Chapter

Design and Development of Zirconia-Alumina Bioceramics Obtained at Low Temperature through Eco-Friendly Technology

Rut Benavente, Maria Dolores Salvador and Amparo Borrell

Abstract

Ceramics are increasingly used as structural materials with biomedical applications due to their mechanical properties, biocompatibility, esthetic characteristics and durability. Specifically, zirconia-based compounds are commonly used to develop metal-free restorations and dental implants. The consolidation of ceramics is usually carried out through powders by means of processes that require a lot of energy, as long as processing times and high temperatures (over 1400°C) are required. In the recent years, new research is being developed in this field to reduce both energy consumption and processing time of ceramic powders. One of the most promising techniques for sintering ceramics is microwave heating technology. The main objective of this chapter is to obtain highly densified zirconia-alumina compounds by microwave technology. After sintering, the materials are characterized to determine whether the final properties meet the mechanical requirements for their final applications as dental material. Finally, the characterization of specimens treated by low-temperature degradation (LTD) is carried out after each 20 h of LTD exposure up to 200 h. In addition, the quantification of monoclinic phase by micro-Raman spectroscopy, analysis by AFM and Nomarski optical microscopy and assessment of the roughness and mechanical properties (hardness and Young's modulus) by nanoindentation technique have been studied.

Keywords: zirconia-alumina, microwave sintering, bioceramics, mechanical properties, hydrothermal degradation

1. Introduction

In recently years, zirconia and alumina have been recognized as the most relevant ceramic materials due to their outstanding properties, such as hardness, fracture toughness, Young's modulus, chemical stability, wear and mechanical strength. Due to these excellent properties, ZrO_2 and Al_2O_3 are appropriate materials for a wide range of applications, such as the manufacture of sensors, fuel cells, thermal barriers, implants and structural engineering applications [1–3]. Recent studies claim that the

incorporation of alumina into the zirconia matrix, alumina-reinforced zirconia (ATZ) materials, improves the mechanical properties (i.e., hardness, toughness and wear resistance) [4, 5], as these composites combine the unique properties of alumina and zirconia. These characteristics make ZrO_2 and Al_2O_3 promising composites for prosthetic and dental implant applications. There are a number of reports on the sintering of ATZ composites in the literature. Li et al. [4] have sintered a ZrO_2 (3YTZP) + 20% wt% Al_2O_3 composite by spark plasma sintering (SPS) a non-conventional sintering technique. The hardness and fracture toughness values achieved for these samples sintered by SPS at 1400°C were 12.5 GPa and 5.3 MPa-$m^{1/2}$, respectively. The ATZ (ZrO_2 (3YTZP) + 10% vol. Al_2O_3) composite was also studied by Meena and Karunakar [5]; in this case, the maximum hardness values for samples sintered by SPS at 1300°C were 19.8 GPa. In summary, the final properties and microstructure of the materials depend on the densification process of the material, sintering mechanisms and methods [6–9].

Substantial improvements in dental prostheses and implants have been achieved through the employment of ceramic-based materials, mainly thanks to the advent of yttria-stabilized zirconia polycrystalline (Y-TZP) as biomaterials [10–13]. The replacement of metal parts in orthopedic and dental applications with ceramics is currently on the rise. Ceramic materials provide several advantages over metals, such as biocompatibility, outstanding mechanical properties and esthetics.

However, an important characteristic of these materials is completely stabilized zirconia tetragonal (t) phase with the addition of an oxide (Y_2O_3, CeO_2,...), for example, with ~3.0 mol% of yttria for dental applications. This is important because when Y-TZP materials are subjected to humidity at 25–280°C [14–16], a sharp decline in their mechanical properties occurs over time. This phenomenon is referred to as low-temperature degradation (LTD) [17, 18]. The t-m transformation produces volume changes and defects in the material, and the mechanical and esthetic properties are affected. Thus, it is highly relevant to research the susceptibility of Y-TZP-based materials to LTD. LTD process is influenced by different factors, such as the tetragonal stabilizing dopant, the grain size or the porosity. Several of these elements are related to the process of sintering and its heating mechanisms.

In the late 1990s, the Y-TZP femoral heads used for the hip replacement failed catastrophically within the human body and these failures were attributed to the hydrothermal aging process [19, 20]. The conditions that promote LTD are found in the dental cavity; therefore, it is critically necessary to investigate the effects of LTD on Y-TZP-based materials for these uses.

