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Toward vacuum sintering of YAG transparent ceramic using divalent dopant as sintering aids: Investigation of microstructural evolution and optical property

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ABSTRACT

Transparent YAG ceramics were fabricated by solid state reaction sintering using divalent dopants (CaO and MgO) as sintering additives without TEOS doping, and the effects of divalent dopants on their microstructure evolution and optical properties were investigated. It was found that CaO was more effective with respect to inhibiting grain growth than MgO, but not as effective as MgO in promoting densification. Fully dense, transparent YAG ceramics with excellent optical qualities could be achieved by optimizing the doping concentrations of CaO and MgO; the transmittance at 1064 nm was as high as 84.5% for 3 mm thick sample at the molar ratio of Ca: Mg=1:4, after sintered at 1840 °C for 8 h in vacuum.

1. Introduction

Since Ikesue et al. firstly demonstrated the possibility of elaborating transparent Nd: YAG ceramic with excellent optical quality for solid state lasers in 1995 [1,2], transparent ceramic material has attracted extensive attention by people and developed rapidly, which has been widely adopted in many significant fields, especially as laser gain medium for solid state lasers [3–5]. Compared with their single crystal counterparts grown by traditional Czochralski method, transparent ceramic materials have many advantages such as shorter production cycle, flexibility in scales and shapes, lower production cost and better homogeneity [6,7]. Until now, various kinds of transparent ceramic materials have been investigated by researchers, typically including garnet (YAG, LuAG, etc) [8–10], sesquioxide (Y_2O_3, Lu_2O_3, etc) [11–13], spinel ($MgAl_2O_4$, etc) [14,15] and non-oxide ceramics (ZnS, AlN etc) [16,17]. Among them, transparent yttrium aluminum garnet (YAG) is the most commonly adopted transparent ceramic material, thanks to its relative ease in fabrication and considerable physical and chemical properties. Enormous application prospects are foreseen.

Owing to relatively low sintering activity of YAG powders prepared by solid state reaction sintering method, tetraethyl orthosilicate (TEOS) is accordingly adopted as sintering aid to promote sintering [18–20]. TEOS will be decomposed into SiO_2 under heat treatment, and then SiO_2 react with YAG to form liquid phase at around 1400 °C according to YAG- SiO_2 phase diagram [21]. In this case, the densification rate is dramatically enhanced, thanks to liquid phase diffusion mechanism [22]. However, the employment of TEOS as sintering aid has its intrinsic disadvantage. First, TOES doping easily leads to dramatic grain growth [23,24], which is not beneficial to the mechanical property of YAG ceramic. Also, TEOS will tremendously deteriorate the conversion efficiency of Cr^{4+} ion or Yb^{3+} ion in YAG ceramics, resulting from charge compensation [25,26]. Our previous report showed that the conversion efficiency of Cr^{4+} ion in Cr: YAG ceramic that doped with only 0.15 wt% TEOS was only one-eighth compared with that of the sample without TEOS doping [25].

With respect to YAG transparent ceramic without silicon doping (or reduced silicon doping), Lee et al. reported a successful elaboration of YAG transparent ceramic using hot isostatic press sintering (HIP), and

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the in-line transmittance of as prepared ceramic was ~82% at 1064 nm, when the addition amount of silica was limited as 0.02 wt.% [23]. Qin et al. demonstrated a vacuum sintered YAG ceramic without silicon addition, and the in-line transmittance at 1020 nm was 82% after sample sintered at 1780 °C for 6 h [27]. Lu et al. reported a 10 at.% Yb: YAG ceramic with thickness of 2 mm fabricated by co-precipitation method, and the in-line transmittance at 1100 nm was 83.4% after HIP sintered at 1700 °C for 2 h under 200 MPa [28]. The optical qualities of the above reported YAG ceramics were relatively low, that could not meet the requirement of laser application. One reason is that compared with silicon doped YAG ceramic, non-silicon series YAG ceramic is harder to densify. Even though HIP treatment can effectively promote densification by providing external pressure, but it requires high-level equipment and energy consumption.

Until now, however, there is few report conducted a systematic study concerning YAG ceramic with excellent optical qualities without silicon addition. In this article, high quality transparent YAG ceramics were successfully fabricated by solid state reaction sintering under vacuum using divalent dopants as sintering aids, and microstructure evolution as well as optical properties of as-prepared YAG ceramics were investigated systematically.

2. Experimental process

In the present study, high- purity α- Al_2O_3 (99.99% purity, Sumitomo Chemicals, Tokyo, Japan) and Y_2O_3 (99.99%, Alfa Aesar, Ward Hill, MA) powders were used as starting materials. They were weighted precisely in stoichiometric proportions to form $Y_3Al_5O_{12}$ in the resultant YAG ceramics. CaO (99.999%, Alfa Aesar, Ward Hill, MA) and MgO (99.999%, Alfa Aesar, Ward Hill, MA) were selected as sintering additives. The molar ratio between Ca and Mg was varied, and the total addition amount of divalent dopants of each sample was kept as 0.2 mol% according to Al^{3+} content.

