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Broadband dielectric characterization of TiO_2 ceramics sintered through microwave and conventional processes

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Abstract

In this work the microwave sintering (MW) of pure submicron rutile TiO_2 powder has been conducted in complete electric field using a single mode cavity of 2.45 GHz and without any susceptor. The sintering conditions were varied and similar sintering cycles were also done using a conventional furnace (CV), in carefully measuring the temperature in both processes. The dielectric properties, from kHz to GHz were determined and a comparison analysis was made between microwaved and conventional sintered specimens. It is shown that microwave sintering allows to obtain dense material (> 95%) in a very short time (10–15 min) at a sintering temperature ranging from 1000 °C to 1300 °C. Some samples are fully dense (> 99% theoretical density) after being microwave heated for ~10 min at ~1300 °C. Using the microwave heating, the processing temperature to get high dense material (i.e. > 94%) is lowered by ~150–175 °C compared to conventionally sintered samples. It is also shown that an annealing in air at ~800 °C for ~4 h, leads to very low loss TiO_2 ceramic in the entire frequency range investigated. Owing to the lowest sintering temperature provided by microwaves, the low frequency dielectric losses are smaller for MW samples than for CV sintered samples. Among the highest reported microwave Q factors (~7350) have been measured on pure TiO_2 samples exhibiting the largest grain size (~1.5 μm) and density (> 96%).

Keywords: Microwave processing; Dielectric properties; TiO_2

1. Introduction

Titanium dioxide (TiO_2) has been extensively studied in electronic applications including type I capacitors, Low Temperature Co-fired Ceramic substrate (LTCC) and varistors [1,2]. Because of its relatively high dielectric constant ($k\sim100$) at room temperature and high electric breakdown strength (BDS) (> 100 kV/cm), TiO_2 is considered as a potential dielectric material for high energy density capacitors applications [3]. Otherwise, titania is found in a wide range of dielectric resonator materials despite the fact that titania based ceramics are very sensitive to reduction [4]. In case of pure TiO_2, the reduction phenomenon may be described principally in terms of either the formation of oxygen vacancies, Ti^{4+} interstitials, Ti^{3+} interstitials, or oxygen vacancies and Ti^{3+} species in octahedral lattice sites [5]. In terms of crystal structure, TiO_2 exists in three forms: rutile, anatase and brookite and the two latter structures irreversibly convert to rutile in the temperature range of 700–900 °C [6,7]. In order to avoid any structural change through the thermal process, rutile TiO_2 has been investigated in the current study. As a consequence of the titanium mixed oxidation state (Ti^{3+}, Ti^{4+}), it is well established that TiO_2 dielectric and electric properties are quite sensitive to processing conditions such as oxygen partial pressure and temperature, time as well as the presence of impurities and dopants. For instance, sintering TiO_2 in H_2 atmosphere leads to a highly reduced Ti_nO_{2n-1} ($4\leq n\leq9$) compound referred to as Magneli phase in which nonstoichiometry results from shearing mechanism [8]. When TiO_2 is sintered at high temperature in air, partial reduction of Ti^{4+} into Ti^{3+}