Often, ceramics are full consolidated using a thermal treatment at high temperatures (>1200°C), where the temperature and dwell time are the most significant parameters since they establish the mechanical properties and microstructure of the densified material. Conventional sintering requires long-processing times and high temperatures and consequently high-energy expenditure. For this reason, non-conventional and fast sintering methods, such as microwave heating technology, are being implemented in the last decade. Microwave sintering is founded on the absorption of electromagnetic radiation, resulting in an increase in the temperature of the material [21–23]. The mechanism of microwave heating differs from the one used in conventional sintering, since the temperature gradient is, conversely, from the inside to the outside. It is referred to as volumetric heating [24]. Other investigations have also explored the influences of the processing requirements, the addition of second phases such as Al_2O_3 or Nb_2O_5, and the incorporation of Y_2O_3 stabilizers on the behavior of LTD for dental materials sintered by the traditional method [25, 26].

Nevertheless, no comprehensive research has been conducted on the effect of microwave sintering on 3Y-TZP/Al_2O_3 composites when exposed to LTD conditions.

The aim of this research was to assess the impact of microwave sintering on the LTD resistance of dental materials based on zirconia-alumina nanocomposites by comparing them with materials sintered by the traditional technique. This includes the evaluation of surface roughening, monoclinic phase transformation progression, as well as the mechanical properties as a function of the degradation time in simulated laboratory settings.

2. Materials and methods

A commercial zirconia-alumina powders (ATZ) from the company Tosoh (TZ-3Y20A, Tosoh, Tokyo, Japan) have been studied in this work. Specifically, it is composed of 80 wt% of 3Y-TZP and 20 wt% of Al_2O_3.

The specimens have been pressed uniaxially (50 MPa of pressure load) with a universal testing machine (Shimadzu AG-X Plus) to obtain circular shapes of 10 mm (diameter) and 3 mm of height. All green samples had a geometric density of ~55% in relation to the theoretical density.

The sintering of the samples was carried out using two different techniques: microwave technology (a non-conventional fast technique) and conventional oven. Microwave sintering (MW) has been performed in a single-mode circular cavity microwave oven operating in the TE_{111} mode with a resonant frequency of 2.45 GHz (**Figure 1**, [27]). The microwave sintering conditions have been 1300°C of maximum temperature during 10 min of dwell time and 50°C/min of a heating rate. It is worthy of being mentioned that the zirconia-alumina composites are poorly microwave-absorbent materials at low temperatures, since its dielectric loss factor at room temperature is less than 0.01 [28]. This fact makes hybrid heating necessary, using silicon carbide as a susceptor [29, 30]. The sample temperature is controlled by an infrared radiation pyrometer (Optris CT-Laser GH5, 5 μm), which focuses on the sample surface through the tiny circular opening in the cavity wall. The emissivity and transmissivity of the final temperature material are determined prior to sintering test.

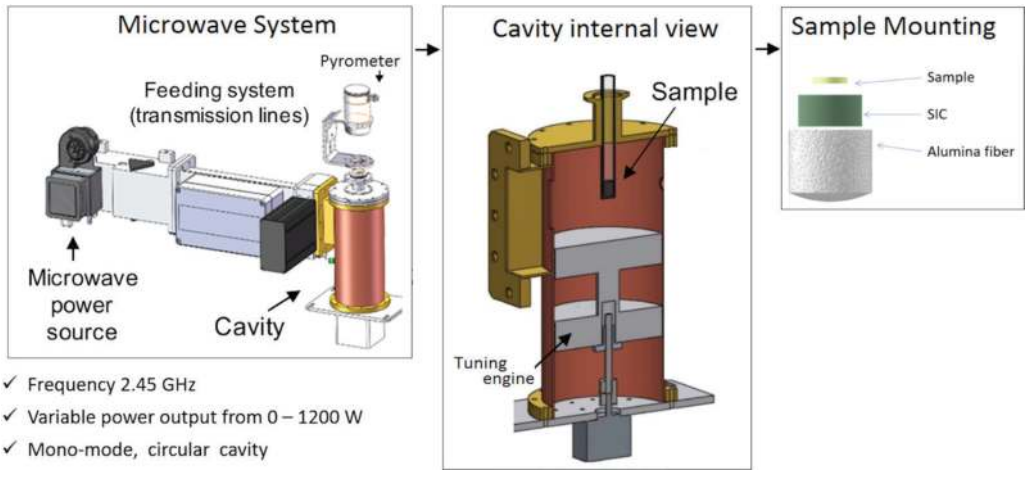

Figure 1.
Details of the experimental microwave system of 1 kW at 2.45 GHz connected to a mono-mode circular cavity.