The powder mixtures were ball milled in 99.9% ethyl alcohol for 12 h with high purity Al_2O_3 balls. The milled slurry was dried at 60 °C in an oven for 24 h and sieved through a 100 mesh screen, then uniaxially pressed at 20 MPa in a stainless steel mold to obtain pellets with a diameter of 16 mm. The obtained pellets were then cold isostatic pressed at 210 MPa for 5 min, followed by calcined at 800 °C for 4 h in air to remove the volatile organic residues. The compacted greed bodies with a relative density of ~56% were then sintered in a tungsten mesh heated vacuum furnace under 10^{-6} Torr between 1540 °C~1780 °C for 4 h in order to check their microstructure involution during vacuum sintering, and also sintered at 1820 °C and 1840 °C for 8 h under the same vacuum condition to investigate their optical properties. Finally, all samples were mirror polished on both surfaces using diamond slurries, and the thickness of all samples after mirror polishing was 3 mm. YAG ceramic without dopant as sintering aid prepared by the same process was also adopted as a reference.

Densities of the obtained samples were measured by Archimedes method. The phase compositions of the sintered YAG ceramics were identified by an X- ray diffraction machine (XRD; D2, Bruker, Karlsruhe, Germany) equipped with a copper target X- ray tube. Microstructures of the sintered samples were measured using a scanning electron microscope (SEM, JSM-6510, JEOL, Japan) as well as the optical transmission microscopy (Carl Zssis, Axio Scope. A1, Germany). For the measurements of the in- line transmittance of mirror polished YAG samples, a UV–VIS–NIR spectrophotometer (Lambda 950, Perkin Elmer, Waltham, MA, America) was applied. The scanning range varied from 1500 nm to 200 nm, and the measurements were carried out with a standard, dual light beam arrangement with adjustable slit width. The shrinkage behaviors of YAG ceramics as a function of temperature were measured by a thermal mechanical analyzer (DIL 402 E, NETZSCH, Geratebau, Germany). The heating rate was kept as 10 °C/min, and the measurement range varied from 200 °C to 1800 °C.

3. Results and discussion

Fig. 1 indicates the relative densities of as prepared YAG ceramics vacuum sintered at different temperatures for 4 h. The densities of all samples were reached to the theoretical value of YAG when the sintering temperature was above 1740 °C. The relative densities of ceramics increased along with the increasing amount of MgO at each sintering temperature when the sintering temperatures were below 1700 °C. For undoped YAG sample, the relative density reached to 98% only at a sintering temperature of 1540 °C, which was much higher than that of samples doped with divalent dopants as sintering aids.

The average grain sizes of doped and undoped samples versus sintering temperatures are shown in Fig. 2. The average grain sizes were confirmed by calculating the average grain diameters of at least 200 grains obtained from SEM images. For samples doped with divalent dopants, the average grain sizes increased in proportional to the doping amount of MgO at each sintering temperatures. The grain growth rate slightly changed for Ca doped samples, which only increased from 0.5 μm at 1540 °C to 1.7 μm at 1780 °C, but changed significantly when MgO was chosen as the only sintering aids (0.8 ~4.5 μm). For undoped YAG sample, however, the average grain size was bigger than 10 μm when the sintering temperature was as low as 1660 °C, and abnormal grain growth occurred if further increase the sintering temperature. So it is clear that both CaO and MgO could

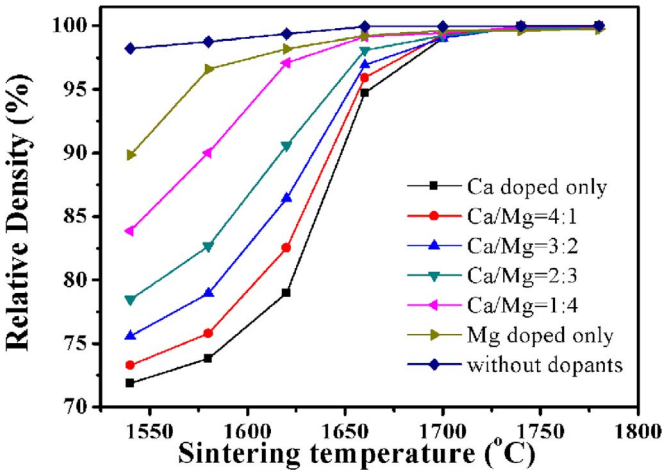

Fig. 1. Relative density versus temperature for doped and undoped YAG ceramics.

