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# Tailoring the microstructure of mechanoactivated $Al_2O_3$ and $SiO_2$ mixtures with $TiO_2$ addition

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ABSTRACT

This paper reports on the effect of $TiO_2$ on mullitization, anisotropic grain growth and densification of $Al_2O_3$ and $SiO_2$ mixtures, which are activated by a high-energy ball milling process using tungsten as milling media. High-energy activation significantly promotes mullitization in the mixture of $Al_2O_3$ and $SiO_2$ powders, and is a pre-requisite for the occurrence of anisotropic grain growth of mullite. There is a subtle interplay and competition between anisotropic grain growth and densification, depending on the amount of $TiO_2$. Small amount of $TiO_2$ has a positive effect on the phase formation and anisotropic grain growth of mullite, while high concentration of $TiO_2$ suppresses anisotropic grain growth, and thus is beneficial to densification. As a result, the microstructure and densification of the mullite ceramics can be manipulated by the doping levels of $TiO_2$.



## 1. Introduction

Mullite ($3Al_2O_3{\cdot}2SiO_2$) has a special crystal structure, where strong-bounded chains lie along the crystallographic *c*-axis, which allows its grains grow anisotropically in an unconstraint environment. This is why whiskers or needlelike mullite can be easily formed via vapor–solid synthesis [1–3] or molten salt flux [4]. Mullite with elongated grains has been widely used to fabricate reinforced ceramic materials. However, the techniques for synthesis of these elongated mullite grains have several disadvantages. For example, the vapor–solid synthesis requires facilities with high airtight. Reaction involving gaseous phases makes the processing complicated. The products produced by this way are therefore not cost-effective. An alternative way to produce interlocking microstructured mullite ceramics is via *in situ* processing technique. For a conventional solid-state reaction process, *in situ* anisotropic grain growth usually takes place at very high temperatures (>1600 °C) [5–8]. This is because mullite ceramics prepared in this way experienced densification before mullite phase formation. This suppressed anisotropic growth of mullite grains. In this case, ultra high temperature is essential for the formation of a eutectic liquid to maintain the anisotropic grain growth of mullite in a fully densified sample [5–8].

Kong et al. [9] recently developed an alternative technique to produce mullite whiskers at relatively low temperatures. They demonstrated that mullite whiskers can be readily fabricated in the $Al_2O_3$ and $SiO_2$ mixtures activated by a high-energy ball milling process (as referred to mechanical alloy or mechanoactivation). It is worth mentioning that although mechanical alloy has long been applied to the fabrication of mullite ceramics [10–14], mullite whiskers were first synthesized by Kong et al. [9]. Such an achievement may be mainly attributed to the two reasons. High density milling media (e.g., tungsten carbide) are essential to the development of high-quality mullite whiskers [9], while other researchers used lighter media, such as porcelain [10], silicon nitride [11,12] and alumina [13,14]. As a result, they have not observed obvious anisotropic growth. It seems that there could exist an energy threshold created by the milling process for the formation of mullite whiskers. On the other hand, it is important to use oxides (e.g., $Al_2O_3$ and $SiO_2$) as starting materials because hydroxides or hydrous materials could mitigate the energy created by high-energy balling milling [11].

$TiO_2$ is a common impurity present in mullite. As a result, studies on $TiO_2$ doping effects have been carried out by many researchers [15–19]. It is reported that solubility limits of $TiO_2$ are in the range of 3.8–4.2 and 4.1–4.4 wt% $TiO_2$ for 3:2 and 2:1 mullites [19], respectively. Anisotropic growth takes place at >1500 °C in gel-derived millite ceramics with the addition of $TiO_2$ exceeding the solubility limit [17,18]. There is no reports regarding $TiO_2$ doping on mechanoactivated mixtures of $Al_2O_3$ and $SiO_2$ (with tungsten car-

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bide media), although a range of other additives, such as $Fe_2O_3$ [20], CoO [20], NiO [20], $B_2O_3$ [21], and $W_2O_3$ [9], have been explored. These dopants have been identified to play a single role in affecting mullitization, densification, or anisotropic growth of the milled mixtures of $Al_2O_3$ and $SiO_2$. Interestingly, as will be presented in this study, $TiO_2$ can play multiple roles in affecting mullitization, densification and anisotropic growth. Also, some observations regarding $TiO_2$ doping found in the present work are significantly different from those observed when using gel-derived mullite precursors [16,17,19].