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occurs to a lesser extent without significant stoichiometry change [9]. This partial reduction, associated with point defects formation (oxygen vacancies, interstitials, etc), induces semi-conduction behavior and loss factor degradation. For instance, Pullar et al. [9] showed that undoped TiO_2 sintered at high temperature ($\sim$1500 °C), exhibits high density ($>$98%) but low Qf product ($<$6000 GHz at 3 GHz), due to the partial Ti^{4+} reduction into Ti^{3+}. This partial reduction is often accompanied by a slight change of color of the sample, towards darker side (coring effect). Another study reports similar *Qf* value (*Qf* < 6000 GHz) of undoped TiO_2 sintered at 1500 °C in either air or in pure oxygen [5]. To overcome these drawbacks, the addition of divalent or trivalent cations, in particular, with appropriate ionic radii, has been already investigated [5,9]. For instance the addition of 0.05 mol% of Cu^{2+} in TiO_2 increases the Qf factor from 6000 GHz (undoped) to 32,000 GHz (Cu^{2+} doped) [5]. It has been proposed that the presence of M^{2+} in solid solution prevents Ti^{4+} from reduction by a compensation mechanism [5]. However, in case of applications involving high electrical field, such as high energy density capacitors applications, it may be detrimental to add such dopants. As a matter of fact, solute addition may cause undesirable effects such as electromigration, ageing phenomenon or leakage current, etc. especially under high electric field [10]. Therefore, keeping in mind the interest of TiO_2 in the field of high energy density capacitors, this work is focused on sintering pure rutile TiO_2. As explained above, TiO_2 properties are strongly correlated to processing conditions, therefore it would be interesting to study the microwave sintering of TiO_2 and to characterize its resulting dielectric properties in a large frequency range (from kHz to GHz). Surprisingly, only a few works report the likely specific effects of microwave sintering of pure TiO_2 [11–13]. In Ref. [11], a multimode microwave furnace was used to heat treat nano-sized TiO_2 powder at very low temperature ($<$450 °C) and the authors focus on phase transformation rather than densification. In Ref. [12], they succeeded in getting sintered TiO_2 ceramic with high density ($\sim$90% of the theoretical value), through a multi-mode microwave sintering process performed in argon at 1000 °C. They showed that microwaves enhanced densification behavior over conventional process but the dielectric properties were not measured. Ref. [13] depicts the TiO_2 phase transformation and densification behavior of anatase TiO_2 using a multimode microwave cavity equipped with SiC suceptors. They did not mention any dielectric properties either but they reported enhanced densification through microwave process over conventional sintering. Microwave sintering is quite well known to offer the ability to lower the sintering temperatures and durations required to achieve dense materials over conventional process [14,15]. As a result, the microwave sintering may lead to low intrinsic defect concentration and finer grain size in TiO_2. In previous works related to microwave sintering of TiO_2, multi-mode cavities equipped with susceptors employ a combination of electric field, magnetic field and radiant heating. Therefore, in this work the microwave sintering of pure submicron rutile TiO_2 powder has been conducted in pure electric field using a single mode cavity of 2.45 GHz and without any susceptor. In doing so, the specific effects of microwaves on TiO_2 sintering and dielectric properties could be clearly understood. In this study, the sintering and annealing conditions were varied. The dielectric properties, from kHz to GHz were determined and a comparison analysis is made between microwaved and conventionally sintered specimens. Identical heat treatments in both microwave and conventional furnaces were conducted so that a direct comparison between both processes can be made.

2. Experimental

The TiO_2 powder, used in this work, was a commercial grade which was produced by Ishihara Corp., USA (commercial label CR-EL). The chemical composition is given in Table 1. The powder has a specific surface area of 6.8 g/cm^2 and a particle size D50 of 0.25 μm. The starting powder was manually ground in an agate mortar to remove the agglomerate and disks were pressed by uniaxial pressing without binder (12.73 mm diameter × 2.5 mm thickness and 12.73 mm diameter × 5.0 mm thickness) at 90 MPa. The resulting compacts had a green density of approximately 56%, assuming a theoretical density of 4.23 g/cm^3. The single mode microwave sintering system consisted of a TE_{103} microwave rectangular cavity, working with a 2.45 GHz–3 kW microwave generator. The sample disks were placed at the center of a thermal insulation package made of light and porous fiberfrax (Duraboard® 3000 from Fiberfrax-Niagara Falls, NY) and then this package was loaded inside the cavity so that the disk was located at the maximum of the electrical field. The temperature was measured using a monochromatic infra-red pyrometer from Raytech (MA2SC working from 350 °C to 2000 °C), focused on the larger surface of the disk, in order to make sure that the spot size was smaller than the sample surface. Conventional sintering was conducted in a silicon carbide type resistor furnace in which temperature was measured by thermocouple (TC). In order to make sure that the temperature measured from the pyrometer can be directly compared to the one measured in the conventional furnace, a sintered TiO_2 sample has been conventionally heated up at 100 °C/h up to 1350 °C and the pyrometer was simultaneously used to record the relationship between the actual temperature (temperature given by the TC located in the vicinity of the sample) and the temperature coming from the IR pyrometer, focused on the TiO_2 sample. The sample was located close enough to the furnace refractory wall so that

Table 1
Chemical composition of the rutile TiO_2 powder from Ishihara Corp., USA.

Compositions	TiO_2	Fe_2O_3	Al_2O_3	SiO_2	Na_2O
Content (at%)	99.97	0.0031	0.006	0.013	0.002

parasites infra-red radiations were supposed to be negligible. In doing so, a 'true' temperature has been systematically deduced from the IR pyrometer information for the samples being heated by microwaves. Systematically the corrected temperature is indicated through this study. The incident microwave power was manually tuned so that the sample was subjected to the desired temperature–time cycle. The pyrometer is connected to a PC equipped with software that allows temperature data recording. The sample apparent density was simply determined from the weight and the sample dimensions and microstructures were obtained using Scanning Electron Microscopy (Hitachi S-3000, Tokyo, Japan). Microstructures were observed on fracture surface and grain size was estimated using the intercepts method and the Mendelson's relation calculating the grain size G as $G=1.56L$, with L=average length between the intercepts [16]. For dielectric property measurement, the samples were electroded with DC-sputtered films (thickness $\sim$50 nm) of gold on both sides. Measurements of permittivity and loss as a function of temperature and frequency were determined using a precision LCR meter (Model HP 4284A, Palo Alto, CA) over temperature 25–180 °C and the frequency range 100 Hz–1 MHz. The resistivity versus temperature relationship was estimated from the low frequency (100 Hz) permittivity and Loss factor measurements, assuming that the equivalent circuit is an R–C parallel dipole, and using the following equation: $R=1/\omega C\tan(\delta)$. In this latter, R is the value of the electrical resistance of the equivalent R–C parallel dipole. Using the sample dimension, the resistivity ρ is calculated. Permittivity at microwave frequencies was measured by the Hakki–Coleman dielectric resonator method using a vector network analyzer (Model HP 8510C, Palo Alto, CA) [17]. The quality factor (Q) of the sintered ceramic resonator was measured at a frequency of $\sim$4 GHz by a resonant cavity method using $TE_{01\delta}$ mode [18]. With the sample centered in a silver cavity, the S_{21} parameter on the network analyzer was measured. The resonant frequency of the $TE_{01\delta}$ mode was used for the calculation of Q values.

