Microstructural freezing of highly NIR transparent Y₂O₃-MgO nanocomposite via pressure-assisted two-step sintering

Ho Jin Ma, Wook Ki Jung, Seok-Min Yong, Doo Hyun Choi, Do Kyung Kim

Abstract

Y₂O₃-MgO nanocomposite has attracted attention for use in infrared windows and domes. However, the large difference in refractive index between the two phases of this materials phases induces inevitable grain boundary scattering. To overcome this drawback, it is indispensable to greatly reduce the grain size and eliminate residual pores. Two-step sintering has been extensively used to produce fine-grained ceramics, but long incubation time and an additional process are required. As a robust approach, pressure-assisted two-step consolidation of Y₂O₃-MgO nanocomposite ceramics is investigated to achieve full density while retaining domain size. A sintering path for pressure-assisted two-step sintering and related kinetic window are obtained for the first time. By effectively suppressing grain growth and eliminating residual pores via two-step hot-pressing, outstanding mid- and near-infrared transmittance were achieved. The results indicate that pressure-assisted two-step sintering is a promising alternative strategy that can decrease the domain size and achieve full density of ceramics.

Keywords: Near-infrared, Transparent ceramics, Y₂O₃-MgO nanocomposite, Two-step hot-pressing, Microstructure

1. Introduction

Infrared transparent ceramics have been developed over several decades for use in many fields of application, including infrared windows and domes [1-5]. Especially, infrared transparent materials, which have outstanding mechanical and thermal properties have been highlighted for use in harsh environments. Recently, due to its superior optical and thermomechanical characteristics, Y₂O₃-MgO nanocomposite has been introduced as a suitable infrared optical material [6]. While Y₂O₃-MgO nanocomposite can maintain high transparency in the mid-infrared region, it loses optical properties in the visible and near-infrared regions as a result of severe optical scattering at grain boundaries. The large difference in the refractive index between the Y₂O₃ and MgO phases serves as a scattering center, which restricts applications to a specific wavelength region. To overcome this limitation of the grain boundary scattering, a grain size finer than the desired wavelength is a decisive factor for the Y₂O₃-MgO nanocomposite [7,8].

In order to ameliorate the suppression of domain coarsening of transparent polycrystalline ceramics, various sintering methods have been studied including spark plasma sintering (SPS) [9,10], hot-pressing [8,11,12] and microwave sintering [13,14]. Pressure-assisted consolidation, one of the most popular special sintering techniques, significantly lowers the sintering temperature and dwell time, resulting in a very fine-grained microstructure [15]. This process still has a limitation in that it does not greatly reduce the grain size of Y₂O₃-MgO nanocomposite or allow it to become transparent in the near-infrared wavelength region; this problem is similar to those of sintering methods that have been studied so far. Two-step sintering, introduced by Chen and Wang, was developed as an effective technique to avoid unintended grain growth while achieving high density during consolidation [16]. This process hampers junction and grain boundary mobility during sintering, thereby inhibiting grain coarsening. In general, high temperature is reached in the first step, and the next step drastically decreases the temperature, which is then held for a long time to obtain full densification without further grain coarsening. The mechanism of conventional two-step sintering is well-known from many previous studies [17,18]. In the normal two-step sintering procedure, the difference between the maximum and incubation temperatures is low, below 100°C, and the dwell time is prolonged to 20 h to achieve high fractional density of above 99%, such that inhibition of grain growth is restricted. Also, transparency of normal two-step sintered ceramics is introduced only when a further HIP procedure is applied. Therefore, an improved strategy should be considered. As mentioned above, the applied pressure can effectively reduce the sintering time and temperature. Nevertheless, until now research on transparent ceramics sintered by pressure-assisted two-step sintering has not been carried out.
systematically. Few researchers have studied how grain growth suppression of bulk TiO$_2$ based on two-step SPS and WC-MgO composite based on two-step hot-pressing can be achieved [19,20]. Indeed, investigation results of the mechanism and characterization of pressure-assisted two-step sintered transparent ceramics are still ambiguous. In addition, the final density and transmittance obtained by conventional two-step sintering are insufficient, revealing that optimized conditions of two-step hot-pressing will be essential in the field of future optical ceramics.

