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Additional Information

## Microstructure and mechanical properties of 5.8 GHz microwave-sintered $\text{ZrO}_2/\text{Al}_2\text{O}_3$ ceramics

Lorena Gil-Flores<sup>1\*</sup>, Maria D. Salvador<sup>1</sup>, Felipe L. Penaranda-Foix<sup>2</sup>, A. Fernández<sup>3</sup>, M. Suarez<sup>3</sup>, Roberto Rosa<sup>4</sup>, Paolo Veronesi<sup>4</sup>, Cristina Leonelli<sup>4</sup>, Amparo Borrell<sup>1</sup>

<sup>1</sup>Instituto de Tecnología de Materiales, Universitat Politècnica de València, Camino de Vera s/n, 46022, Valencia, Spain

<sup>2</sup>Instituto de Aplicaciones de las Tecnologías de la Información y de las Comunicaciones Avanzadas (ITACA), Universitat Politècnica de València, Camino de Vera s/n, 46022, Valencia, Spain

<sup>3</sup>Centro de Investigación en Nanomateriales y Nanotecnología (CINN) (CSIC-UO-PA), Avenida de la Vega 4-6, 33940 El Entrego (Asturias), Spain

<sup>4</sup>MAG\_Microwave Application Group, Dipartimento di Ingegneria “Enzo Ferrari”, Università degli Studi di Modena e Reggio Emilia, Via P. Vivarelli 10, 41125 Modena, Italy

\*Corresponding author: Instituto de Tecnología de Materiales (ITM), Universitat Politècnica de València, Camino de Vera s/n, 46022, Valencia, Spain. Tel.: +34963877007; Fax: +34963877629. E-mail address: *logiflo@upv.es*

### Abstract

Aim of the present study is to sinter zirconia nanocomposite powders doped with ceria and toughened with alumina (10Ce-TZP/ $\text{Al}_2\text{O}_3$ ) by non-conventional means, i.e. microwave sintering technology. The sintering effects of various microwave applicators and frequency generators were evaluated using an optimised experimental set-up. The microwave-sintered samples were compared with the composites sintered by the conventional method. The mechanical properties of the ceramic composites were evaluated by their hardness, fracture toughness and Young's modulus. Likewise, their density and microstructure were analysed.

**Keywords:** Zirconia-alumina; Ceria; Microwave sintering; Bioceramics; Mechanical properties; Microstructure

## 1. Introduction

The biomaterials market is currently on the rise, as the use of materials as a substitute for different tissues and parts of the human body has increased in recent years due to increased life expectancy and quality of life.

Depending on the item to be repaired or replaced, very different materials can be used, including metals, polymers and ceramics. Each group has advantages and disadvantages. Indeed, ceramic materials are being increasingly used because of their biocompatibility and bio-inertness, but their hardness and fragility still represent their main disadvantages [1,2].

Zirconia-based composites are commonly used to develop metal-free restorations and dental implants because of their superb mechanical properties, biocompatibility and aesthetics. Moreover, in accord with a new report carried out by Grand View Research, Inc., S. Francisco, USA, the global bio-ceramics market is expected to achieve 19.05 billion dollars by 2022; the most used bio-ceramics are and will continue to be alumina, zirconia and their composites – the material to be studied in this work is an alumina-toughened zirconia composite [3]. The addition of alumina to the zirconia matrix causes an increase in hardness and fracture toughness, while its grain growth slows down to significantly reduce its size [4,5].

Tetragonal zirconia polycrystalline (TZP) is stabilised with ceria oxide ( $\text{CeO}_2$ ); this is a less known dopant, as the one commonly used for the stabilisation of zirconia is yttrium oxide. Using  $\text{CeO}_2$  as a stabiliser makes it possible to improve the fracture toughness and reduce the low temperature degradation (LTD) suffered by the TZP samples [6]. Low temperature degradation is considered a big problem for biomaterials; zirconia is especially susceptible to be degraded in the presence of water [7–9]. Due to this aging, 400 femoral heads broke down in a brief period of time in 2001 [8]. This creates a huge demand for a bio-ceramic with reduced hydrothermal ageing. Therefore, the composite studied in this work was 10 mol% Ce-TZP/ $\text{Al}_2\text{O}_3$  in the ratio of 65/35 vol%. Only a few values of fracture toughness for the 10 mol% Ce-TZP/ $\text{Al}_2\text{O}_3$  composite using the

Vickers indentation method have been reported in the literature. According to Tenaka et al. [10], toughness is measured at  $20.1 \text{ MPa}\cdot\text{m}^{1/2}$  for a nanocomposite of 10 mol%  $\text{CeO}_2$ -stabilised TZP doped with 0.05 mol% of  $\text{TiO}_2$  and 30 vol% of  $\text{Al}_2\text{O}_3$  as a second phase.