Conventional sintering (CS) has been performed in an electric oven (Carbolite Gero, HTF 1800) for 120 min in atmospheric conditions at 1500°C and a heating rate of 10°C/min. Both the MW and CS sintering conditions have been extracted from a previous study, where their main mechanical and microstructural properties were analyzed [31]. These chosen settings are based on previous research by our group, in which the sintering conditions of zirconia-based materials were optimized [32, 33].

The study of low-temperature degradation (LTD) of ZrO_2-Al_2O_3 materials has been developed using procedures that simulate and intensify the process of hydrothermal aging. The samples are then autoclaved (MARK) with steam at 125°C and 1.6 bar [19].

The aging samples are characterized after each 20 h of exposition to LTD until they reach 200 h. This characterization consists of the following:

a. quantification of phase content by Raman spectroscopy (Horiba-MTB Xplora). The linear model suggested by Lim et al. is utilized to calculate the content of the monoclinic phases (m) in the samples [34]:

$$V_m = \frac{I_m^{181} + I_m^{190}}{0.33 \cdot \left(I_t^{147} + I_t^{265} + I_m^{181} + I_m^{190}\right)} \quad (1)$$

where V_m is the volume fraction of monoclinic phase and I is the integrated peak intensity (low peak area). The intensities of the t-phase are 147 and 265 cm^{-1}, while those of the m-phase are 181 and 190 cm^{-1} [35].

b. assessment of the topography and roughness (Ra) of the surface characterized by atomic force microscopy (AFM Multimode, Veeco) for which samples were mirror polished with diamond paste before sintering, so that the changes in topography caused by the transformation of degradation can be effectively assessed. Two topographic photos have been obtained for every specimen: an image with a scan area of 1 μm × 1 μm for a close-up view of the grains and a second image with a scan area of 5 μm × 5 μm to provide a representative roughness value.

c. microscopic analysis of damaged surfaces by Nomarski optical microscopy. Nomarski microscopy is another method to visualize the specimen's topography in three dimensions.

d. evaluation of their mechanical characteristics (hardness and Young's modulus) according to the nanoindentation technique. The method used is a nanoindenter (G-200; Agilent Technologies). Tests were carried out with a Berkovich tip calibrated with silica standard and operated at a maximum depth of 2000 nm. The continuous stiffness measurement was used to determine the contact stiffness and calculate the hardness profiles and elastic modulus [36]. A matrix with 25 indentations was made for every material.

3. Results and discussion

3.1 Microstructure and phase transformation

The relative densities of sintered composite obtained by microwave technology at 1300°C with 10 min of dwell time (MW1300_10) and conventional oven at 1500°C

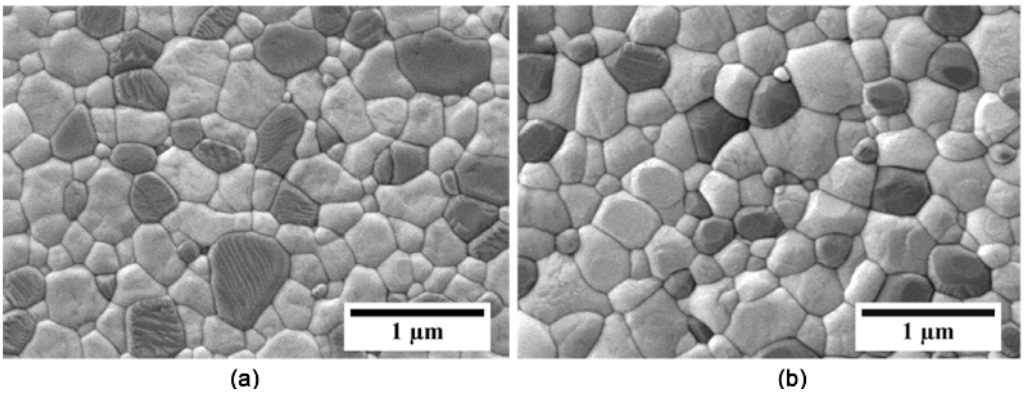

Figure 2.
FE-SEM micrograph of ATZ composites under different sintering conditions: (a) MW1300_10 and (b) CS1500_120.

with 120 min of dwell time (CS1500_120) were around 99.8 and 99.2%, respectively, and its grain sizes below 500 nm (**Figure 2**).