3. Results and discussion

3.1. TiO_2 coupling with microwaves and typical thermal cycle

A typical temperature–time curve recorded during microwave sintering of TiO_2 is shown in Fig. 1a. Once the microwave power is turned on (around 200 W) and the cavity length tuned (TE_{103} mode), the temperature steadily goes up. In roughly 15 min, the target temperature is achieved (here about 1300 °C) and by adjusting manually the incident power, this latter is stabilised with $\pm$3 °C uncertainty. The high temperature stage duration was fixed at 10 min and the parameters (incident power and tuning plunger position) were then adjusted in order to cool down the sample to RT in about 30 min. This temperature increasing profile clearly indicates that the TiO_2 material easily couples with microwaves and that it can be directly

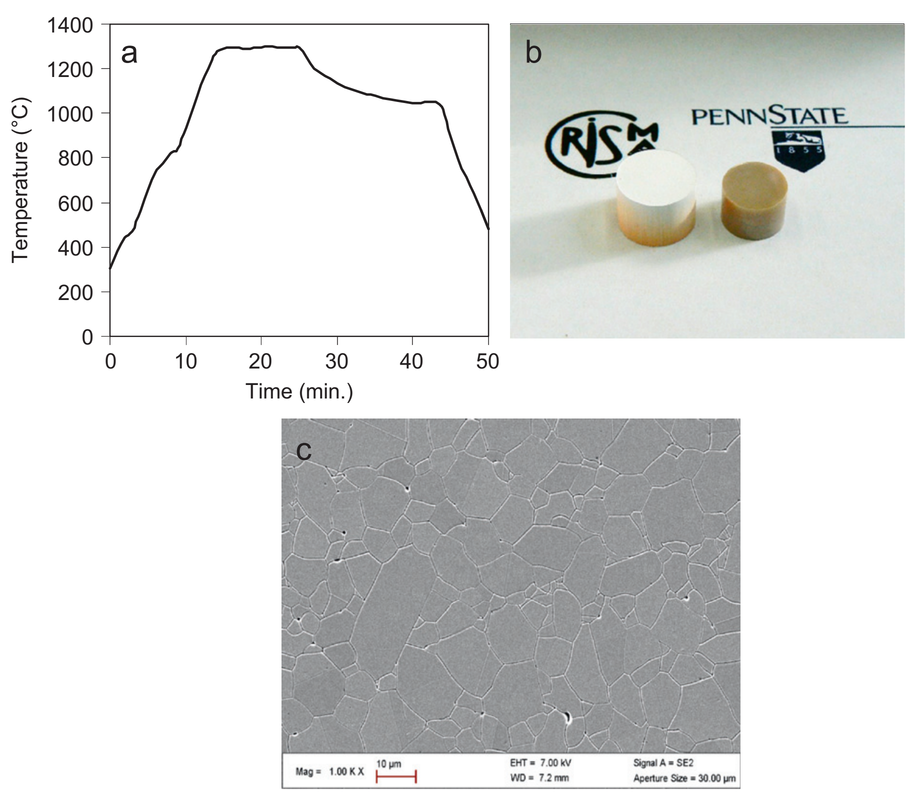

Fig. 1. (a) Typical temperature versus time cycle recorded during direct microwave sintering of TiO_2, (b) picture of the green and MW sintered sample and (c) SEM microstructure showing a density >99%.

heated up at various temperatures through microwave heating. After being subjected to the thermal cycle depicted in Fig. 1a, the TiO_2 sample exhibits a very high density of $>99\%$ and its color has turned from white to shiny 'yellowish brown' (Fig. 1b). As testified by a typical SEM microstructure (Fig. 1c), the sample is at least 99% dense with very little inter-granular porosity, a grain size of $>10\ \mu m$, and a quite large grain size distribution. Abnormal grain growth has likely occurred since both small and big grains are observed. Keeping in mind the goal to get low loss TiO_2 materials with fine grain size, an improved sintering temperature profile must be searched: lower the sintering temperature is, finer grains and lower loss are expected [9].