However, obtaining dense ceramics with adequate properties and energy-efficient processes is currently a great challenge. The choice of sintering technology is very important to produce sustainable ceramic materials. A change in sintering mechanisms – and consequently the properties of the sintered sample – depends on the sintering technology used [11]. Therefore, innovative non-conventional approaches for ceramic processing, such as microwave sintering, are emerging to decrease time and energy consumption and, consequently, its environmental impact. In the field of biomaterials, the development of these innovative technologies has been induced by the increase in demand for materials which are able to support new specifications, as well as the need to cut processing times and costs. In this study, conventional and non-conventional processes, i.e. microwave dielectric heating, were compared to obtain 10 mol% Ce-TZP/ $\text{Al}_2\text{O}_3$  nanocomposites.

Microwave sintering does not require energy to heat the entire furnace, so many of the components of a conventional furnace are not necessary. Hence, the use of this technology substantially reduces energy expenditure, especially in high-temperature processes, thus thermal losses increase considerably as sintering temperatures rise.

The heating processes involved in conventional and microwave sintering are completely different; in conventional sintering, the direction of heating is from the surface of the material to its interior. Nevertheless, microwave heating follows the opposite direction, heat flows from the inside to the outside; this is known as volumetric heating [12–14].

Ever since the early investigations on the microwave sintering of zirconia [15–17] and  $\text{ZrO}_2/\text{Al}_2\text{O}_3$  composites [18–20], ceria has also been added in addition to zirconia [21,22]. But a proper approach to microwave sintering has only recently led to the complete process control of  $\text{ZrO}_2$  sintering [23,24].

The shape of the ceramic samples as well as the microwave frequency used can cause temperature gradients that make it difficult to heat the body uniformly. However, most of the research carried out to date uses microwave applicators with the most diffuse

frequency (2.45 GHz), although many ceramics exhibit low dielectric loss or in other words, poor microwave absorption characteristics at this frequency.

In this sense, the main objective of this study was to obtain highly densified samples of 10 mol% Ce-TZP/ $\text{Al}_2\text{O}_3$  through microwave sintering using two different cavities operating at two frequencies: 2.45 GHz and 5.8 GHz. The suggested study opens up the possibility of investigating changes in geometry as well as the frequency of the microwave source and its effect on both energy efficiency and processing time.

This work studies the correlation between the microstructure and mechanical properties of microwave-densified samples, and the results are compared with corresponding 10 mol% Ce-TZP/ $\text{Al}_2\text{O}_3$  materials obtained through conventional sintering in an electric resistance furnace.

## 2. Materials and methods

The studied composite was prepared in the laboratory; the starting powders were  $\text{ZrO}_2$  with 10 mol%  $\text{CeO}_2$  (10Ce-TZP, Daiichi Kigenso Kagaku Kogyo, Japan),  $d_{50}$  35 nm, and alumina (SPA 0.5, Sasol, Germany),  $d_{50}$  380 nm. The proportion of the composites was 65 vol% of 10Ce-TZP and 35 vol%  $\text{Al}_2\text{O}_3$  (10Ce-TZP/ $\text{Al}_2\text{O}_3$ ). This composition was based on a previous study [25].

10Ce-TZP/ $\text{Al}_2\text{O}_3$  nanocomposite was prepared by mixing and drying. Stable slurry with 67 wt% of solids was prepared in distilled water by attrition milling at 45 Hz for 3.5 hours, using Dolapix CE-64 as a dispersant. Subsequent to this attrition milling, the slurry was spray-dried using Optapix PA4G as a binder to ensure the formation of the granules. A peristaltic pump was used to introduce the slurry into hot-air stream, which was cooled through the evaporation of the water from the surface of the particles. After the spray-drying process, the powder presented a greater fluidity. Green samples were uniaxially pressed at 80 MPa with a Shimadzu AG-X Plus to obtain cylindrical bodies with a diameter of 10 mm and a height of 3 mm, which were then employed for the sintering tests.

Conventional sintering (CS) was performed in an electric furnace at two different temperatures (1,400 and 1,500 °C) with a heating rate of 10 °C/min and a dwell time of 120 minutes, under atmospheric conditions.

Microwave technique (MW) was conducted in two microwave systems operating at two different magnetron frequencies, 2.45 and 5.8 GHz. The first heating test was carried out in a cylindrical cavity operating in single-mode ( $TE_{111}$ ) at 2.45 GHz frequency. The cavity has a hole with a diameter of 30 mm at the top where a quartz tube, which contained the sample, was introduced. The temperature sensor was also placed in the same hole.

Both the position and dimension of the hole was designed to avoid leakage of microwave from the cavity and make sure the insignificant perturbation of the resonant mode. The sample was located in the centre of the cavity, where the maximum electric field magnitude was held. A moving short-circuit at the bottom of the cavity made it possible to vary the cavity dimensions and favour the dielectric properties of the sample during the sintering process [26]. An optical pyrometer, previously calibrated in this temperature range, was used to measure the sample temperature.