The relative density is calculated from the theoretical density of the ATZ sample, 4.89 g/cm^3. The results indicated that all specimens reached a high degree of densification. It should be noted that the samples sintered by MW at 2.45 GHz exhibited a relative higher density than samples sintered by CS, achieving 99.8% for MW1300_10. It is important to note that the sintering temperatures and holding times employed in microwave sintering are considerably lower than in a conventional process. In conclusion, while the sintering time needed to achieve relative densities above 99% with conventional sintering is 350 min, microwave technology produces denser samples in only 35 min. It should be noted that the final economic cost is considerably reduced due to the decrease in processing time and energy consumption and, consequently, the environmental impact also decreases. Therefore, microwave technology is considered to be a more environmentally friendly technique than conventional sintering.

The FE-SEM micrographs of the MW and CS densified ATZ composite are shown in **Figure 2**. The samples are very dense and have a high homogeneity, since the alumina grains (the darker ones) are uniformly dispersed in the zirconia matrix. These results are consistent with the relative density values. The darker grains correspond to alumina, while the lighter grains are zirconia. The average grain sizes of zirconia and alumina have been measured from their micrographs. The grain size increases with residence time and sintering temperature.

As for the sample densified by (MW1300_10), the average grain size of ZrO_2 and Al_2O_3 reached approximately 280 and 400 nm, respectively. In the case of the sample sintered by conventional furnace, the evolution of the average grain sizes was similar to that of MW; the higher the sintering temperature, the larger the grain size. In sample HC1500_120, the grain sizes of ZrO_2 and Al_2O_3 were 330 and 450 nm, respectively [31]. Wu et al. [37] researched the effects of Al_2O_3 addition in 3Y-TZP on the mechanical properties and microstructure of the composite. The increase in alumina content favored slightly the grain growth during the densification. Thus, the average grain size of ZrO_2 is slightly higher than that reported in the literature [38].

Raman spectra for conventional and microwave-sintered ATZ composites are shown in **Figure 3**. It is possible to check the phase transformation by Raman spectroscopy, thanks to the characteristic doublet of the *m*-phase at 181–190 cm^{-1}.

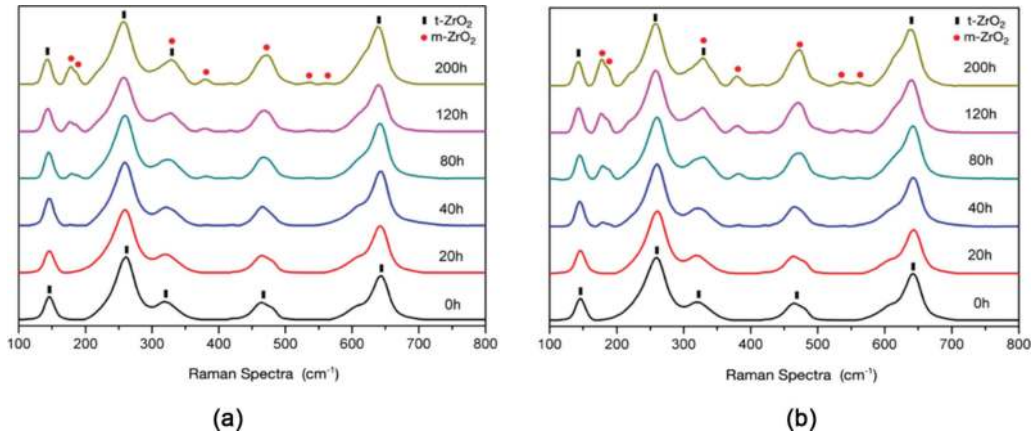

Figure 3.
Raman spectrums of different LTD times of exposure for sintered ATZ material by (a) MW1300_10 and (b) CS1500_120.

As can be seen, no monoclinic phase peaks are present in any of the samples after sintering (0 h of exposure to LTD) and also after 20 h of LTD exposure. As ATZ samples are exposed to LTD for longer, the intensity of the monoclinic phase peaks increases. After 40 h, such peaks can be recognized for sample CS1500_120, while for MW1300_10, they appear after 80 h. As the degradation time increases, the transformation becomes rather important with a distinct presence at 181–190 cm^{-1}.