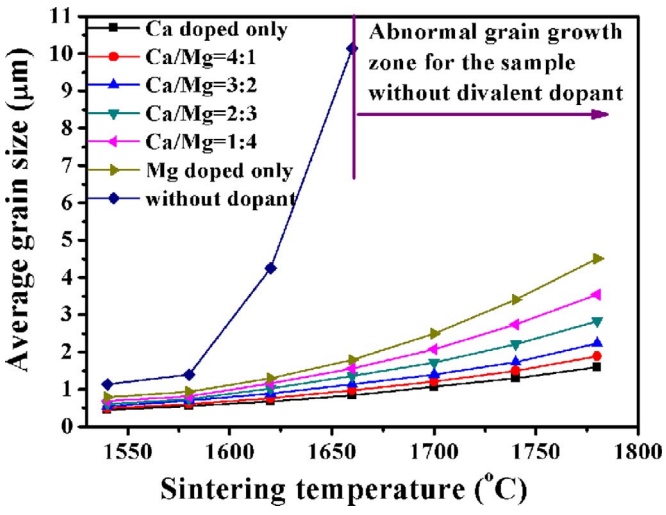

Fig. 2. Grain sizes as a function of sintering temperature between 1540 °C and 1780 °C for YAG ceramics.

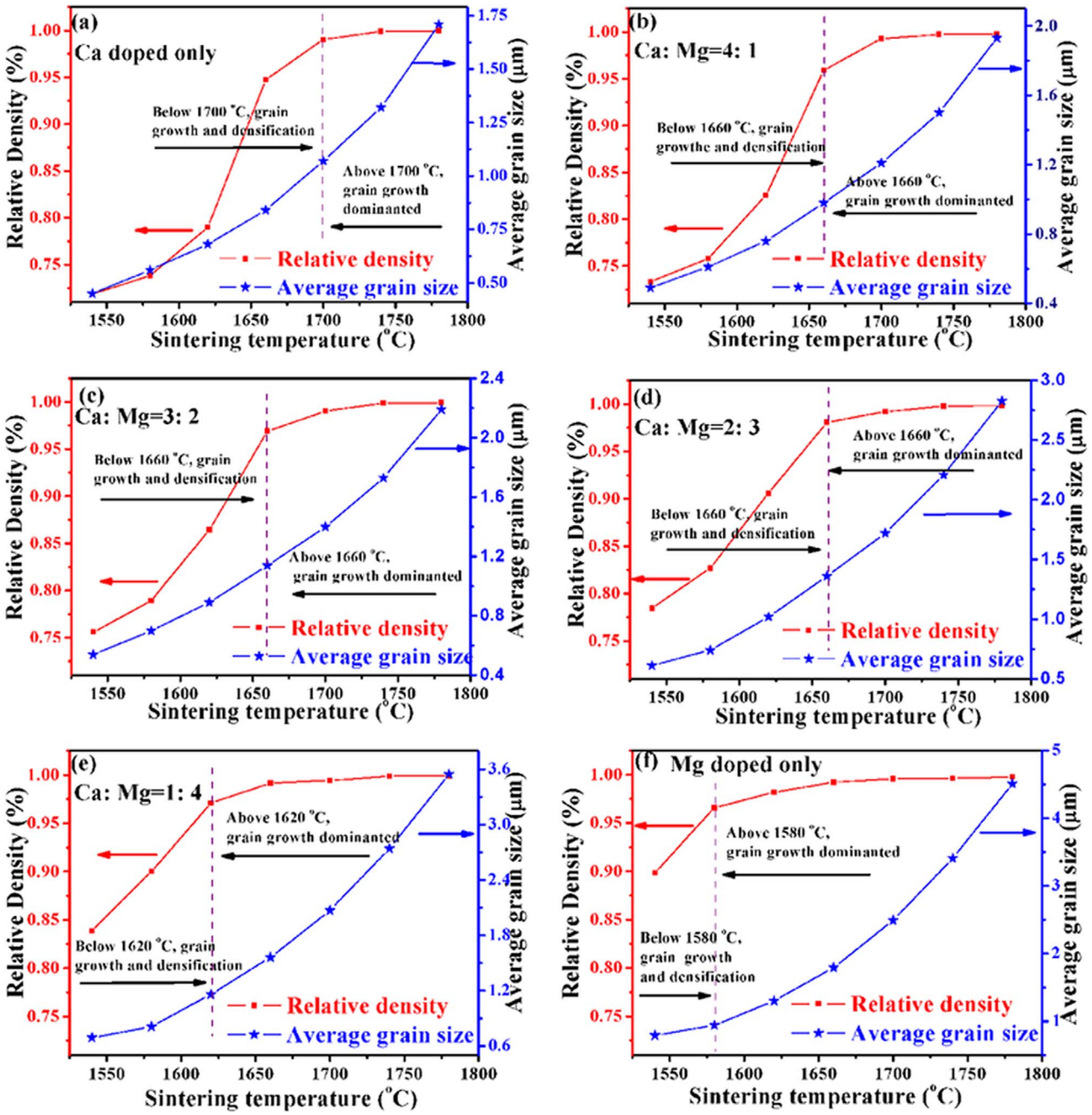

Fig. 3. Sintering trajectory for YAG ceramics doped with different amounts of divalent dopants as sintering aids.