The 5.8 GHz single-mode applicator used for this study had a cavity with rectangular geometry (WR159) shorted by a moving plunger. The temperature was monitored simultaneously with the microwave sintering using a sapphire fiber (MIKRON M680 Infraducer, Mikron Infrared Inc., Santa Clara, CA, USA), that directly touched the free upper surface of the sample and was connected to a signal conditioner.

A specific temperature detection procedure was applied to correctly monitor the sintering cycle, optimised by the Microwave Application Group at the University of Modena and Reggio Emilia, Italy [27].

Samples were introduced into the two single-mode cavities with different frequencies, which were adjusted to optimize the microwave absorption of the material as well as to maintain the heating rate of 50 °C/min. Two final temperatures were selected, 1,200 and 1,300 °C, with a dwell time of 10 minutes at the sintering temperature [28,29]. A silicon carbide susceptor was employed in both cavities in order to aid the heating of the sample in the microwave cavity.

The Archimedes method was used to evaluate the final densities of the sintered samples in line with the ASTM-C-373 standard. Mechanical properties were assessed on surfaces that had been polished down to 1  $\mu$ m using diamond paste. Hardness ( $H$ ) and the Young's Modulus ( $E$ ) were measured by nanoindentation technique, using a Berkovich tip (G-200; Agilent Technologies, Barcelona, Spain). Tests were calibrated

with silica standard and operated at a maximum depth of 1,500 nm. The continuous stiffness measurement was used to determine the contact stiffness and calculate the hardness profiles and elastic modulus. Each sample was tested with a matrix of 16 indentations, whose amplitude were set to 2 nm at a frequency of 45 Hz.

Fracture toughness,  $K_{IC}$  [ $\text{MPa}\cdot\text{m}^{1/2}$ ], was calculated through the indentation fracture method following the equation of Evans et al. [30]:

$$K_{IC} = 0.16 \cdot \left(\frac{c}{a}\right)^{-1.5} \cdot H_V \cdot a^{0.5}$$

where  $H_V$  is the Vickers hardness and  $c$  [m] and  $a$  [m] are the Palmqvist crack and the half length of the Vickers impression, accordingly. The loads applied in this test were 20 kg for 15 seconds with a Centaur HD9-45 (Metrol Centaur, S.L., Bilbao, Spain). Five measurements were done for each sample.

Specimen microstructures were characterised by means of field emission-scanning electron microscopy. A 30 minutes thermal etching was performed at 100 °C below the maximum temperature to reveal grain boundaries. The linear intercept method was used to measure the average grain sizes of both zirconia and alumina [31]. Approximately 100 grains were considered for each phase.

### 3. Results and discussion

#### Densification

The relative density of the studied composite 10Ce-TZP/ $\text{Al}_2\text{O}_3$ , can be seen in Figure 1. Theoretical density has been measured from the dense powdered material with a helium pycnometer (AccuPyc 1330, Micromeritics, Georgia, USA), which value is  $5.06 \text{ g/cm}^3$ . With the increase of the sintering temperature, the relative densities underwent a constant rise from 98.5 to 99.5%.

With regard to the results of microwave sintering, the first significant outcome is that the density values of the material vary with the frequency of the electromagnetic field applied. The composites that were sintered at 2.45 GHz show a slightly lower density than those sintered at 5.8 GHz. An increase in microwave frequency can lead to an improvement in the absorption of microwaves by the ceramic material, thereby

transmitting more energy to the material. As a result, a slight increase in the final density of the ceramic was registered.

When both sintering technologies are compared, the density of microwave-sintered materials are higher (from 98.7% at 1,200 °C – 10 minutes at 2.45 GHz – to 99.5% at 1,300 °C – 10 minutes at 5.8 GHz), even though they were processed with lower temperatures and sintering times than the conventionally processed material. Thus, the sample sintered by CS at 1,500 °C for 120 minutes has the same relative density as those sintered by MW for 10 minutes at 5.8 GHz and 1,300 °C.

Briefly, to achieve relative densities above > 99.4% requires 350 minutes and 1,500 °C in conventional sintering, while the MW needs only 35 minutes at 1,300 °C using 5.8 GHz to obtain similar dense specimens. Therefore, one remarkable result is that microwave sintering allows highly sintered samples to be obtained in less processing time and at considerably lower temperatures than conventional sintering method. It is important to note the high demand for reduced processing times in many fields like the dental sector. Even if we assume an incorrect temperature determination in the case of microwave sintering, a shorter process time can still be observed – having used perfectly identical specimen geometries and masses (for a detailed discussion on the measurement of temperature under microwave irradiation, see Refs. [27,32]).

The differences in relative densities between the two sintering methods: MW and CS samples can be associated to the different mechanisms used to heat the samples. The heating in CS is generated by heating elements, such as resistors, and transferred to the specimen by radiation, conduction and convection, whereby the outside of the sample is heated before the core. The heat penetrates from the exterior to the bulk through the conduction phenomena.