If both sintering methods are compared, differences in the intensities of the doublet characteristic of the monoclinic phase can be observed between the spectra. The monoclinic peaks are higher in the sample CS1500_120. This fact suggests a greater vulnerability to transformation induced by LTD in conventionally sintered samples than by microwave technique [39].

The phase transformation can be quantified from the volume fraction of the m-phase, Vm, measured by Raman spectra (Eq. 1). The results of the quantification of Vm are shown in **Figure 4**.

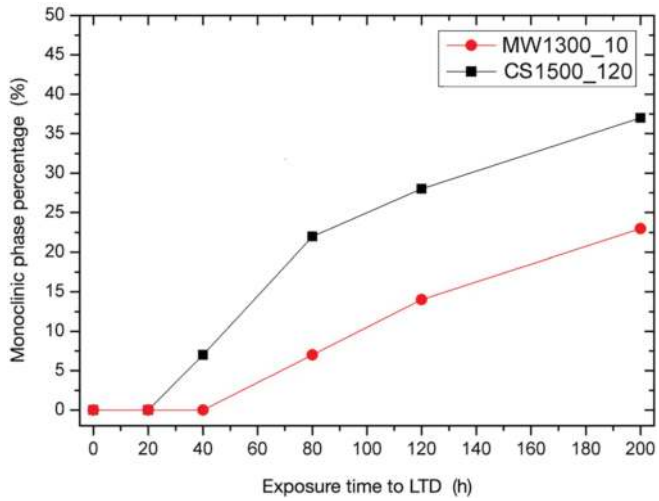

Figure 4.
Volume fraction of the m-phase, Vm, a result of the LTD exposure time for ATZ material in either sintering conditions (MW1300_10 and CS1500_120).

The lines show differences in the kinetics of the phase change. The MW1300_10 sample sintered by MW degrades more slowly compared with the conventionally densified CS1500_120 sample. After 200 h of degradation, Vm is 24% for MW1300_10 and 37% for CS1500_120. Therefore, the sample obtained by microwave is less susceptible to LTD.

After these results, it is verified that adding alumina to zirconia, forming an ATZ composite causes the zirconia to degrade more slowly compared with monolithic zirconia. Presenda et al. concluded that Y-TZP transforms approximately 90% after 100 h of testing under the same conditions as this work [17]. Ultimately, the resistance to aging is increased with the addition of alumina in the material composition.

3.2 Topography and surface roughness

A volume increases of about 3–4% accompany the t- to m-phase transformation of ZrO_2-based composites. **Figure 5** shows the AFM images of the ATZ composite sintered by CS and MW, where it is possible to analyze the surface changes induced by LTD exposure. The average surface roughness, Ra, has also been identified as a way to measure this variation.

As the exposure time increases, the topography becomes more irregular, increasing the surface roughness. After 200 hours of exposure to LTD, the surfaces of the samples tested are found to have increased in roughness, with the appearance of bulging.

In purpose of comparing the roughness of the samples sintered by different sintering methods, the Ra values at different exposure times have been determined. These values are presented in **Figure 6.**

Sample MW1300_10 has given lower Ra values than CS1500_120, showing greater variability in rugosity, particularly beyond the first 20 h. A strong increase