3.2. Relationship between microwave sintering cycle, densities and subsequent dielectric properties

Different disk samples (12.73 mm diameter × 2.5 mm thickness) were sintered according to the thermal cycle of Fig. 1a, with sintering temperatures ranging from ~940 °C to ~1280 °C. The resulting apparent densities are plotted in Fig. 2A. As the sintering temperature goes up, the density monotonically increases, from ~86% to ~99% of the theoretical value. The sintering time being fixed at 10 min, sintering temperatures higher than 1070 °C lead to densities higher than 95%. Some microwave sintering experiments were also done at a lower temperature (i.e. 940 °C) for different sintering durations, ranging from 5 min to 1 h. The densities obtained are plotted in Fig. 2B. At this low sintering temperature, 60 min dwell sintering time is needed to achieve a density close to 95% while 30 min dwell time leads to ~92% of density. All samples having the highest densities ($\geq 95\%$) were subsequently characterized for dielectric properties versus temperature from 100 Hz to 1 MHz. Selected room temperature and low frequency (100 Hz) dielectric data are summarized in Table 2. At the exception of the sample sintered at low temperature (940 °C), which has a RT ε of 104 (at 100 Hz), no other sample exhibits the expected relative permittivity (polycrystalline TiO_2 is supposed to have an ε value around 100 [9]). The samples sintered at 1075 °C, 1145 °C and 1210 °C, have respective RT permittivities of 211, 393 and 344. These values correspond to an additional space charge polarization that is correlated to the high dielectric losses that have been measured for all samples sintered at $T \geq 1075$ °C. The RT tan δ values at 100 Hz are ~4.28%, 7.5% and 12.2% for samples sintered at 1075 °C, 1145 °C and 1210 °C, respectively. The general trend is that dielectric losses increase with increasing the sintering temperature. Fig. 3a shows tan δ against temperature and frequency for the sample sintered at ~1075 °C-10 min. The temperature dependent loss peaks move to lower temperatures, indicating an activated conduction process that is related to titania reduction. The tan δ value is higher than 4% in the entire range of temperature and frequency investigated. It can be indeed noted that the sample sintered at the lowest temperature (940 °C) exhibits the lowest tan δ value (~0.69%) at RT and low frequency (100 Hz). Otherwise, its plot against temperature (Fig. 3b) exhibits a more expected trend for dielectric materials: tan δ factor monotonously increases with increasing temperature and tan δ goes down with increasing frequencies. To clearly state about this semi-conduction behavior which appears with increasing processing temperatures, the resistivity of all dense samples has been plotted against $1/T$ (Fig. 4). It is clearly seen that all samples have a typical semi-conducting type trend, the resisistivity decreases with increasing temperature. It is also noticeable that for sintering temperature increasing from 940 °C to 1280 °C, the room temperature resistivity goes

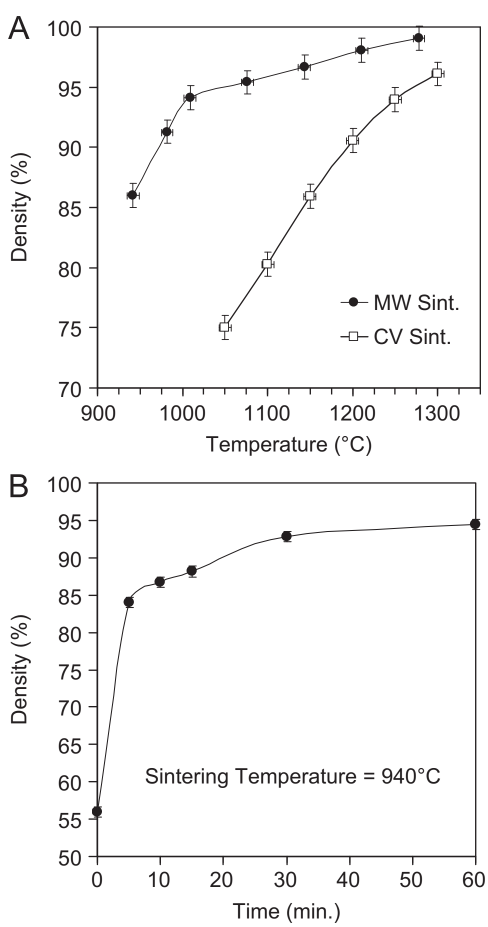

Fig. 2. (A) Apparent densities versus sintering temperature of samples microwave sintered for 10 min (in bold circle) and conventionally sintered (square plot) and (B) apparent densities of samples microwave sintered @940 °C versus dwell time.

Table 2
Selected dielectric data of samples sintered through one step microwave heating cycle.