In contrast, microwave heating is able to reach greater heating rates than conventional methods. This fact is because microwave power is absorbed directly by the material and it is heated. This is why microwave sintering does not only depend on thermal conduction to transfer heat [33]. Additionally, it has already been demonstrated that dielectric heating activates the surface mass transport mechanism, which efficiently closes the pores [32].

It should be noted that the generation, propagation and absorption of microwaves within the volume of the material as well as the temperature or the evolution and distribution of



the electromagnetic field in the resonant cavities, influence the densification and therefore the final properties of the material obtained. It has been that the microwave power absorbed by the material depends to a great extent on their position in the cavity [34].

On the other hand, in this study, it has been observed that the use of 5.8 GHz frequency for the sintering of ceramic bodies has some benefits compared to the use of a 2.45 GHz microwave source. Another important factor is the geometry of the cavity and homogeneity of temperature distribution. In this work, a rectangular cavity was used at 5.8 GHz and a cylindrical cavity at 2.45 GHz. A small change in the microwave power absorbed by the material can lead to enhanced densification.

### **Microstructure and grain size**

FE-SEM micrographs of 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> composite obtained by microwave sintering at 2.45 GHz and 5.8 GHz and its comparison to conventional sintering are shown in Figures 2 and 3.

As can be seen, the samples that were sintered by microwave at lower temperature (1,200 °C) show little residual porosity compared with samples sintered at 1,300 °C. Small differences in MW-processed samples can be observed as temperature and frequency increases. At 5.8 GHz, the samples show a dense microstructure with less residual porosity than those obtained at 2.45 GHz.

The grain sizes of zirconia and alumina are smaller than 550 nm for the composites obtained at both frequencies. The grain size of alumina (dark grains) is maintained between 270 and 380 nm, while that of zirconia tends to increase with temperature and frequency (from 240 nm at 1,200 °C and 2.45 GHz to 520 nm at 1,300 °C and 5.8 GHz). In the sintering process, finely distributed particles of zirconia delay the movement of the alumina grain boundaries, inhibiting the growth of the alumina grain [35]. The increase of the zirconia grain size is promoted once the sample is completely dense.

Specimen 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> prepared by conventional sintering shows a slightly higher grain size for both zirconia and alumina phases. Figure 3b shows the fully dense microstructure and the finer alumina grains (~430 nm) were dispersed homogeneously in the zirconia matrix (~570 nm). In Figure 3a, a small porosity can be observed. This matches the relative density data previously illustrated in Figure 1.

As an overall conclusion, microwave sintering demonstrated the expected advantages of grain size retention together with an evident reduction in energy consumption and processing times; indeed, the typical and high speed of microwave heating can contribute to obtaining better quality products, leading to final materials with thinner microstructures with fewer defects.

### **Hardness measurements**

With respect to the mechanical properties, the values obtained match the calculated relative density. The hardness values obtained through the different heating modes and frequencies are presented in Figure 4.

Samples with the lowest relative density have the lowest hardness values. As can be seen, there is a difference in the hardness values of the samples sintered by MW at a different frequency. When microwave sintering is carried out at 2.45 GHz, the hardness of the composites at 1,200 and 1,300 °C is slightly lower (~13 GPa) in relation to the values obtained at 5.8 GHz, suggesting that the effects of residual porosity are still significant. However, all composites show higher hardness values at 5.8 GHz: Particularly at 1,200 °C and 1,300 °C with 10 minutes of dwelling time, the hardness values are 14.2 GPa and 14.6 GPa, respectively. The latter is close to the maximum hardness value obtained in this work (15.1 GPa), which corresponds to the sample prepared by conventional sintering at 1,500 °C.

It can therefore be noted that MW-sintered samples show Hv values comparable to conventionally sintered samples despite being sintered for less time at a much lower temperature. However, the differences in Hv have a tendency to be more pronounced considering the two different frequencies adopted for MW sintering, with the sintering frequency of 5.8 GHz resulting in a significant improvement of the Hv values for the investigated material.

### **Young's Modulus**

Young's modulus values are presented in Figure 5. There are no substantial differences between the samples prepared using different techniques. In general, higher density leads to higher Young's modulus values, although their relationship is not linear. Many factors affect E values, such as porosity, grain size, grain distribution, and pores, among others.

The highest and lowest values correspond to the samples sintered by CS at 1,500 °C and 1,400°C for 120 minutes, (278 and 220 GPa respectively). However, the Young's modulus of samples sintered by MW are also between 220-270 GPa, while slightly higher values are observed for samples prepared at a higher frequency.

It should be noted that the Young's modulus values are very similar to those found in the literature for similar composites (Table 1). Moreover, these results confirm that the use of Ce as a stabilising agent has no remarkable influence on the Young's modulus, since the values are approximately those obtained for the samples stabilised with Yttria [36].