Here, to our knowledge for the first time, we report a two-step hot-pressing fabrication method for a Y$_2$O$_3$-MgO nanocomposite with enhanced near-infrared transparency. We produced the nanocomposite with fine grains and full densification by applying pressure and changing the maximum and holding temperatures. We demonstrate the difference between the paths of conventional and pressure-assisted two-step sintering. A kinetic window for two-step hot-pressing, indicating a region of densification without grain coarsening, is achieved. Furthermore, unlike the degraded optical properties of transparent ceramics fabricated by conventional two-step sintering, outstanding near-infrared transmittance can be obtained through microstructure control. We also demonstrate the relation between the optical-mechanical properties and the microstructure of the sintered Y$_2$O$_3$-MgO nanocomposite.

2. Methods

2.1. Y$_2$O$_3$-MgO nanopowder preparation

50:50 vol. % of Y$_2$O$_3$-MgO nanoparticles were synthesized by a glycine nitrate process (GNP). As starting precursor, yttrium nitrate hexahydrate (Y(NO$_3$)$_3$·6H$_2$O, ≥ 99.8%, Sigma Aldrich), magnesium nitrate hexahydrate (Mg(NO$_3$)$_2$·6H$_2$O, ≥ 99%, Sigma Aldrich) and glycine (NH$_2$CH$_2$COOH, ≥ 99%, Sigma Aldrich) were used. The raw materials were dissolved in distilled water (200 mL). The employed stoichiometric molar ratio of nitrate-to-glycine was 0.75. The precursor solution was put into a heating mantle, and then heat-treated for a few minutes. Following the auto-ignition reaction, the as-synthesized particles were ball milled for a half day using zirconia media in ethyl alcohol. The mixed solution was dried, and then calcination was performed at 800 °C for 5 h in air atmosphere in a furnace.

2.2. Sintering of Y$_2$O$_3$-MgO nanocomposite

The calcined Y$_2$O$_3$-MgO nanoparticles were sieved and then sintered via conventional and two-step hot-pressing techniques using a graphite sleeve and punches. Specimens with diameters of 12 mm and thicknesses of 0.85 mm were prepared. In conventional hot-pressing, pellets are heated to different temperatures from 1150 to 1400 °C at a heating rate of 15 °C/min under argon atmosphere. The samples in this study were maintained at maximum temperature for 1 h with uniaxial pressure of 50 MPa. In two-step hot-pressing sintering, the samples were...
first heated to the maximum temperature at a constant heating rate of 15 °C/min, and then cooled at 25 °C/min to the holding temperature. All samples were maintained at lower temperatures for 1 h, and then cooled at 5 °C/min to room temperature. For some specimens, to investigate the densification behavior of the Y$_2$O$_3$-MgO nanocomposite at low temperature, the holding time was changed from 1 to 10 h. After hot-pressing, to eliminate oxygen vacancies and residual carbon, a post-annealing process was performed at 1000 °C/min for 20 h in air.

2.3. Characterisation

The as-synthesized and calcined powders were characterized by scanning electron microscopy (SEM) (Philips XL 30 FEG, Philips) and conventional powder X-ray diffraction (XRD) with CuKα radiation at room temperature and a scan rate of 10 °C/min between 15 and 80 °C (SmartLab, Rigaku). The particle size and morphology were examined by transmission electron microscopy (TEM) via the generation of selected-area electron diffraction (SAED) images (Tecnai G2F30 S-Twin, FEI). After hot-pressing, the fractional densities of all sintered samples were measured by the Archimedean method. To obtain microstructure images, the pellets were thermally etched at 1100 °C for 2 h before using the SEM equipment (Philips XL 30 FEG, Philips). The grain size was obtained by multiplying the average linear intercept length of 200 grains by 1.56 [21]. UV-vis/NIR spectra were used to evaluate the line transmittance and cut-off wavelength in a range of 500–3000 nm (Lambda 1050, PerkinElmer). The Vickers hardness was also measured using a Vickers hardness tester (VLPK2000, Mitutoyo) with a load of 1 kgf placed on a polished surface.