Process	Microwave sintering—one step cycle			
Temperature (°C)	940	1075	1145	1210
Sint. time (min)	60	10	10	10
Density (%)	94.4	95.4	96.7	98.1
ε @ RT, 100 Hz	104	211	393	344
Tanδ (%)	0.69	4.28	7.5	12.2

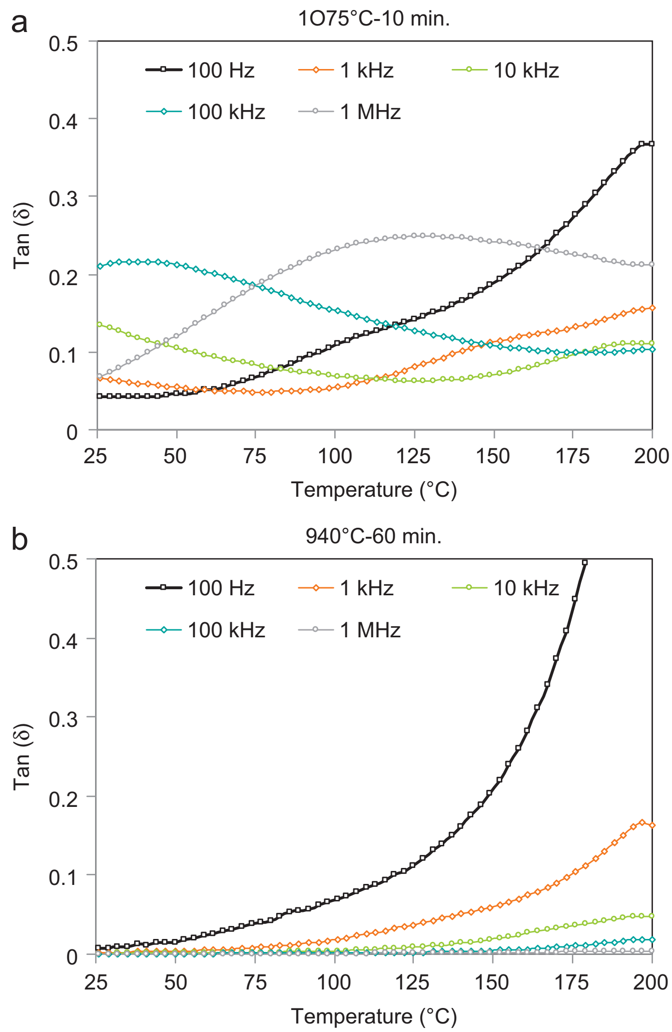

Fig. 3. tan δ versus temperature and frequency for samples microwave sintered at 1075 °C-10 min (a) and at 940 °C-60 min (b).

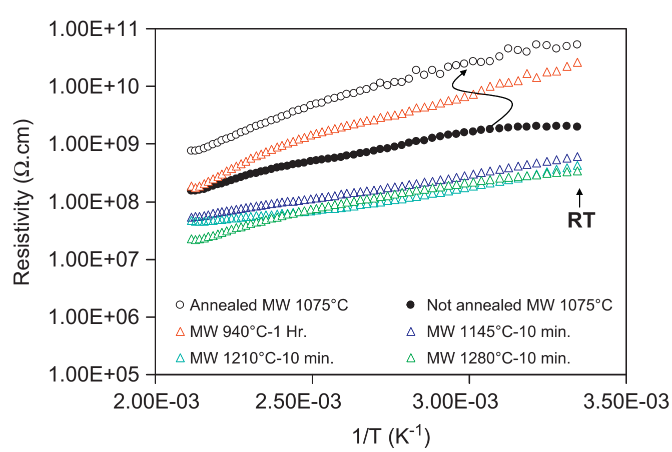

Fig. 4. Resistivity of TiO_2 (Ω cm) versus $1/T$ (K^{-1}) against the sintering and annealing conditions.