## 2. Experimental

$Al_2O_3$ (AKP-30, Japan), $SiO_2$ (Alfa Aesar-Johson Matthey Copmoany, USA) and $TiO_2$ (99.5+% purity, Aldrich Chemical Company Inc., USA) were used as starting materials. Mixtures of $Al_2O_3$ and $SiO_2$ with a composition of mullite ($3Al_2O_3{\cdot}2SiO_2$), doped with $TiO_2$ of 1–10 wt%, were first thoroughly mixed for 5 h with a conventional milling. After that, each composition was divided into two parts, one of which was subjected to a high-energy ball milling. About 20 g powders were milled for 5 h, using a Retsch PM400 type planetary ball milling system. A 250 ml tungsten carbide vial, together with 100 tungsten carbide balls of 10 mm, was used as milling media. The weight ratio of ball-to-powder was about 40:1 and milling speed was 200 rpm. The milled and unmilled mixture powders were then pressed uniaxially into pellets of 10 mm diameter, at a pressure of 50 MPa. Green pellets were then sintered in a Carbolite RHF 1600 type furnace in air for 5 h at temperatures ranging from 1100 to 1500 °C at heating and cooling rate of 10 °C/min. Selected samples were polished using a 0.5 μm diamond paste and annealed for 0.5 h at temperatures of 50 °C lower than the corresponding sintering temperatures.

X-ray diffraction analysis on the as-milled powders and the sintered samples was performed at room temperature. All samples were scanned over 20–45° at a scanning speed of 1 °/min, using a Rigaku ultima+ type diffractometer (XRD) with Cu Kα radiation at 50 kV and 30 mA. Densification behaviors of the samples were monitored in air using a Setaram Setsys 16/18 type dilatometer at a heating rate of 10 °C/min. Microstructures of the sintered samples were examined using a JEOL JSM-6340F type field emission scanning electronic microscope (FESEM). For the samples doped with 0–10 wt% $TiO_2$ and sintered at 1500 °C for 5 h, the well polished and thermally etched ones were used for microstructural observation, while the rest of SEM micrographs are obtained from the natural surfaces of the sintered mullites. Densities of the sintered samples were estimated from their mass and physical dimension.

## 3. Results

### 3.1. Mullitization

In the mixture of $Al_2O_3$ and $SiO_2$, without high-energy ball milling and $TiO_2$ addition, the formation of mullite is very difficult. Only a trace of mullite is detectable by XRD in the sample sintered at 1400 °C, while full mullitization cannot be achieved after sintering at 1500 °C for 5 h [9]. The presence of $TiO_2$ exhibits a positive effect on the phase formation of mullite from the mixture of $Al_2O_3$ and $SiO_2$. It is demonstrated that mullite is detectable in the mixture with 5 wt% $TiO_2$ after sintering at 1300 °C, which is 100 °C lower than that of the undoped samples. At 1500 °C, almost full mullitization can be achieved (Fig. 1). However, the enhancement in mullite formation is not simply increasing with increasing concentration of $TiO_2$. Further increase in $TiO_2$ content (>5 wt%) leads to a poor mullitization behavior. Interestingly, as will be shown later, for the milled samples, 1 wt% $TiO_2$ is the best concentration for mullitization.

Fig. 2 shows the XRD patterns of the samples, derived from the milled mixture of $Al_2O_3$ and $SiO_2$ without $TiO_2$, sintered at different temperatures for 5 h. After sintering at 1100 °C, no mullite phase can be detected by XRD (Fig. 2a). The diffraction peaks can be ascribed to quartz and α-$Al_2O_3$. A trace of cristobalite is found in the XRD pattern (Fig. 2a), due to partial and total phase transformation of quartz and alumina during sintering at this temperature. Compared to the unmilled samples, 5-h-milled mixture became much more reactive. Almost entire mullitization (IDD No. 15-776) is observed in the sample sintered at 1200 °C (Fig. 2b). This temperature is nearly 200–300 °C lower than that required by the unmilled mixture, which is even comparable to gel-derived diphasic mullite precursors. The great enhancement in mullitization in the high-energy ball milled mixture can be readily attributed to the significant refinement in the particles/grains of $Al_2O_3$ and $SiO_2$ [9,20] as well as the formation of Al–O–Si bonds created during the milling process [11,12].

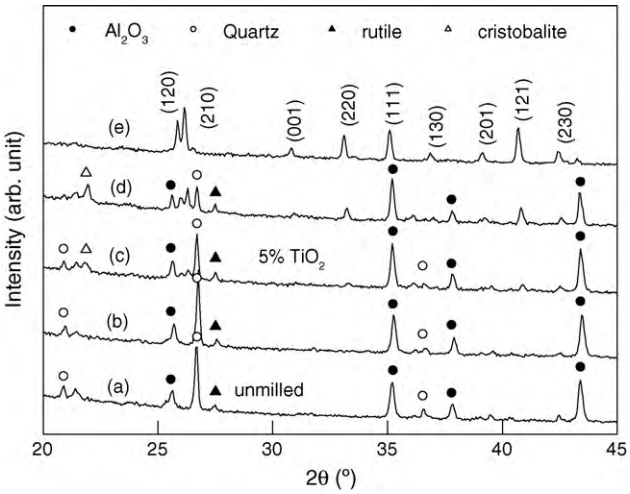


**Fig. 1.** XRD patterns of the unmilled mixtures with 5 wt% $TiO_2$ sintered at 1500 °C for 5 h at various temperatures: (a) 1100 °C, (b) 1200 °C, (c) 1300 °C, (d) 1400 °C and (e) 1500 °C.