### Fracture toughness

One of the key mechanical properties of bio-ceramics is fracture toughness,  $K_{IC}$ . In the case of 10Ce-TZP/ $Al_2O_3$  composite, the addition of ceria as a stabiliser induces a considerable increase in its fracture toughness in comparison with yttria-doped zirconia (Y-TZP) materials, producing a material much more resistant to fracture, which is of vital importance when used for implants and prostheses. Figure 6 presents the values of  $K_{IC}$  for 10Ce-TZP/ $Al_2O_3$  sintered by microwave at different frequencies and conventionally at a different temperature.

The CS-sintered samples have similar  $K_{IC}$  values ( $10.0 \pm 0.4$  and  $10.8 \pm 0.5$   $MPa \cdot m^{1/2}$ , for 1,400 and 1,500 °C, respectively). The samples sintered by microwave heating present more pronounced differences in terms of their fracture toughness values. Particularly by increasing the sintering temperature at 2.45 GHz, the fracture toughness values decrease from  $11.2 \pm 1.1$  to  $10 \pm 2.2$   $MPa \cdot m^{1/2}$ . This drop can be explained by the increase of zirconia grain size ( $> 100$  nm), as the density also increases slightly.

On the other hand, the fracture toughness values of samples sintered at 5.8 GHz increase with the sintering temperature, reaching similar  $K_{IC}$  values,  $9.6 \pm 1.3$   $MPa \cdot m^{1/2}$  and  $11.2 \pm 1.0$   $MPa \cdot m^{1/2}$  at 1,200 and 1,300 °C respectively, with the latter also being superior to the value obtained by conventional sintering at 1,500 °C for 120 minutes.

The samples sintered by MW at 5.8 GHz and 1200 °C and MW at 5.8 GHz and 1300 °C have similar  $K_{IC}$  values, although this last sample has a higher density. This is also due to the increased grain size of the zirconia (240 and 520 nm, respectively).

The  $K_{IC}$  values estimated for 10Ce-TZP/ $Al_2O_3$  show that MW sintering results in higher (or comparable) values both at sintering temperatures and at frequencies than those

obtained by conventional sintering. If sintered samples are compared, it can be determined that MW enhances the  $K_{IC}$  of this material. These results coincide with those found by other authors when sintering  $Al_2O_3$ - $ZrO_2$ , with a different percentage of  $ZrO_2$  by microwave at 2.45 GHz.[26].

Following a thorough inspection of the literature, it may be stated that not many studies have focused on the composite with a similar composition of ceria-doped zirconia and alumina. Table 1 contained a summary of hardness, as well as Young's modulus and fracture toughness values found in the literature [37–39], comparing them with the best results obtained in the present study. Nawa and Tenaka et al. also obtained materials with higher fracture toughness values using additives such as  $TiO_2$  and  $MgO$  [38,39].

The most remarkable result is that MW-sintered material has high  $H$ ,  $K_{IC}$  and  $E$  values that are at least comparable with those found in the literature for materials with a similar composition and, therefore, our composites present values within the admissible range for applications as implants and prostheses.

Table 1. Values of hardness, Young's modulus and fracture toughness of the composite tested by different researchers and in the present work.

Material	Authors	$H_v$ (GPa)	E (GPa)	$K_{IC}$ ( $MPa \cdot m^{1/2}$ )
<b>Conventional sintering</b>				
12Ce-TZP	[37]	9.8-10.8 <sup>a</sup>	-	7.8*
70vol% 10Ce-TZP+ 30vol% $Al_2O_3$ (1500 °C)	[38]	11.6 <sup>a</sup>	248	18.1 <sup>c</sup> 9.2 <sup>d</sup>
70vol% 10Ce-TZP + 30vol% $Al_2O_3$ + $TiO_2$ (1440 °C)	[39]	11.7 <sup>a</sup>	247	20.1 <sup>c</sup>
65vol% 10Ce-TZP + 35vol% $Al_2O_3$ (1500 °C)	Present study	15.1 <sup>b</sup>	278	10.8 <sup>c</sup>
<b>Microwave sintering</b>				
65vol% 10Ce-TZP + 35vol% $Al_2O_3$ (1300 °C,	Present study	14.6 <sup>b</sup>	270	11.2 <sup>c</sup>

5.8 GHz)

Hardness has been estimated by: (a) microhardness, and (b) nanohardness. Fracture toughness has been estimated by: (c) the indentation-fracture method, and (d) SEVNB method (except for value with \*, that was measured by the Double Torsion method).

## Conclusions

This study has evaluated two different sintering methods and the varying properties of 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> depending on the sintering conditions.

The sintering behaviour of 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> composite has been investigated through conventional and microwave sintering, applying two different frequencies: 2.45 GHz and 5.8 GHz. No previous studies had been found where the behaviour of sintering in ceramic materials at high temperature was investigated and compared by using these frequencies.

Overall, microwave sintering has proven to be an exceptional alternative for sintering 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> composite, due to the fine microstructure and good mechanical properties of the resulting materials. Furthermore, this technology requires lower sintering temperatures and dwelling time than conventional sintering, leading to a reduction in energy costs and processing times and, consequently, the microwave technique has a lower environmental impact.