down by two orders of magnitude (2.6×10^{10} Ω cm to 3.39×10^{8} Ω cm). The above results show that as the sintering temperature increases, the defects concentration (oxygen vacancies, Ti^{3+} interstitials, etc.) associated with Ti^{4+} reduction increases, which leads to semi-conduction behavior. This observation is in good agreement with the usual and well known fact that defects concentration increases with increasing temperature. However, in case of rutile TiO_2, Pullar et al. [9] showed that undoped TiO_2 Q factor, measured at GHz, can be improved after annealing the ceramic at a very high temperature (1500 °C) for 10 h in O_2 or air, using a conventional process. It is consistent with other from defect chemistry arguments that sintered TiO_2 annealing will decrease oxygen defect concentration, but from the thermodynamic point of view, this annealing should likely be performed at low temperature. In the following section, samples have been heated up through two steps thermal cycle, including an annealing at 800 °C for 4 h in air. It is expected that this annealing temperature is high enough to decrease the defects concentration without drastically changing the microstructure (grains size and density). Otherwise, keeping in mind the interest of having small grains size, low defects concentration, and high enough density while taking advantage of the fast sintering process, the sintering temperature of 1075 °C has been selected for the following investigation. Fig. 5 shows both thermal cycles recorded during microwave sintering of titania, with and without thermal annealing. In terms of density, both samples (with or without annealing) have a similar density of ~95% but their dielectric properties are far different. The resistivity versus 1/T of the annealed sample is plotted in Fig. 4. It is observed that the resistivity has increased by more than one order of magnitude over the entire temperature range investigated for the sample annealed compared to the un-annealed sample. The resistivity at room temperature of the annealed sample is $\sim5.25\times10^{10}$ Ω cm whereas it is $\sim2\times10^{9}$ Ω cm for the unannealed one. The direct comparison between the loss factor of both samples (see Figs. 3a and 6a) clearly indicates that the annealing increased the insulation resistance of TiO_2. The loss factor measured at RT and 100 Hz of TiO_2 sintered at 1075 °C, varies from 4.28% (un-annealed) to 0.33% (annealed). In addition, the permittivity is between 90

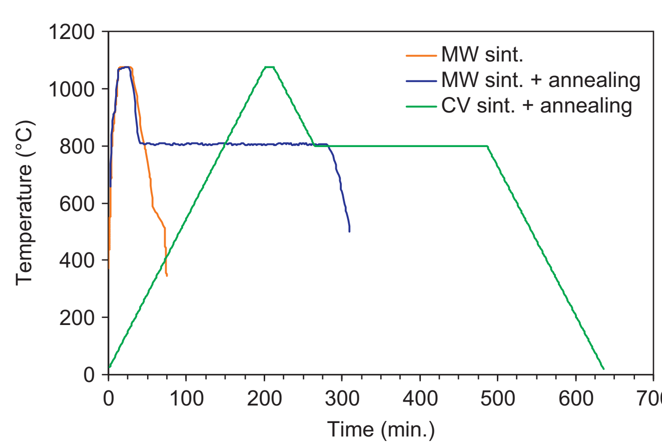

Fig. 5. Typical thermal cycles recorded during microwave sintering, microwave sintering +annealing and conventional sintering +annealing.

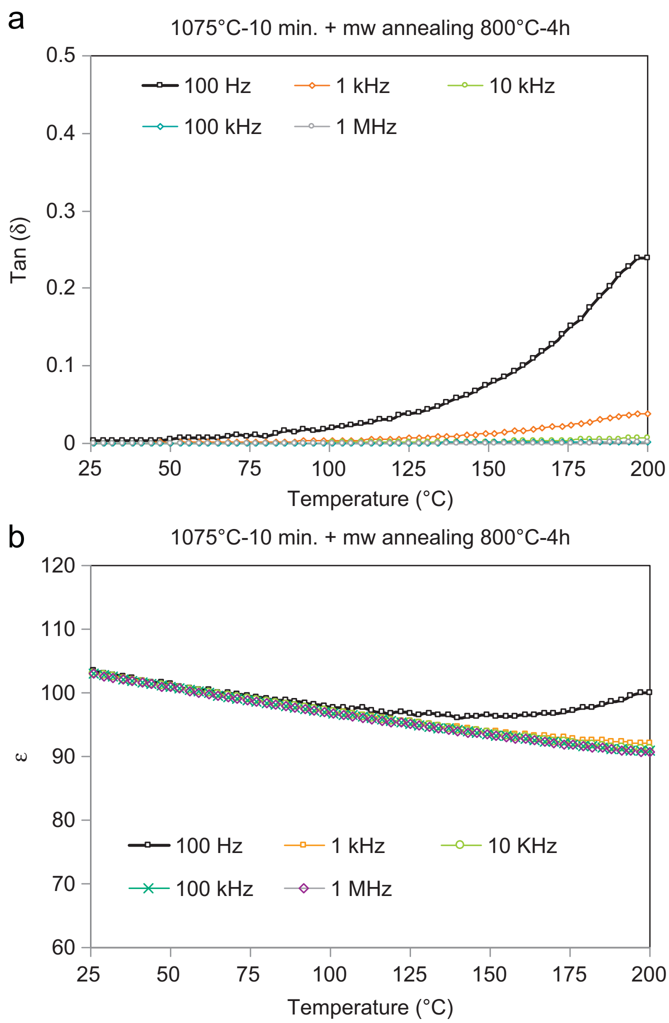

Fig. 6. (a) and (b) Loss factor and permittivity against temperature for sample microwave sintered @1075 °C-10 min and annealed @800°C-4 h.

and 104 within the investigated temperature range, which is the expected trend for not reduced titania (Fig. 6b). After going through different thermal cycles, it has been clearly discovered that the annealing at ~800 °C-4 h in air improves the dielectric properties. The next step is to apply this two steps thermal cycle in both conventional and microwave heating and to compare the subsequent dielectric and microstructures (see typical temperature profiles Fig. 5).