moderate grain growth rate of YAG ceramic during sintering, and CaO was more effective than MgO with respect to the inhibition of grain size, which resulted in the tendency of relative density in Fig. 1. The relationship between grain sizes and relative densities of each sample doped with divalent dopants is summarized in Fig. 3. From Fig. 3 it was obvious that the grain growth/ densification dividing line (dotted line) shifted towards lower temperature zone when Ca/ Mg ratio decreased.

With respect to the inhibition of grain growth, a portion of CaO and MgO can segregate at grain boundaries as solutes, and led to a drag on grain boundaries during sintering. As a result, grain boundary mobility decreased thanks to solute drag effect, and discontinuous grain growth can be inhibited accordingly.

The fracture surfaces of samples doped with different amounts of divalent dopants sintered at 1620 °C for 4 h in vacuum are shown in Fig. 4. It could be clearly seen that the pore volume of samples decreased with the increase amount of MgO dopant. For samples doped with a relatively large amount of CaO [Fig. 4(a) and (b)], the morphology of pores was characterized by open pores, which was the typical characteristic of intermediate stage of sintering. The average pore sizes were getting smaller when further increase the Mg content, and isolated pores began to appear [Fig. 4(c) and (d)]. When the molar ratio of Ca/Mg reached to 1: 4 [Fig. 4(e)], open pores could not be detected, but the quantity of isolated pores were still large, indicating that the sample reached to its final stage of sintering. When MgO was chosen as the only sintering aid [Fig. 4(f)], there was only very few small isolated pores observed, and the fracture mode of the sample was characterized mainly by transgranular and partly by intergranular.

According to the Al_2O_3-Y_2O_3 system, three successive transformations are processed to create YAG phase during solid state reaction [29]:

$$2Y_2O_3 + Al_2O_3 \rightarrow Y_4Al_2O_9(\text{YAM, monoclinic structure}) \quad (1)$$