XRD patterns of the milled samples doped with 1 wt% $TiO_2$ sintered at various temperatures are shown in Fig. 3. Compared to the undoped samples (Fig. 2), 1 wt% $TiO_2$ demonstrates a positive effect on the mullite phase formation. As evidenced by Fig. 3a, mullite is main phase in the 1100 °C-sintered sample.

A careful inspection of the 1200 °C-sintered samples with different $TiO_2$ contents (Fig. 4) indicates that the mullitization of the samples with >3 wt% $TiO_2$ is poorer than the samples with $TiO_2$ levels of ≤1 wt%. It seems that adding more $TiO_2$ prevents the combination of $Al_2O_3$ and $SiO_2$ from forming mullite as the XRD peak intensities of unreacted $Al_2O_3$ increase with increasing the level of $TiO_2$. The optimal concentration of $TiO_2$ (1 wt%) for the milled mixtures is significantly lower than that (5 wt%) for the unmilled samples, which is probably attributed to the fact that the effectiveness of $TiO_2$ is increased as a result of the high-energy ball milling which leads to the refinement of $TiO_2$ particles. In addition,

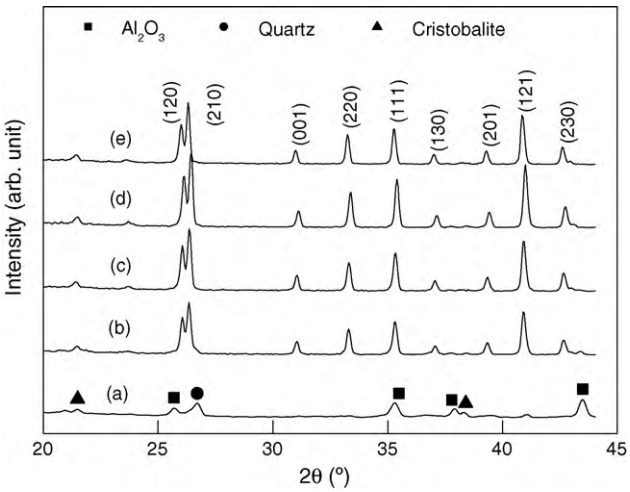


**Fig. 2.** XRD patterns of the milled mixture without $TiO_2$ sintered for 5 h at various temperatures: (a) 1100 °C, (b) 1200 °C, (c) 1300 °C, (d) 1400 °C and (e) 1500 °C.

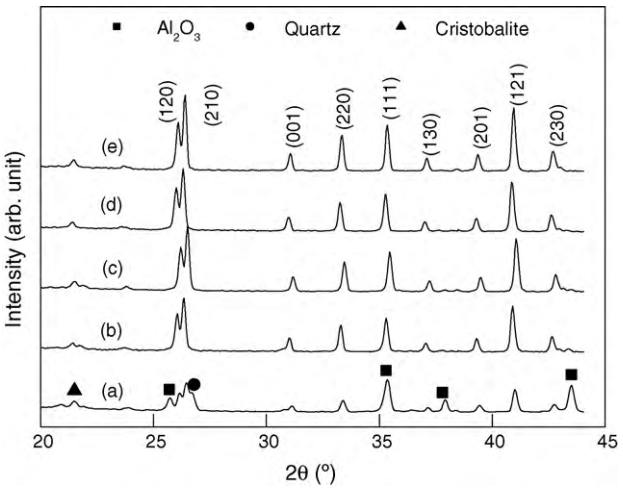


**Fig. 3.** XRD patterns of the milled mixture with 1 wt% $TiO_2$ sintered for 5 h at various temperatures: (a) 1100 °C, (b) 1200 °C, (c) 1300 °C, (d) 1400 °C and (e) 1500 °C.

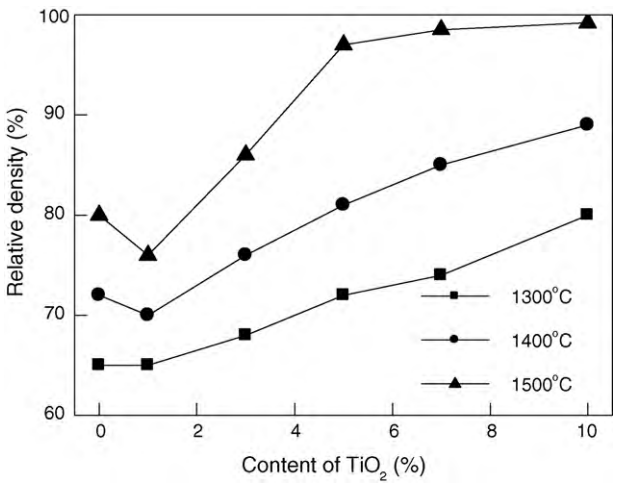


**Fig. 5.** Relative densities of the samples derived from the milled powders as a function of content of $TiO_2$.

no diffraction peaks from $TiO_2$ appear in the XRD patterns for the samples with 1 wt% $TiO_2$ (Fig. 4b), while $TiO_2$ (rutile) is observable in the 3 wt% samples (Fig. 4c). This suggests that the solubility limit of $TiO_2$ in the milled mullite ceramics is below 3 wt% at 1200 °C.

