pukh [27], and the standard specification ASTM F394-78. All tests were obtained at room temperature using the universal machine Instron (Model 856) with a cross-head displacement speed of 0.002 mm s^{-1}. The fracture surface sections of the sintered samples have been observed using a field emission gun scanning electron microscope (FESEM, HITACHI S-4800,

SCSIE of the University of Valencia).

3. Results and discussion

The XRD pattern of the bulk ceramic A/AT composites sintered by spark plasma reaction-sintering (SPRS) at 1250, 1300, 1350 and 1400 ºC are shown in Figure 1.

From the XRD patterns it can be observed that TiO_2 reflections are only observed in the samples treated at 1250ºC whereas for the composite sintered at ≥1300 ºC, no diffraction peaks of TiO_2 are detected, indicating that all of TiO_2 reacted with Al_2O_3 during SPRS process to form Al_2TiO_5 at very low temperature.

Aluminium titanate is the only stable phase in the alumina-titania system above 1280 ºC [11]. Below that temperature it would decompose to produce α-Al_2O_3 and TiO_2 (rutile). Thus it can be observed that full reaction of alumina-titania does not occur at 1250 ºC due to the short temperature and time in the SPRS process. To date, Duan et al. have reported the lowest reaction temperature to form AT from the A and T reaction in SPS [28]. In their research, nano-Al_2O_3 began to react with nano-TiO_2 powder to form the AT phase after SPS consolidation at 1150 ºC under a pressure of 63 MPa; however, the reaction was far to be complete and only a small amount of AT phase was formed in this composite. Yang et al. [29] studied the properties of the A/AT composite sintered by SPS at 1250 ºC with a holding time of 10-min and reported full reaction between A and T at this temperature and time combination. In the

present study, we investigated A/T mixtures sintered at different temperatures (1250-1400 ºC) with holding time of 1-min under a pressure of 80 MPa in vacuum by SPS and obtained that the TiO_2 has been completely reacted with Al_2O_3 to form Al_2TiO_5 after SPS consolidation at 1300 ºC; below this temperature TiO_2 (rutile) peaks still appear in the XRD pattern. It is remarkable that the reaction temperature in SPS is significantly lower than that needed in oxidizing atmospheres under the ambient air pressure [30].

The FE-SEM micrographs of the fracture surface of A/AT bulk ceramic composites obtained by SPRS at different temperatures are shown in Figure 2.

Only small microstructure differences between the composites are observed at this magnification. The ceramics prepared by SPRS exhibit full densification with absence of pores and with fine-grained growth of Al_2O_3 and Al_2TiO_5. The composite consolidated at 1250 ºC (Fig. 2a) shows a dense microstructure and has a narrow grain size distribution ranging from 0.5 μm to 0.7 μm. When the composites are sintered at higher temperatures (Fig. 2b, 2c and 2d), most Al_2O_3 grains are located in the range of 0.7-1.4 μm, but microstructural features maintain similar to that of the composite sintered at 1250 ºC. As it has been observed, the Al_2O_3 grain size has a homogeneous distribution, ranging from 0.5 μm to 1.4 μm, and the fracture mode observed in these materials is predominantly transgranular.

In Figure 3, characteristic fracture surfaces of the composites sintered at 1300 and 1400 ºC are observed at higher magnification. Nanosized aluminium

titanate is homogeneously distributed and mainly located at alumina triple points and grain boundaries, and no titania is detected, according to XRD.

As it is shown in Figure 3, the largest differences are found between grain sizes of alumina and aluminium titanate, which are much more smaller (<0.1 μm). As the sintering temperature increases (1300 to 1400) ºC, the alumina grain growth is greater, so that at 1400 ºC an average grain size ~1.4 μm is observed. This grain size is very small compared to that of monolithic alumina sintered by SPS at 1400 ºC reported elsewhere [32], which is ~5.0 μm. Therefore, the nanoparticles of the second phase to the aluminium titanate leads to an inhibitory effect in alumina grain growth.

The optimised green processing conditions and the non-conventional sintering by SPRS technique with simultaneous application of current and pressure is beneficial to obtain completely reacted and homogeneous sintered materials. The mechanical properties of the ceramic Al_2O_3/Al_2TiO_5 composites can be seen in Table 1.

The optimised colloidal processing led to high green and sintered densities of the composites (~62.5% and >98% of TD, respectively), which are roughly the theoretical density for ≥1300ºC. This means that it is possible to obtain full density A/AT materials by reaction-sintering in very short processing cycles (15-20 min) with non-conventional sintering technique (SPRS) with enhanced mechanical properties due the presence of Al_2TiO_5 nanoparticles as a second phase. Moreover, other important effect of the SPRS technique is a small grain

growth of alumina.

In addition, the excellent mechanical properties of the obtained bulk ceramic composites are also attributed to the use of SPRS. Therefore, SPRS is a promising technique for preparing ceramic products because of grain boundary strengthening and extensively preferential necking among particles occurring at relatively lower temperatures due to the simultaneous application of current and pressure.