With regard to the relative density values, the samples sintered by MW at 2.45 GHz or 5.8 GHz present a little variation in densification, though higher values were obtained in the case of 5.8 GHz, irrespective of the sintering temperature. The same applies to its mechanical properties ( $H_V$ ,  $E$ ,  $K_{IC}$ ), reaching values like 14.6 GPa, 270 GPa and 11.2 MPa·m<sup>1/2</sup>, respectively, for the sample densified at 1,300 °C with 5.8 GHz, and 13 GPa, 225 GPa and 10 MPa·m<sup>1/2</sup>, respectively, for the sample densified at 1,300 °C with 2.45 GHz. The fracture toughness values of all the sintered composites are high enough for structural applications (prostheses and implants).

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Figure Captions:

Figure 1. Relative density values of 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> composites sintered by microwave as a function of frequency and conventional at different temperatures.

Figure 2. FE-SEM micrographs of 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> composites sintered by microwave as a function of frequency at different temperature. 2.45 GHz: (a) 1200 °C-10 min, (c) 1300 °C-10 min, and 5.8 GHz: (b) 1200 °C-10 min, (d) 1300 °C-10 min.

Figure 3. FE-SEM micrographs for 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> materials conventionally sintered at different conditions: (a) 1400 °C - 120 min and (b) 1500 - 120 min.

Figure 4. Hardness values of 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> composites sintered by microwave as a function of frequency and conventionally at different temperatures.

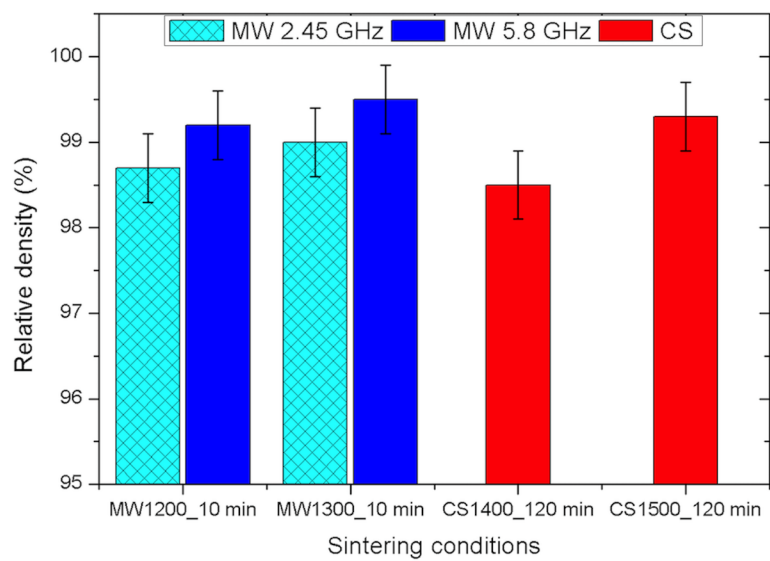
Figure 5. Young's modulus values of 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> composites sintered by microwave as a function of frequency and conventionally at different temperature.

Figure 6. Fracture toughness values of 10Ce-TZP/Al<sub>2</sub>O<sub>3</sub> composites sintered by microwave as a function of frequency and conventionally at different temperature.

Table 1. Values of hardness, Young's modulus and fracture toughness of the composite tested by different researchers and in the present work.