### 3.2. Measured densities

Measured densities of the milled samples sintered at 1300–1500 °C as a function of $TiO_2$ concentration are plotted in Fig. 5. The density of the samples sintered at 1300 °C increases almost linearly with increasing the concentration of $TiO_2$. However, the maximum relative density is still less than 80%. Similar trend is found for the 1400 °C-sintered samples, except for the pure one. After sintering at 1500 °C for 5 h, the density increases linearly as the concentration of $TiO_2$ is increased from 1 to 5 wt%. At this sintering temperature, almost fully dense mullite ceramics are achieved for the sample doped with 5 wt% $TiO_2$. In contrast, the pure sample has reached only 80% of the theoretical density of mullite. This observation indicates that the presence of $TiO_2$ has a positive effect on the densification of mullite derived from the mixture of $Al_2O_3$ and $SiO_2$ treated via the high-energy ball milling. It is noted that the densification behaviors of the unmilled samples, no matter whether doped with $TiO_2$ or not, are much poorer than the milled samples. It implies that the high-energy milling treatment is essential for the full densification of the mullite ceramics.

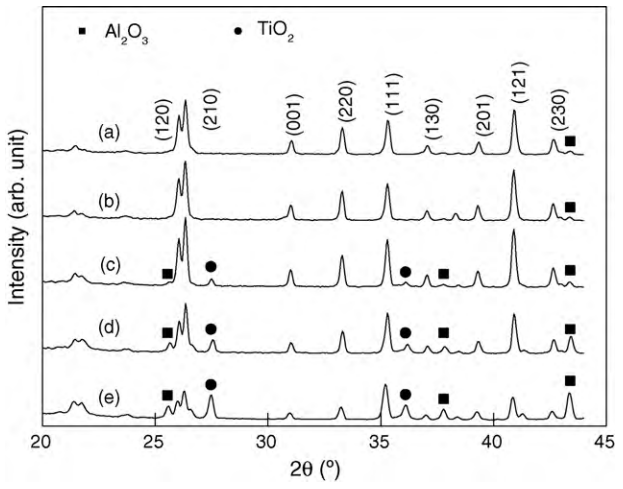


**Fig. 4.** XRD patterns of the milled mixture sintered at 1200 °C for 5 h with different concentrations of $TiO_2$: (a) 0 wt%, (b) 1 wt%, (c) 3 wt%, (d) 5 wt% and (e) 7 wt%.

### 3.3. Microstructural development

From Fig. 6 (1200 °C), the effect of $TiO_2$ on the anisotropic grain growth of mullite from the milled mixtures can be discernable. The undoped sample consists of rod-like grains, with an average length of about 1 μm and thickness of about 0.5 μm, demonstrating that an anisotropic grain growth occurred at this temperature. In contrast, the anisotropic grain growth is more pronounced in the sample doped with 1 wt% $TiO_2$. At the same time, dimension of the mullite grains is larger than that of the undoped sample. As the concentration of $TiO_2$ is increased to 3 wt%, the dimension of mullite grains further increased. Meanwhile, the uniformity in grain size distribution becomes poor as compared to the sample doped with 1 wt% $TiO_2$. For example, there are occasionally some grains with thickness of larger than 1 μm, as shown in Fig. 6c. Such a phenomenon becomes worse for the sample doped with 5 wt% $TiO_2$. The 7 wt% $TiO_2$ sample has a similar feature to the 5 wt% one, but the former exhibits more irregular grains as compared to the latter (Fig. 6d and e). Anisotropic grains are almost totally absent in the sample doped with 10 wt% $TiO_2$. Similar trend is observed for the samples sintered at higher temperatures (Fig. 7).

Fig. 8 shows the microstructures of representative samples treated by polishing and post-annealing. It is evident that microstructural evolution in the sample interior is well consistent with that on the sample surfaces (Figs. 6 and 7). The interesting observation from Fig. 8 is that the samples with low concentration of $TiO_2$ demonstrate better anisotropic grain growth while those with high concentration of $TiO_2$ possess better densification behavior. It demonstrates that anisotropic grain growth and densification are competitive each other in the milled $Al_2O_3$ and $SiO_2$ mixture during the phase formation and sintering. This is in a good agreement with the measured densities as shown in Fig. 5, where full densification requires $TiO_2$ concentration of ≥5 wt% and sintered at a sintering temperature of 1500 °C. It should be highlighted that this temperature is much lower than those reported in the open literatures [22]. On the other hand, the milled samples with low concentration of $TiO_2$ and sintered at relatively low temperatures (≤1400 °C) are very porous. More importantly, these materials possess *in situ* interlocking structure (Figs. 5 and 8), which might have improved mechanical strength. This is particularly critical for fabricating high-quality porous mullite ceramics for a range