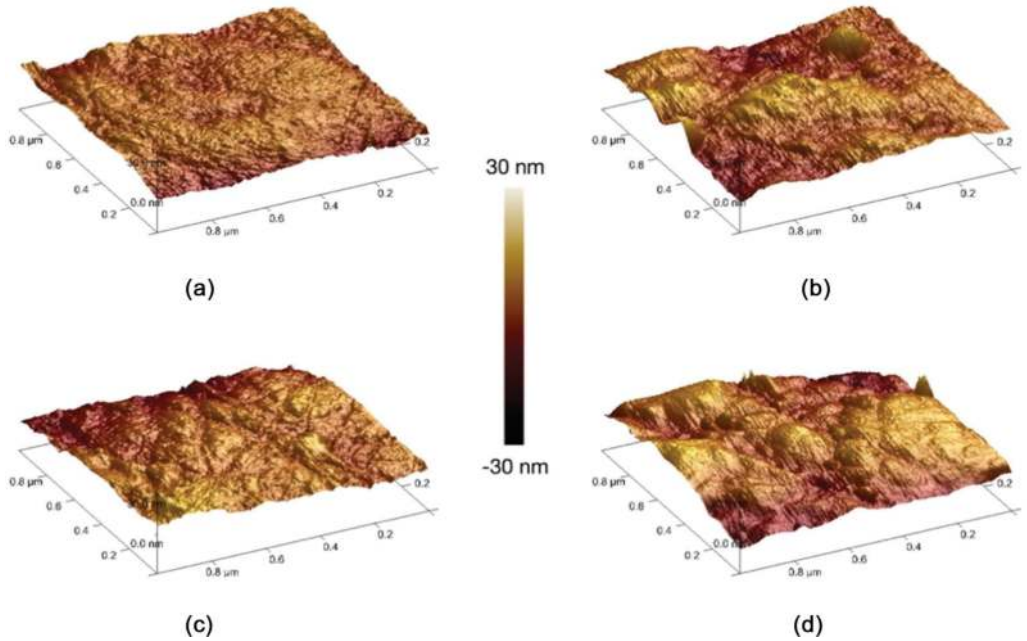

Figure 5.
AFM topographic images of ATZ composite at various exposure times of LTD for MW1300_10: (a) 0 h, (b) 200 h; and CS1500_120: (c) 0 h and (d) 200 h.

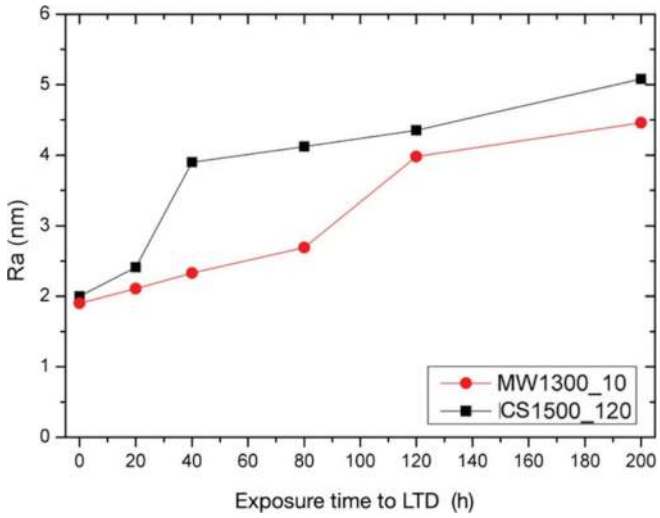

Figure 6.
Average surface roughening at various LTD times for each specimen.

is observed after the first 20 h for CS1500_120, reaching about 5.1 nm after 200 h of LTD. However, it was not until after 80 h that a sharp change in Ra was observed in sample MW1300_10. This performance suggests that the more meaningful topographical changes vary depending on the sintering method.

These results are consistent with the transformation of the *t*-phase to *m*-phase according to the Raman spectra obtained (**Figure 3**); because after 80 h, the monoclinic peak begins to be observed in the Raman spectra for the sample MW1300_10, which is when the sudden jump in the Ra values appears. The same happens for the sample CS1500_120.

Although in the Raman spectra of both samples there are no monoclinic peaks until after 20 h of LTD exposure, by observing the surface changes at the submicrometric scale with AFM, it can be seen these microstructure changes are occurring before the peaks appear. As the exposure time of the samples to LTD increases, the irregularities in the surface of the samples also increase because of the push of the grains to the surface to explain the expansion in volume.

3.3 Nomarski optical microscopy

Nomarski optical microscopy is another technique used to study degraded surfaces. **Figure 7** shows the micrographs of the surfaces of the samples just after sintering (at 0 h) and after 80 h of LTD time of exposure.

The effects of degradation appear as surface roughness by volume due to phase transformation. After 80 h, changes are already observed on the surface, where an increase in roughness is observed. It is true that the irregularities, as well as the phase transformation, are lower than those observed in monolithic zirconia samples, such as, for example, Y-TZP [17]. Being the yttria in both materials the dopant of zirconia, the addition of alumina in the composition is what makes the aging of zirconia difficult.

3.4 Effect on mechanical properties

After exposure to LTD, the structural quality of the material is evaluated, analyzing the hardness and Young's modulus, depending on the aging time. The tests were performed on the polished surfaces that were exposed to LTD. The values are shown in **Figure 8**.