3.3. Microwave sintering versus conventional sintering: microstructure and dielectric characterization

Fig. 2A shows the plot of the apparent density versus sintering temperature and the process. It is clearly shown that a sintering temperature higher than 1200 °C (the dwell time being fixed at 10 min) is required to get a density higher than 90% using the conventional process. Using the microwave heating, similar densities are obtained at processing temperatures lowered by ~150–175 °C. These results confirm those reported in Ref. [12] which shows that 90% density is achieved with a microwave sintering temperature of 1000 °C on TiO_2 powder with an average particle size of ~20 nm. Fig. 7 shows typical microstructures of microwave samples sintered at 1075°C, 1010°C and conventionally sintered at 1250 °C. It is remarkable that the conventionally sintered sample at 1250 °C has a microstructure (density ~94.9% and grain size ~1 µm) that is similar to the microwave sintered sample at 1075 °C (density ~95.4% and grain size ~1 µm). The sample microwave sintered at 1010 °C has still a quite high density (~94.1%) and a grain size slightly lowered (~0.910 µm). Therefore, the densification is undoubtedly enhanced through microwave processing but in the frame of this study, grain growth has not been drastically reduced. Mazaheri et al. [19] have discussed the densification of titania nanoceramic using Two Steps Sintering cycle (TTS), including both a dwell at 800 °C for 1 h and a second step at 700 °C for 25 h. Using this TSS cycle and

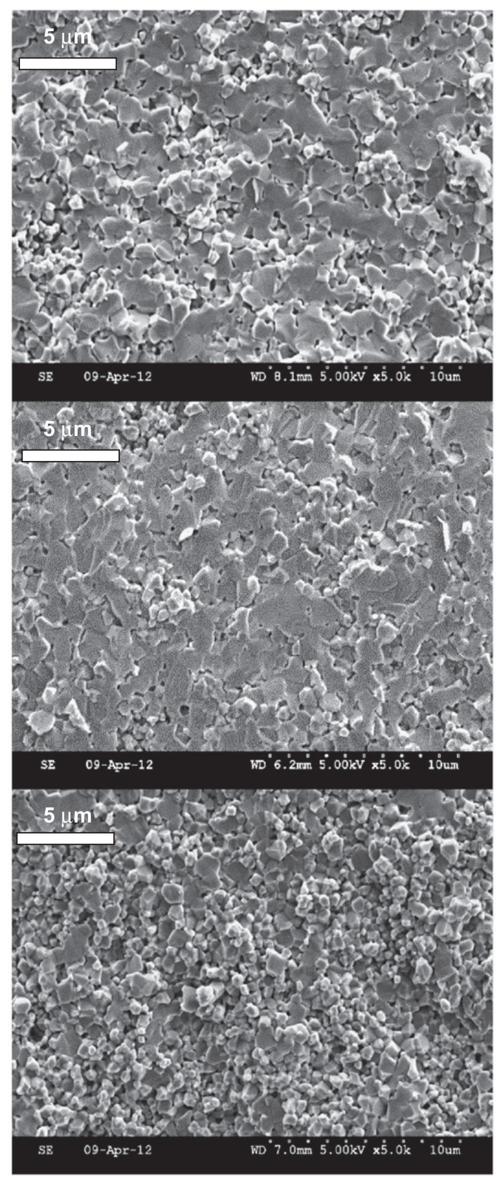

Fig. 7. Microstructures of samples conventionally sintered @1250 °C and microwave sintered @1075 °C and 1010 °C (from top to bottom).