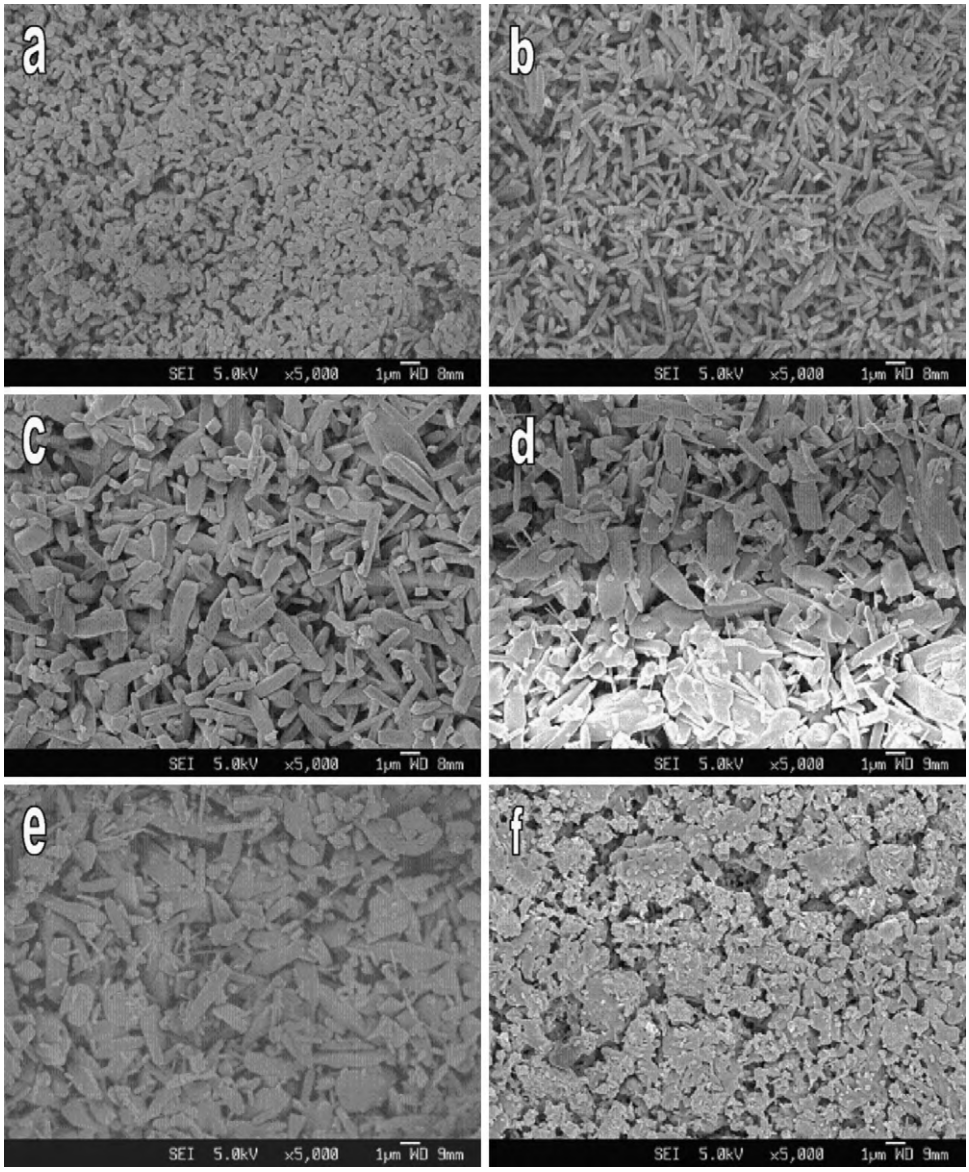


**Fig. 6.** SEM images of the samples derived from the milled powders sintered at 1200 °C for 5 h: (a) 0 wt% $TiO_2$; (b) 1 wt% $TiO_2$; (c) 3 wt% $TiO_2$; (d) 5 wt% $TiO_2$; (e) 7 wt% $TiO_2$ and (f) 10 wt% $TiO_2$.

of industrious applications since mullite materials usually have relatively low fracture toughness.