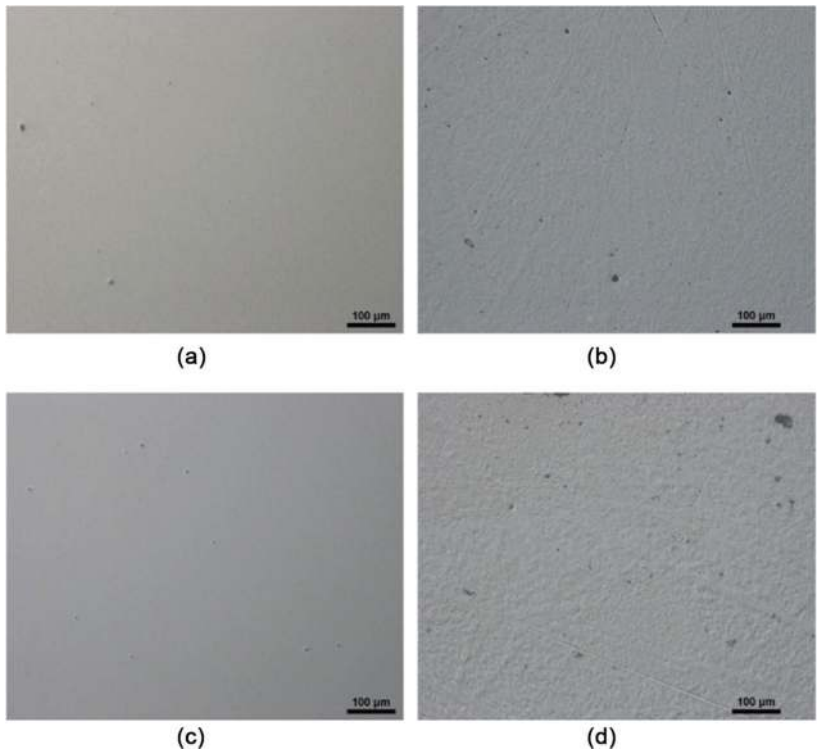

Figure 7.
Nomarski microscopy images of ATZ composite in various exposure times LTD for MW1300_10: (a) 0 h, (b) 80 h; and CS1500_120: (c) 0 h and (d) 80 h.

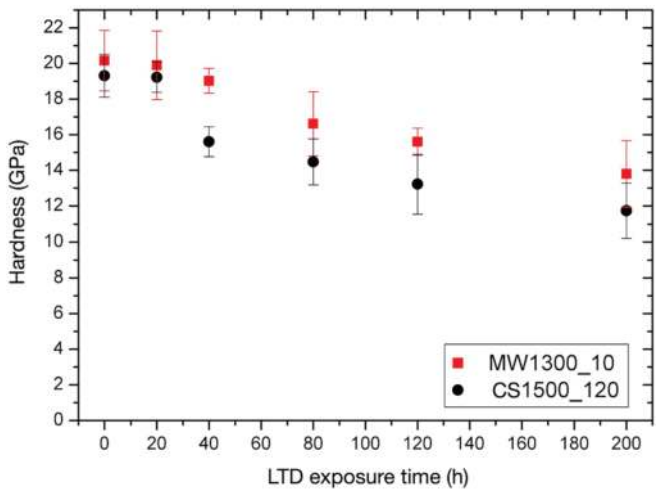

Figure 8.
Hardness values for samples as a function of the LTD time of exposure.

As can be seen, the hardness is affected differently by the sintering method used, with the sintered sample offering more resistance to wear by MW, which corroborates the results obtained in the other techniques. After sintering, before exposure to LTD, the samples show a hardness value of 20.3 and 19.5 GPa for MW1300_10 and CS1500_120, respectively. At 40 h, the hardness for CS1500_120 drops sharply to 15.7 GPa. However, the sample MW1300_10 decreases its hardness more slowly,