starting from nano-sized TiO_2 particles, ranging from ~11 nm to ~27 nm, they succeeded to get highly dense titania (>98%) and grain size ~250 nm. They claim that due to the very low mobility of triple junction at low temperatures, the grain boundary mobility is therefore inhibited and the TSS process leads to ultrafine microstructures. The investigation of TSS sintering starting from nano-sized particles (~20 nm instead of 250 nm as obtained here) and using our as-designed microwave process could be an interesting perspective of the present work. Table 3 summarizes the density obtained, the grain size and the dielectric properties measured (at 100 Hz and ~GHz) on samples having densities higher than 94%, as a function of the processing temperature (for both processes). Both processes lead to similar dielectric properties at GHz frequency when grain size and density are close (see MW 1075 °C/CV 1250 °C and MW 1210 °C/CV 1300 °C). There is also a general trend in which the Q value increases with sintering temperature for both the microwave and conventionally sintered samples and Q increases with density for both sintering routes. These results are consistent with earlier work in the Q value of alumina correlates with the amount of porosity in a ceramic [20]. The highest Q factor (Q~7350 at ~4.4 GHz) is obtained for samples sintered at the highest temperature (MW 1210 °C and CV 1300 °C), i.e. for samples having the highest grain size (~1.5–1.7 μm). It is remarkable that this Q factor is among the highest value ever reported for undoped rutile TiO_2 [5,9], this shows the beneficial effect of the 800 °C annealing. The Q factor of pure and dense TiO_2 is indeed often lower than 2000 [5] because of the partial Ti^{4+} reduction. Otherwise, the Q factor is well correlated to the grain size: the higher the grain size, the higher the Q factor (whichever process is used). It is often reported that lowering of the grain boundary area leads to decreasing anharmonic vibration modes and as a result dielectric losses are lowered [21]. All microstructural parameters being almost similar (grain size and density), the Q factor is similar whatever the process used—this is not the case for low frequencies dielectric losses. It is clearly seen on the Table 3 that the tan δ measured at very low frequency (100 Hz) is significantly lower in case of microwave sintered samples than for samples conventionally sintered. Several microwave sintered samples, processed at temperatures below 1200 °C, indeed exhibit tan δ (at 100 Hz) in between 0.33% and 0.5% whereas conventional sintered samples have loss factor (at ~100 Hz) ~1.4–1.5%, all micro structural parameters being almost similar. It is also noticeable that the sample microwave sintered at the highest temperature (~1210 °C) and annealed at 800 °C for 4 h has quite high low frequency dielectric losses (tan δ ~4%). It is believed that due to both the highest density (almost 99% dense) and largest grain size (~1.72 μm) on this sample, the oxygen diffusion rate through grain boundaries is lowered [22]. As a result, the annealing process has not allowed this sample to be re-oxidized enough. Low frequency dielectric losses are related to DC electrical conductivity and so point defect concentration is associated with Ti^{4+} reduction (Ti^{4+} interstitials, Ti^{3+} interstitials, or oxygen vacancies). It is so believed that MW processing in lowering the sintering temperature by at least ~150 °C, allows a significant decrease of point defect concentration and, as a result, the low frequency dielectric losses are significantly lowered, as shown in the presented data. This is true as far as the oxygen diffusion through the microstructure can occur fast enough.

4. Conclusion

A single-mode TE_{103} microwave cavity has been used to sinter TiO_2 ceramic in pure E field without any susceptor. It has been shown that TiO_2 can be heated up and sintered in a few minutes in the temperature range of 1000–1300 °C. Almost fully dense TiO_2 samples were obtained. By measuring carefully the temperature in both processes, the microwave process allows lowering the sintering temperature by at least 150 °C with respect to the conventional process. One high temperature microwave sintering stage (at $T > 1050$ °C) for 10 min is enough to obtain dense ceramics (>94% dense) but their dielectric properties are degraded due to Ti^{4+} partial reduction. It is clearly observed that a subsequent annealing performed at 800 °C for 4 h allows recovering satisfactory dielectric properties. Because of the low sintering temperature provided by microwaves, the low frequency dielectric losses are lowered in comparison with

Table 3
Broadband dielectric properties of TiO_2 microwave conventionally sintered and subsequently annealed in air at 800 °C for 4 h.

Process	Microwave sintering (MW)				Conventional sintering (CV)		
Temperature (°C)	1010	**1075**	1145	1210	1200	**1250**	1300
Density (%)	94.1	**95.4**	96.7	98.5	88.1	**94.9**	96.1
Grain size (μm)	0.910	**1.06**	1,12	1.72	< 800 nm	**1.05**	1.5
ε 100 Hz, RT	97	**102**	102	105.5	Low density	**96.6**	102.2
Tan δ 100 Hz	0.42%	**0.33%**	0.5%	4%		**1.5%**	1.4%
ε	95	**99**	104	106		**100**	104
@f (GHz)	@4.58	**@4.58**	@4.44	@4.42	not measured	**@4.50**	@4.46
$Q \times F$ (GHz)	9880	**19,280**	23,860	30,800		**17,955**	31,110
Q	2265	**4505**	5640	7351		**4185**	7356

the conventional process. This is due to the fact that defects concentration related to Ti^{4+} reduction is a thermally activated process. Otherwise, as the microwave properties are less sensitive to DC conductivity than low frequency properties, being given microstructural parameters (grain size and density), neither the Q factor nor the permittivity is sensitive to the processing way. A very high Q factor ($\sim$7350) has been measured on MW or CV samples exhibiting the highest grain size ($\sim$1.5 μm) and density ($>$96%). Microwave processing is then a very suitable way to get at lower temperature dense TiO_2 dielectric materials, with improved dielectric properties, especially at low frequencies. It is also believed that by using two steps sintering cycle (at low temperature and for longer time), the direct microwave sintering would lead to ultrafine microstructure with very low frequency dielectric losses.

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