### 3.4. Dynamic densification behaviors

Dynamic densification behaviors of the milled mixtures with different concentrations of $TiO_2$ are shown in Fig. 9. A common feature of the densification curves is that there exists a sharp expansion starting at ~600 °C and abrupt shrinking starting at ~1200 °C. The degree of the expansion decreases while that of the shrinkage increases with increasing concentration of $TiO_2$. Careful inspection reveals that the shrinkage is not observed in the undoped and 1 wt% $TiO_2$-doped samples. Instead, there is a visible expansion at 1200 °C. $TiO_2$ has been found to promote the densification of mullite ceramics [16] (also as evidenced in the present study), which is most likely related to a liquid phase sintering. In general, high-energy ball milling process could result in a significant refinement of raw materials as well as dopants (i.e., $TiO_2$ in our case), leading to the formation of a transient liquid phase at relatively low temperatures. This may be the reason that a rapid shrinkage occurs at temperatures of as low as ~1200 °C in the milled $TiO_2$-doped samples. However, in the samples with low contents of $TiO_2$, there should be no sufficient amount of liquid phase to drive sintering since Ti atoms would incorporate into mullite lattice. Actually, in the sample with 1 wt% $TiO_2$, there is no significant shrinkage observed, which behaves like the undoped sample. For clarity, the sintering curve of this sample is omitted from Fig. 9. In addition, all the samples used exhibit a slight shrinkage at ~1500 °C. The expansion at 600 °C is not explainable at the moment. It may be due to either the burning out of binder or phase transformation of $Al_2O_3$, $SiO_2$ and $TiO_2$. The enhancement in densification behavior of the samples doped with >3 wt% $TiO_2$ is in agreement with the results of SEM (Fig. 8) and the measured densities (Fig. 5) as presented above.

## 4. Discussion

Mullitization in a mixture of $Al_2O_3$ and $SiO_2$ requires a very high temperature due to the poor reactivity of the starting materials (with rough grain/particle morphologies). In general, the mullitization temperature of mixed oxide precursors could be significantly

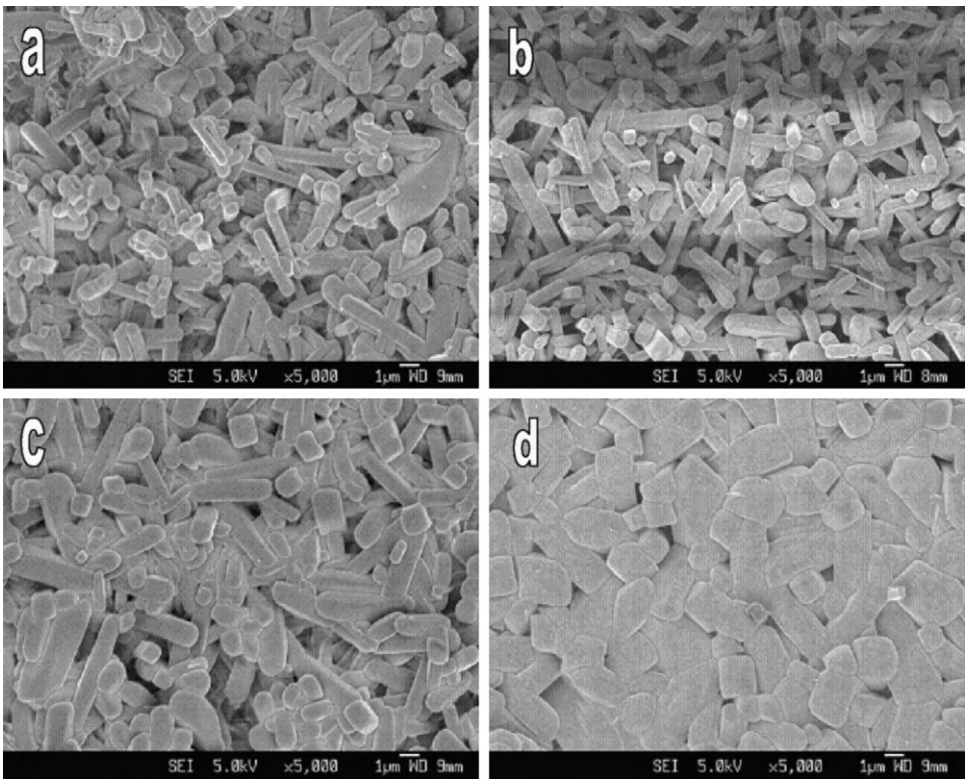


**Fig. 7.** SEM images of the samples derived from the milled powders sintered at 1500 °C for 5 h: (a) 0 wt% $TiO_2$; (b) 1 wt% $TiO_2$; (c) 3 wt% $TiO_2$ and (d) 5 wt% $TiO_2$.

reduced (~200–300 °C) by a dry ball milling process. This has been verified by many researchers [9,11]. A prolonged milling duration of 40 h could further reduce the mullitization temperature below 900 °C [13], which is comparable to monophasic mullite gels [23], and even much better than diphasic gels [24]. However, few researchers have reported the anisotropic grain growth of mullite in milled mixtures at a temperature of as low as 1100 °C. Our recent studies [9,21] demonstrated that the degree of anisotropic growth is proportional to the energy provided by the dry milling process. Using high density milling media is essential for the production of mullite whickers. Among all the media used, tungsten carbide has been proven to be the most efficient tool for this purpose [21]. This may be the main reason that both Ebadzadeh [14] and Behmanesh et al. [13] could not repeat the work of Kong et al. [9], due to the usage of low density milling media (alumina). Kong et al. [25] recently observed the anisotropic grain growth in a commercial mullite powder treated by high-energy ball milling with tungsten carbide media. However, unfortunately, the mechanisms underlying the anisotropic grain growth induced by the high-energy dry milling process are unavailable until now.