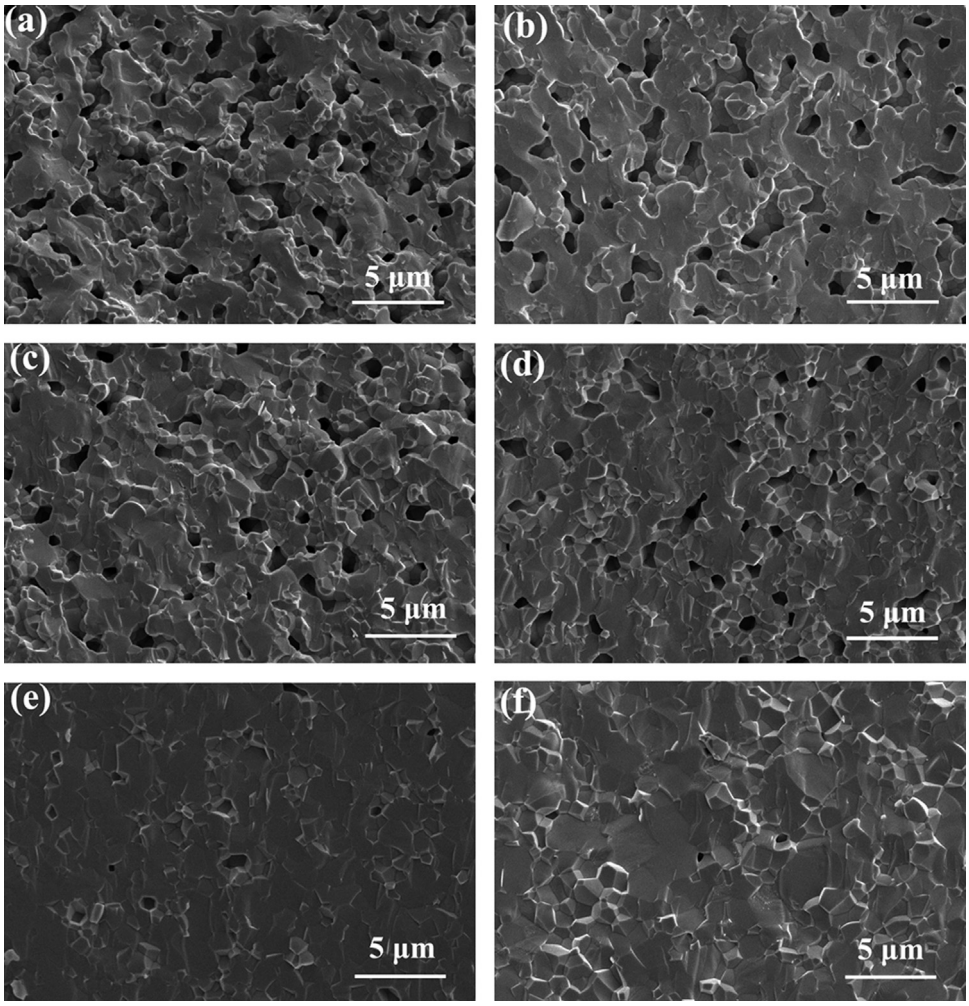

Fig. 4. SEM micrographs of the fracture surface of YAG ceramics doped with (a) Ca doped only; (b) Ca/Mg=4: 1; (c) Ca/Mg=3: 2; (d) Ca/Mg=2:3; (e) Ca/Mg=1:4; (f) Mg doped only sintered at 1620 °C for 4 h.

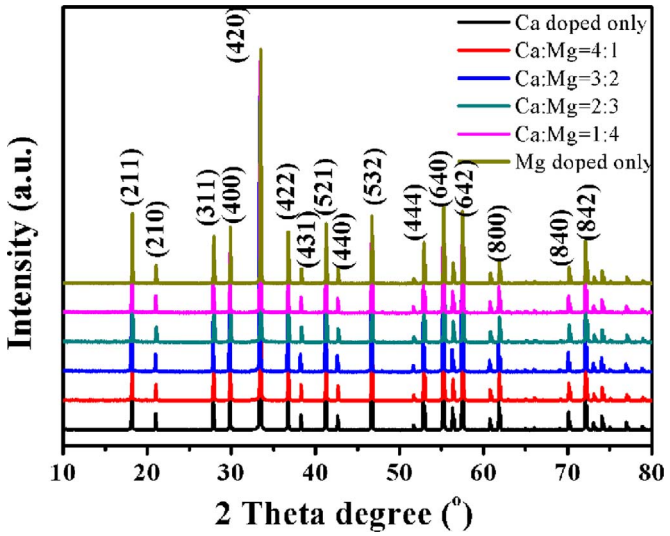

Fig. 5. XRD patterns of YAG ceramics sintered at 1840 °C for 8 h in vacuum.

$$YAM + Al_2O_3 \rightarrow 4YAlO_3(YAP, \text{perovskite structure}) \quad (2)$$

$$3YAP + Al_2O_3 \rightarrow Y_3Al_5O_{12}(YAG, \text{cubic structure}) \quad (3)$$

Fig. 5 shows the XRD patterns of the prepared YAG samples sintered at 1840 °C for 8 h in vacuum. According to equation (1) and (2), two intermediate phases, i.e., YAM and YAP phase are formed before the formation of YAG phase. If they are not totally eliminated during solid state reaction, they will act as scattering centers and deteriorate the optical qualities of obtained YAG ceramics. From Fig. 5 it was clear that all the diffraction peaks could be well indexed as the cubic structure of YAG phase, and there was no any impurity phase detected, indicating that solid state reaction process was totally completed when divalent dopants were chosen as sintering aids.

Fig. 6 shows the in-line transmission spectra of the mirror polished YAG samples sintered at 1820 °C and 1840 °C in vacuum for 8 h. It was evident that at a sintering temperature of 1840 °C [Fig. 6(a)], the transmittance of samples initially increased along with the increase of Mg contents, and reached to its maximum when the molar ratio of Ca/Mg was 1: 4. In this case, the transmittance at 1064 nm of the sample was 84.5%, which was nearly reached the theoretical limit of YAG. However, an absorption peak centered at ~320 nm could be observed from the sample, owing to the formation of F-type color centers when electrons were captured by the oxygen vacancies generaged during vacuum sintering. The transmittance decreased when MgO was chosen as the only sintering aid. At a sintering temperature of 1820 °C, the transmission curves were similar to that of samples sintered at 1840 °C, but the highest transmittance at 1064 nm was 83.9% for Ca/Mg=4: 1 sample [Fig. 6(b)], which was slightly lower than that of the sample sintered at 1840 °C. This result proves that even without TEOS doping, YAG ceramics can also be sintered to fully transparent by means of adjusting the addition amount of divalent dopants and sintering temperature appropriately.