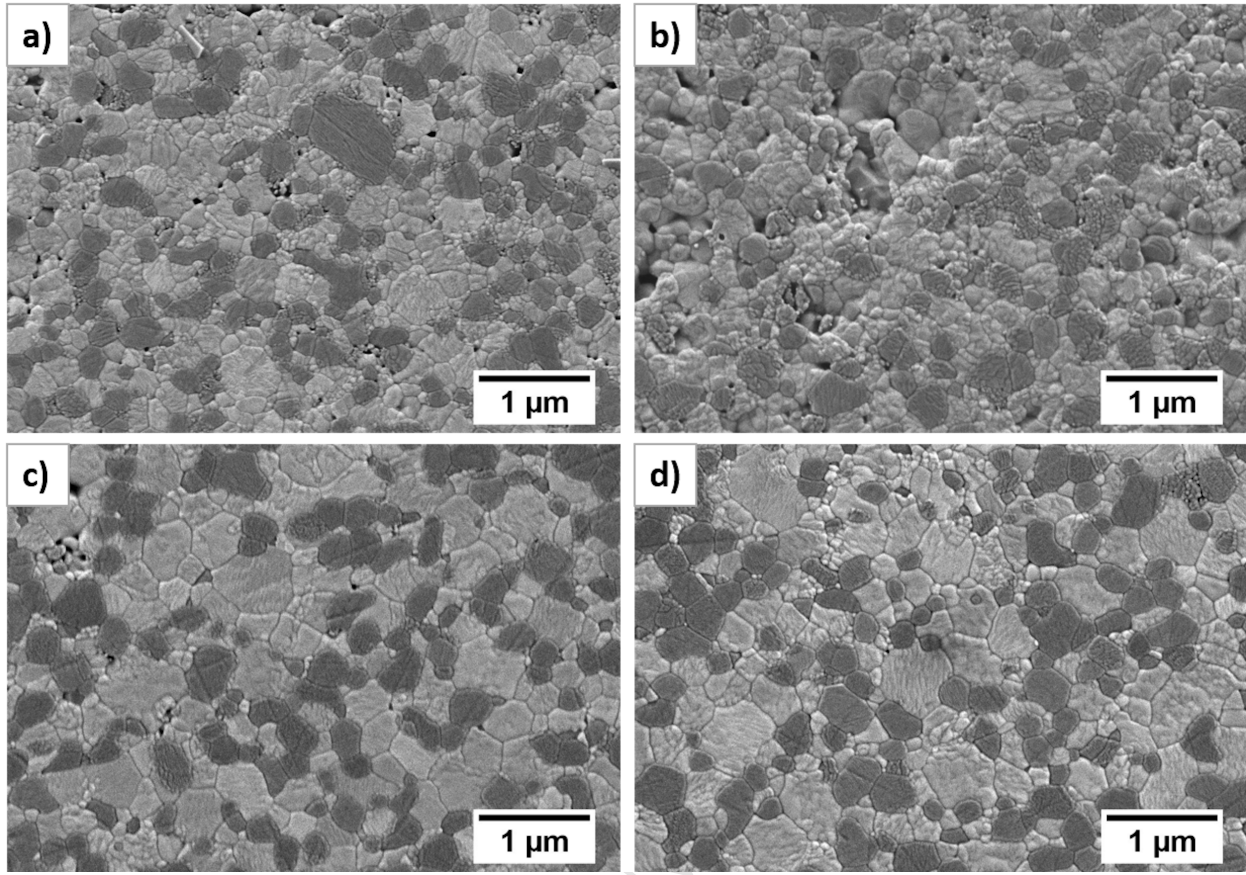
Material	Authors	H <sub>v</sub> (GPa)	E (GPa)	K <sub>IC</sub> (MPa·m <sup>1/2</sup> )
<b>Conventional sintering</b>				
12Ce-TZP	(Chevalier and Gremillard, 2009)	9.8-10.8 <sup>a</sup>	-	7.8*
70vol% 10Ce-TZP+ 30vol% Al <sub>2</sub> O <sub>3</sub> (1500 °C)	(Nawa et al., 1998)	11.6 <sup>a</sup>	248	18.1 <sup>c</sup> 9.2 <sup>d</sup>
70vol% 10Ce-TZP + 30vol% Al <sub>2</sub> O <sub>3</sub> + TiO <sub>2</sub> (1440 °C)	(Tanaka et al., 2003)	11.7 <sup>a</sup>	247	20.1 <sup>c</sup>
65vol% 10Ce-TZP + 35vol% Al <sub>2</sub> O <sub>3</sub> (1500 °C)	Present study	15.1 <sup>b</sup>	278	10.8 <sup>c</sup>
<b>Microwave sintering</b>				
65vol% 10Ce-TZP + 35vol% Al <sub>2</sub> O <sub>3</sub> (1300 °C, 5.8 GHz)	Present study	14.6 <sup>b</sup>	270	11.2 <sup>c</sup>

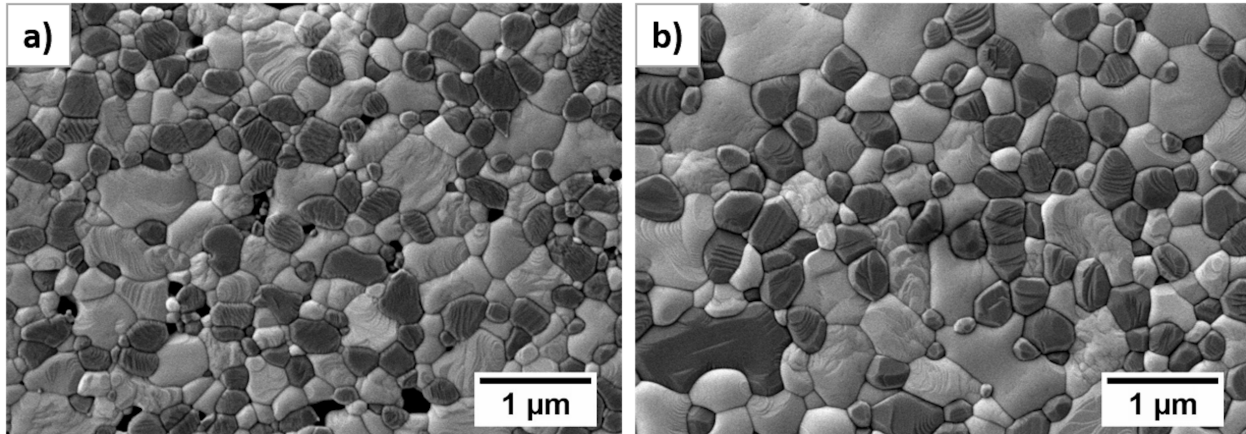
Hardness has been estimated by: (a) microhardness, and (b) nanohardness. Fracture toughness has been estimated by: (c) the indentation-fracture method, and (d) SEVNB method (except for value with \*, that was measured by the Double Torsion method).