Effect of $TiO_2$ on mullite phase formation has been reported in the literature [15–19]. The results indicate that the amount of $TiO_2$ incorporated into $SiO_2$ matrix by the substitution of tetrahedrally coordinated $Ti^{4+}$ for $Si^{4+}$ can be as high as 11.5 wt% $TiO_2$ in a sol–gel processed $TiO_2$-$SiO_2$ system [16]. Incorporation of $TiO_2$ into $SiO_2$ is believed to reduce the viscosity of the liquid siliceous phase at high temperatures, which enhances the diffusion kinetics accordingly. It also possibly improves the incorporation of alumina phase into the siliceous matrix, thus accelerating the alumina dissolution. At the same time, the presence of a small amount of $TiO_2$ particles would provide heterogeneous nucleation sites of mullite to low the nucleation energy of mullite. All these would contribute to the reduction in the mullite phase formation temperature, which might be also applied to $TiO_2$ containing milled samples. But in our case, 1 wt% $TiO_2$ is the optimal concentration to promote mullitization (Fig. 4). This seems different from the observation in $TiO_2$-doped gel-derived mullite, where a higher doping level (up to 5 wt%) showed no difference in phase development although $TiO_2$ could reduce the mullite transformation temperature [16]. The difference in the effect of $TiO_2$ between our observations and those reported by others can also be demonstrated by the following discussion.

The anisotropic grain growth should be detrimental to densification. This is the reason that the milled samples without $TiO_2$ addition cannot reach higher density (only ~ 80% relative density at 1500 °C). The observed density minimum at 1 wt% $TiO_2$ (Fig. 5) further supports this argument because 1 wt% $TiO_2$ is the optimal concentration for mullitization (Fig. 4) as well as anisotropic grain growth (Fig. 6b). Interestingly, higher $TiO_2$ level (>3 wt%) retards mullitization and suppress anisotropic growth. Consequently, almost full densification can be achieved at 1500 °C for the milled samples with $TiO_2$ contents of ≥5 wt%. As stated previously, the addition of $TiO_2$ could reduce the glass viscosity and increase mobility of the diffusing species, thus promoting densification [16,19]. Baudin and Moya [26] reported that the addition of $TiO_2$ below its solubility limit enhances the initial sintering and grain size, while excessive amounts of $TiO_2$ inhibit sintering and drastically increase the total porosity and mean grain size. However, Hong and Messing [17] found that titania enhanced initial and intermediate stage densification in the diphasic mullite gels and led to anisotropic grain growth at the final stage sintering when the amount of $TiO_2$ exceeded its solubility limit. These different (sometimes, contradictory) observations regarding the effects of $TiO_2$ reported by different groups reflect the complexity in the role of $TiO_2$ in mullite ceramics. It should strongly rely on the nature of mullite precursors used and the ceramic processing adopted. Therefore, further work is needed to clarify the role of $TiO_2$ in determining the mullitization, densification and grain growth of mullite, especially from oxide mixtures activated by high-energy ball milling.