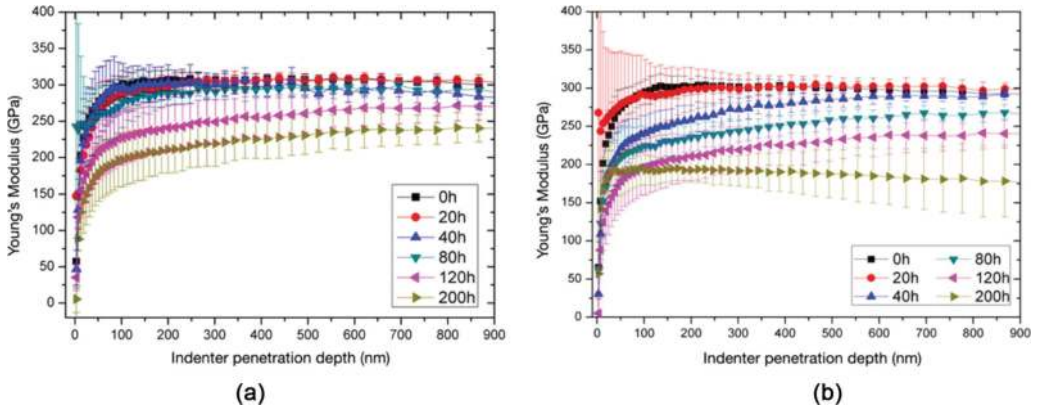

Figure 9.
Young's module values for various LTD exposure of times for sintered ATZ composite by (a) MW1300_10 and (b) CS1500_120.

following a less pronounced trend. After 200 h, the hardness values obtained are 13.8 and 11.9 GPa for MW1300_10 and CS1500_120, respectively. These results show that MW1300_10 resists better the exposure to hydrothermal degradation.

Figure 9 shows Young's modulus values for the composite densified by both sintering methods.

Like hardness, Young's modulus (E) is affected after exposure to LTD. The E values are modified in both samples, although for MW1300_10 to a lesser extent than CS1500_120. The first significant change in the E-value appears at 40 h for CS1500_120, while in sample MW1300_10 such change is not observed until after 80 h, corroborating the results obtained previously. After 200 h, the average values of Young's modulus are 237 and 170 GPa for MW1300_10 and CS1500_120, respectively.

So far, a number of studies have concentrated on the understanding of the LTD process and its effect on 3Y-TZP dental materials sintered *via* conventional methods [40]. The investigation has also explored the influence of processing conditions, the incorporation of other components such as Al_2O_3 and the addition of the stabilizer Y_2O_3 in the LTD [41, 42].

Nevertheless, no comprehensive study has been conducted on the influence of microwave sintering on ATZ materials exposed to LTD environments. Since the sintering process and its associated conditions are a critical factor to determine the LTD behavior of 3Y-TZP-based materials, it is very relevant to research the effect of microwave sintering techniques on this behavior.

Presenda et al. [17] corroborate the high resistance to LTD of the microwave-sintered dense zirconia materials (3Y-TZP) as E and H remain almost unaltered; even after 140 h, E is still around 250 GPa and H is approximately 15 GPa. The starting material also has an important role in the microstructure evolution during sintering and, thus, in the LTD resistance. Microwave-sintered 3Y-TZP does not show any significant evidence of degradation after 200 h of exposure to LTD.

4. Conclusions

Microwave sintering has a very noticeable effect on the properties of ATZ material and therefore on their susceptibility to degradation at low temperatures. The ATZ composite has been obtained with densities close to theoretical densities by

microwave heating, considerably reducing sintering times compared with the conventional sintering method. Microwave heating is accompanied by modification of the densification mechanisms, giving the ATZ material excellent mechanical properties.

In sample MW1300_10, the amount of the monoclinic phase is lower than for CS1500_120 after the same hours of exposure to LTD conditions. Therefore, the non-conventional fast technique of microwave sintering is a great alternative since it requires lower temperatures and times for densification, giving materials more resistant to low-temperature degradation.

The addition of alumina to the composition makes the phase change more difficult, the ATZ composite being more resistant to degradation than monolithic zirconia material.

In summary, there are many variables that can influence the resistance to LTD, including the composition of the material, quantity and distribution of the stabilizer or the sintering mechanisms among others. In this work, the results obtained indicate that both the addition of alumina in the composition and the use of microwaves as a sintering method favor the resistance to aging, occurring at a lower speed. There is no earlier publication on the effect of microwave sintering on the behavior of the LTD on ATZ materials, so this work is an approximation to understand this phenomenon.

Acknowledgements

The authors gratefully acknowledge the funding support of the Spanish Ministry of Science and Innovation (RTI2018-099033-B-C32, RYC-2016-20915), Generalitat Valenciana (GRISOLIAP/2018/168) and Dr. Lorena Gil for contributing to the research work described in this chapter. We would also like to thank the researchers of the ITACA-UPV Institute for their contribution to the design and construction of the microwave equipment.

Conflict of interest

The authors did not report any conflict of interest.

Author details

Rut Benavente, Maria Dolores Salvador and Amparo Borrell*
Institute of Materials Technology, Polytechnic University of Valencia, Valencia, Spain

*Address all correspondence to: aborrell@upv.es

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