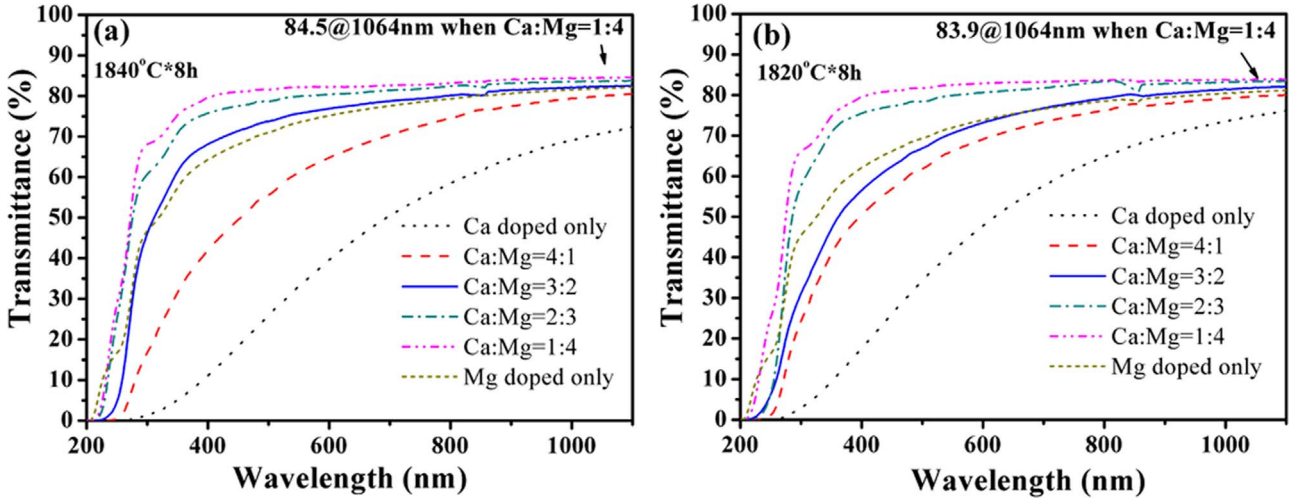

Fig. 6. Optical in-line transmittance spectrum of mirror polished YAG ceramics vacuum sintered at (a) 1840 °C; (b) 1820 °C for 8 h.