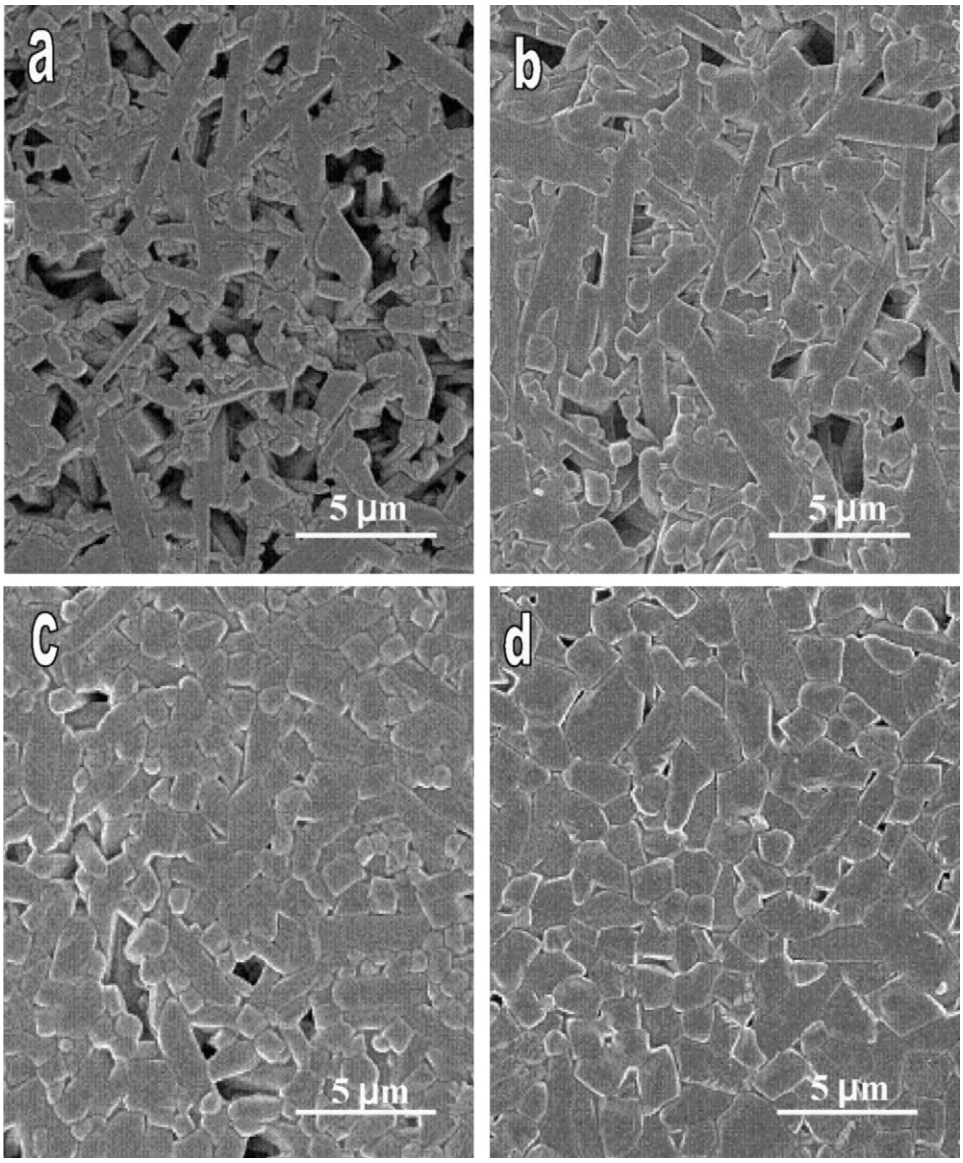


**Fig. 8.** Representative SEM images of the polished samples derived from the milled powders sintered at 1500 °C for 5 h: (a) 1 wt% $TiO_2$, (b) 3 wt% $TiO_2$. (c) 5 wt% $TiO_2$, and (d) 10 wt% $TiO_2$.

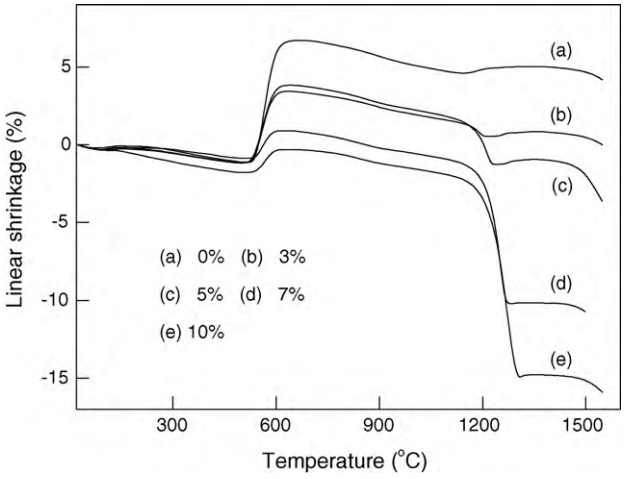


**Fig. 9.** Linear shrinkage curves of the milled samples with different $TiO_2$ contents, sintered at 10 °C/min to 1550 °C: (a) 0 wt% $TiO_2$, (b) 3 wt% $TiO_2$. (c) 5 wt% $TiO_2$, (d) 7 wt% $TiO_2$ and (e) 10 wt% $TiO_2$.

## 5. Conclusions

Phase formation of mullite from the mixture of $Al_2O_3$ and $SiO_2$ was greatly promoted by the activation of high-energy ball milling process. The mullite phase formation temperature was decreased by about 200 °C. The reaction of $Al_2O_3$ and $SiO_2$ was further enhanced by the appropriate addition of $TiO_2$ (~1 wt%). At the same time, the anisotropic grain growth of mullite was observed in the mixture treated by the high-energy ball milling. The anisotropic grain growth was improved due to the presence of $TiO_2$ at low concentrations, but hindered at higher concentrations. Instead, the samples with high concentration of $TiO_2$ exhibited a good densification behavior. Almost full densification can be achieved at 1500 °C for the milled samples with $TiO_2$ contents of ≥5 wt%. On the other hand, the presence of *in situ* interlocking structure in the milled samples with low $TiO_2$ contents (<3 wt%), sintered at relatively low temperatures (≤1400 °C) are particularly important for fabricating high-quality porous mullite ceramics with improved toughness.

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