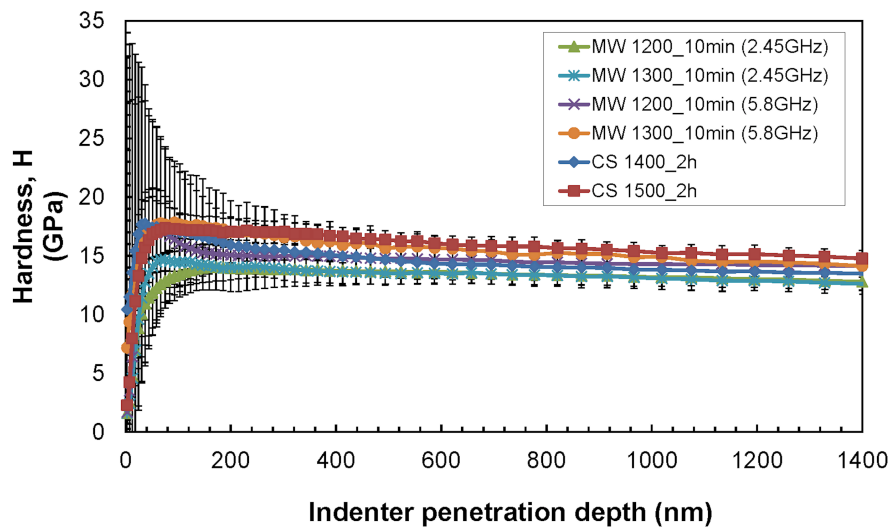


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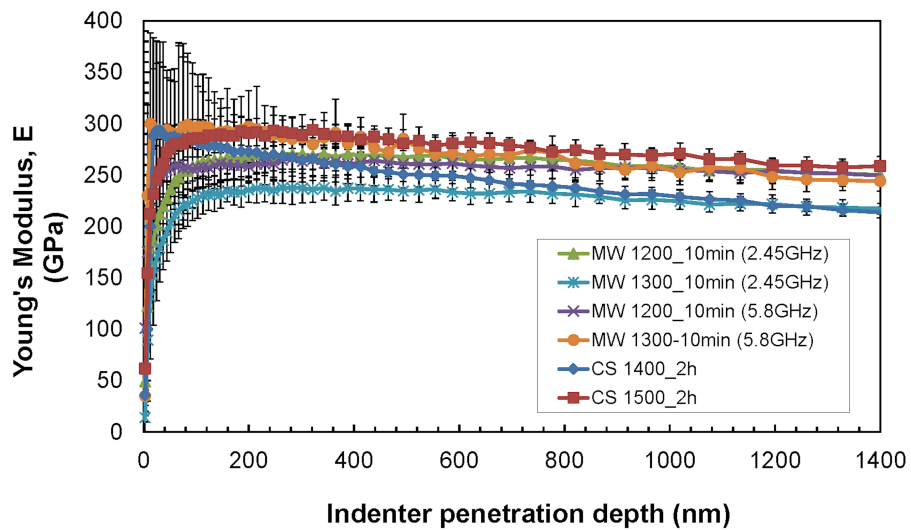
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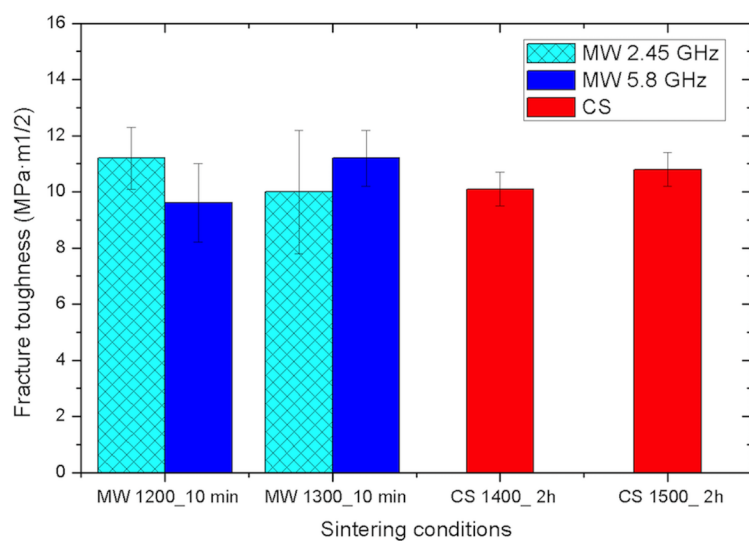












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