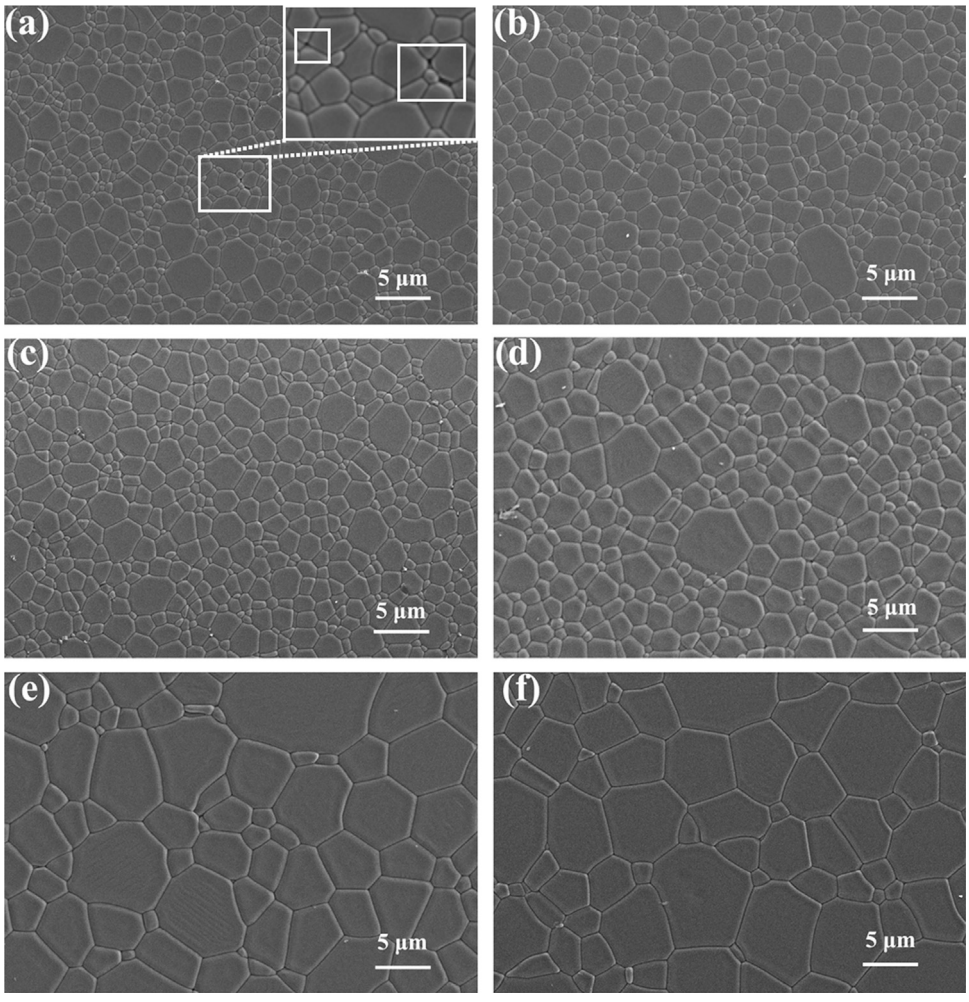

Fig. 7. SEM images of mirror polished surface of YAG ceramics doped with (a) Ca doped only; (b) Ca/Mg=4: 1; (c) Ca/Mg=3: 2; (d) Ca/Mg=2:3; (e) Ca/Mg=1:4; (f) Mg doped only sintered at 1840 °C for 4 h.

Fig. 7 displays the SEM micrographs of thermal etched surfaces of YAG samples vacuum sintered at 1840 °C for 8 h. It could be clearly seen that the average grain size increased with the increase amount of Mg content, ranging from 1.8 μm for Ca doped sample to 5.9 μm for Mg doped sample, and there was no grain boundary phase observed. For Ca doped sample, a few intergranular pores were observed (see insert of Fig. 7(a)), indicating that the sample was not fully densified, leading to the decrease in its transmittance, even though its relative density reached to nearly 100% at the current sintering temperature. The rest of samples exhibited homogeneous and pore free structures, and there was no intergranular nor intragranular pores detected.

In order to investigate the dimensional variation of ceramics using

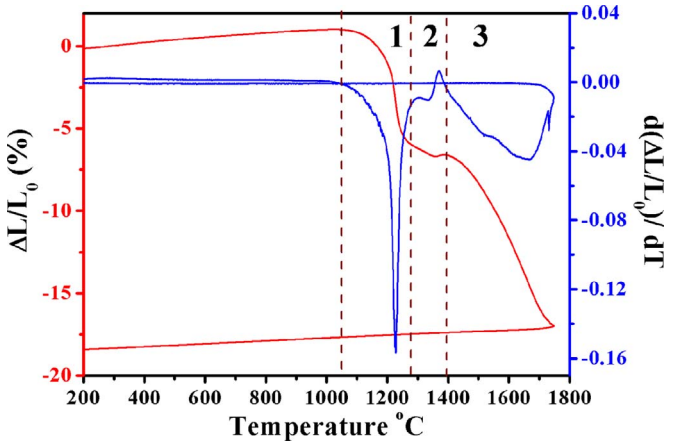

Fig. 8. TMA thermogram of divalent aids doped YAG sample when Ca/Mg was 1: 4.

divalent dopants as sintering aids during sintering, the dilatometric analysis was performed. Fig. 8 shows the TMA thermogram of Ca/Mg=1: 4 sample. The range of (1) could be correlated to the formation of YAP phase, which occurred in the temperature range of 1100~1300 °C. The fastest shrinkage occurred at ~1220 °C, indicating the mass formation of YAP. The range of (2) correspond to YAG phase formation, and a bit swelling occurred from 1360 °C to 1400 °C, thanks to volume expansion when YAP phase (5.35 g/cm^3) transformed to YAG phase (4.55 g/cm^3) with a significant decrease of molar density [30]. Densification process occurred when the sintering temperature was above 1400 °C. Yang et al. investigated the shrinkage behavior of YAG ceramics using TEOS as sintering aid, and the total shrinkage was ~17%, which was similar to our current result [24]. However, according to Yang's result, the shrinkage before 1400 °C was ~4.5%, which was 2% lower than that of the sample doped with divalent dopants shown in Fig. 9. This indicates that divalent dopants could enhance the shrinkage of YAG ceramic especially at a relatively lower temperature (lower than 1400 °C).

Fig. 9 shows the optical transmission micrograph of Ca/Mg=1:4 sample sintered at 1840 °C for 8 h, under both open and crossed polarizers. From Fig. 9(a) it was evident that no scattering centers observed from open Nicols, indicating a nearly pore free structure in the depth direction of sample. Also, perfect optical isotropy was uniquely confirmed in the dark field image using crossed Nicol [Fig. 9(b)].