To the best of our knowledge, the available information reported in the literature about the study of the mechanical properties of A/AT composites sintered by SPRS is very scarce [29,30]. Yang et al. [29] prepared Al_2O_3/Al_2TiO_5 ceramic composites with different microstructure (nano- (N) and microstructured (M)) by SPRS at 1250 ºC with a holding time of 10-min and a pressure of 30 MPa. They obtained the relative density of 92.9% and 99.3% from M and N powders, respectively, and the mechanical properties improved from N and M composites due to enhanced densification and fine microstructure. Also, the authors calculated values of the flexural strength, fracture toughness and Vickers hardness of the M composite which are, 222.5 MPa, 3.55 MPa $m^{1/2}$ and 6.8 GPa, respectively. In this work, we have obtained dense M composite (A/AT) at 1250 ºC with 1-min of holding time by SPRS with an increase of 66% of the Vickers hardness, an increase of 28% of the flexural strength and a similar value of fracture toughness over those reported by Yang et al [29]. For the N composite values, we have obtained an improvement of 15% of the Vickers hardness but the other mechanical properties were worse, despite achieving

the same density (>99% TD).

Furthermore, A/AT composites with AT contents of 10-40 vol.% and sintered by conventional sintering, were studied by other authors [5,31]. Bueno et al. [5] obtained alumina-10 vol.% aluminium titanate composites at 1450 and 1550 ºC with a density of 97.3% TD and flexural strength values 261 and 230 MPa, respectively. Yang et al. [31] obtained alumina-40 vol.% aluminium titanate composites at 1250, 1350, 1450 and 1550 ºC with lower density values of 70.1%, 80.2%, 91.5% and 88.7% TD, respectively. Hence, the mechanical properties of these composites were very low, e.g, the authors obtained flexural strength values of 69, 239, 362 and 316 MPa, respectively. For these values, we have obtained at 1350 ºC by SPRS an increase of 44% of the flexural strength (424 MPa).

Summarising, it is possible to obtain well-dispersed mixtures of Al_2O_3 and TiO_2 through colloidal processing. The high density and uniformity of the green bodies obtained by slip casting of these well dispersed mixtures allowed the subsequent sintering of dense and fully reacted Al_2O_3/Al_2TiO_5 composites. The homogeneous distribution of the nanostructured TiO_2 within the submicron sized alumina matrix, would serve as seed to promote in situ reactions during the sintering and bring the fine Al_2TiO_5 microstructure in the bulk composites. Moreover, the density values show good correlation with the flexural strength values.

4. Conclusions

In conclusion, in situ alumina/aluminium titanate bulk ceramic composites were successfully fabricated by colloidal stability of aqueous suspensions of submicron-sized alumina and nanosized titania particles and consolidated by SPRS at different temperatures with a holding time of 1-min. The density achieved is very high (>98.6% TD) and the alumina and aluminium titanate average grain size ranges in all composites were about 0.5 to 1.4 μm and <0.1 μm, respectively. It must be noted that the full reaction of Al_2O_3 and TiO_2 to obtain Al_2O_3/Al_2TiO_5 ceramic composites occurred at very low temperature, 1300 ºC. Therefore, the mechanical properties obtained are excellent, the composite sintered at 1350 ºC show e.g. (flexural strength 423.9 MPa, fracture toughness 5.4 MPa $m^{1/2}$ and Vickers hardness 24.2 GPa). This improvement is due not only to the nanostructured composite powders obtained but also to the fast sintering technique (SPRS), which allow us to obtain a finer and homogeneous microstructures.

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Figure captions:

Figure 1. XRD patterns of the Al_2O_3/Al_2TiO_5 bulk ceramic composites sintered by SPRS at 1250, 1300, 1350 and 1400 ºC.

Figure 2. FE-SEM micrographs of fracture surface of Al_2O_3/Al_2TiO_5 bulk ceramic composite obtained by SPS at different temperatures: a) 1250 ºC, b) 1300 ºC, c) 1350 ºC and d) 1400 ºC.

Figure 3. Composite Al_2O_3/Al_2TiO_5 sintered by SPS at: (a) 1300 ºC and (b) 1400 ºC. Nanometric second phase grains of aluminium titanate located at the boundaries between the alumina grains are pointed by narrows.

Sintering temperature (ºC)	Relative density (%)	Flexural strength (MPa)	Vickers hardness (GPa)	Fracture toughness (MPa $m^{1/2}$)
1250	98.6	304.8 ± 16.8	19.1 ± 0.7	3.1 ± 0.3
1300	99.7	414.3 ± 16.8	19.0 ± 0.5	5.7 ± 0.9
1350	99.6	423.9 ± 11.5	24.2 ± 1.2	5.4 ± 0.5
1400	99.9	489.7 ± 19.9	22.1 ± 1.3	5.3 ± 0.5

Table 1. Properties of Al_2O_3/Al_2TiO_5 ceramic composites obtained by SPRS at different temperatures.