Fig. 10 shows a photograph of YAG samples doped with different amounts of divalent dopants sintered at 1840 °C for 8 h. Ca doped YAG sample was translucent, and words behind it could be difficult to be recognized. The optical quality of Ca/Mg=4: 1 sample was much better than that of Ca doped sample, but fog still exhibited inside the ceramic. The rest samples were fully transparent, and words behind them could be clearly recognized by the naked eyes, despite their little differences

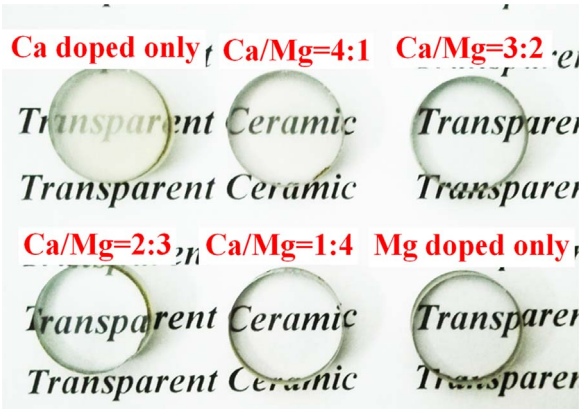

Fig. 10. A photograph of YAG samples sintered at 1840 °C for 8 h.

in terms of transmittance.

Thus, it is obvious that the combination doping of moderate amounts of CaO and MgO could significantly promote sintering and densification of YAG ceramics. With respect to densification, a portion of CaO and MgO were solid solute into YAG lattice by substituting Y^{3+} by Ca^{2+}, and Al^{3+} by Mg^{2+}, owing to their similar ion radius, which can be expressed as follows [31]:

$$2CaO \xrightarrow[Y_2O_3]{} 2Ca_Y^{'} + V_O^{\bullet\bullet} + 2O_O^{\times} \quad (4)$$

$$2MgO \xrightarrow[Al_2O_3]{} 2Mg_{Al}^{'} + V_O^{\bullet\bullet} + 2O_O^{\times} \quad (5)$$

According to Eqs. (4) and (5), oxygen vacancies (point defects) were formed thanks to charge differences between divalent ions and original cations in YAG, and thus accelerate the lattice diffusion coefficient. With respect to divalent dopants on grain sizes and densities of YAG ceramics, one possible reason is that the solid solubilities of Ca^{2+} and Mg^{2+} in YAG lattice as well as their ion radius are different, leading to differences in solid drag effect during grain growth, and thus affected the final grain sizes and densities of YAG ceramics [32]. A complex mechanism like liquid phase sintering that affected the grain sizes and densities of YAG ceramics is also possible. Koji et al. reported that CaO initially reacted with Al_2O_3 on the surface of AlN particles at 1100 °C, and then react with Y_2O_3 to form liquid phase during sintering [17]. Liquid phase mechanism with respect to the sintering of YAG ceramics under divalent dopant system still requirs further investigation.

4. Conclusions

Fully dense, transparent YAG ceramics were successfully fabricated by solid state reaction sintering using divalent dopants as sintering aids without TEOS doping. In particular, the microstructure investigations as well as optical properties of YAG ceramics were studied by varying the types and contents of divalent dopants. First, it was found that both

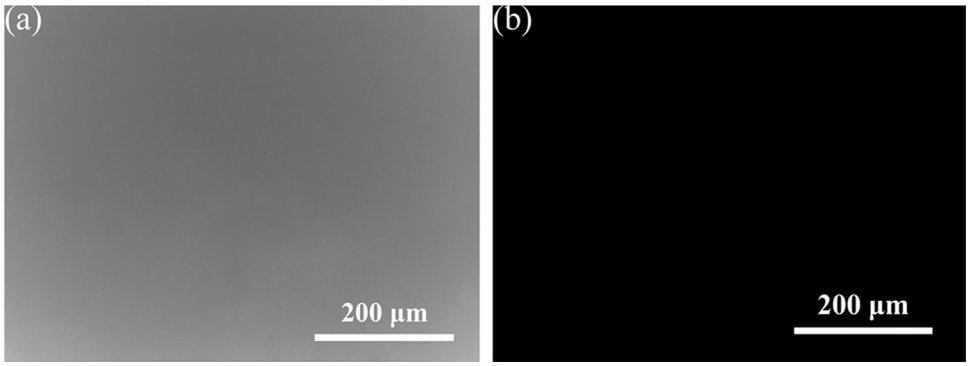

Fig. 9. Optical transmission micrographs of Ca/Mg=1: 4 sample sintered at 1840 °C for 8 h observed by (a) open Nicol and (b) crossed Nicol.

CaO and MgO could moderate grain growth rate during sintering, although MgO could promote densification more efficiently than CaO, but not as efficient as CaO in the suppression of grain growth. Second, transparent YAG ceramics with excellent optical quality could be achieved by optimizing the doping concentrations of CaO and MgO, and the transmittance at 1064 nm was as high as 84.5% at 1064 nm at the molar ratio of Ca/Mg=1: 4. Finally, divalent dopants could enhance the shrinkage of YAG ceramics when the sintering temperature was lower than 1400 °C compared with TEOS doping. Therefore, this study suggests that divalent ions doping is another effective way to elaborate transparent YAG ceramic with excellent optical quality.

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