3. Results and discussion

3.1. Powder characterisation

As can be seen in the SEM image in Fig. 1a, the as-synthesized Y$_2$O$_3$-MgO nanoparticles without any preliminary treatment are foam-like. This form is ascribed to the transient and auto-ignited reaction of the MgO nanoparticles without any preliminary treatment are foam-like. The synthesized Y$_2$O$_3$-MgO nanoparticles were sintered at different sintering temperatures. Dwell time for all sintered samples is 1 h, normal hot-pressing does not effectively prohibit grain growth during sintering at high temperature. Especially, grain coarsening accelerates with increase of the relative density above 99%. When the sample is consolidated by normal hot-pressing at 1400 °C, high fractional density of 99.5% can be obtained, but the grain size shows remarkable growth. This rapid grain growth has a negative effect on the mechanical properties, as well as on the short-wavelength transmittance of the nanocomposite, even though high temperature is required to eliminate residual pores for the transparent ceramics. On the other hand, the two-step hot-pressed samples, shown as open circles, indicate that the grain size does not increase much while the relative density increases. When the holding temperature reduced from 1400 °C to 1200 °C, the domain size is maintained at 159 nm, while the maximum density increases to full density. This result indicates that the grain size of this sample is 100 nm smaller than the grain size of the conventional sample hot-pressed at 1400 °C for 1 h. Especially, when considering the difference in refractive index between the two phases, a grain size of the Y$_2$O$_3$-MgO nanocomposite below 200 nm has a significant effect on the transparency in the near-infrared region. In spite of the grain growth of 15 nm, compared with the conventional sample hot-pressed at 1200 °C for 1 h, this sample clearly shows much better densification when the two-step hot-pressed is carried out.}

![Table 1](image)

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<th>Relative density (%)</th>
<th>Grain size (nm)</th>
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<td>99.5</td>
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*Parentheses represent standard deviation of average grain size.

![Table 2](image)

<table>
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<th>Relative density (%)</th>
<th>Grain size (nm)</th>
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*Parentheses represent standard deviation of average grain size.
External load has an influence on the densification rate. The densification rate can be derived as in Eq. (1) [24–26]:

$$\dot{\rho} = A d^n \sigma_{eff}^m \exp\left(-\frac{Q}{RT}\right)$$  \hspace{1cm} (1)

where $\dot{\rho}$ is the densification rate, $A$ is a constant, $d$ is the domain size, $p$ is the domain size exponent and $\sigma_{eff}$ is the effective stress, which decreases with increasing density. $Q$ is the activation energy for densification, $R$ is the gas constant and $T$ is the absolute temperature. When the fractional density is low, the stress exponent and $\sigma_{eff}$ are so high that the densification rate is fast. When the density is sufficiently high, $\sigma_{eff}$ is close to the applied stress, such that densification is delayed [24]. In this work, two-step hot-pressing with high initial density was employed to obtain full density. High applied stress and maximum temperature led to fast densification at short hot-pressing time, with a densification rate high enough that there was no problem achieving high density in 1 h [25]. When critical pressure was not applied to the sintered samples, the insufficient initial density failed to lead to full density in a short holding time. In general, the stress exponent $n$ is strongly dependent on the relative density and applied pressure. Even though a high initial density leads to slow densification rate, appropriate applied stress can increase that rate. Furthermore, since the two-step method is applied at an extremely low holding temperature and short holding time, grain boundary diffusion is inactive and surface diffusion is dominant [22]. So, when the initial density is low prior to the second step, densification does not take place. As a result, there is an apparent difference from the conventional two-step sintering method in that it is important to obtain high initial density prior to dropping the holding temperature in the current method.

Microstructural images of nanocomposites with different sintering conditions are provided in Fig. 3a–c. The data show obvious differences in grain size of nanocomposite according to sintering temperature and methods. As can be seen in Fig. 3a, when the nanocomposite is sintered by traditional hot-pressing at 1150 °C, fine grains with size of 98 nm can be achieved. However, the relative density is poor at 94.1% and there are many residual pores at the grain boundary. Even though the applied pressure serves as a driving force to stimulate densification via particle rearrangement, plastic sliding and deformation, the low temperature hampers grain boundary diffusion [27]. With an increase of the sintering temperature to 1400 °C, as can be seen in Fig. 3c, the sufficient thermal energy causes considerable grain growth to 282 nm. On the other hand, as shown in Fig. 3b, domain coarsening of the nanocomposite can be effectively limited, and there is an absence of fine pores during two-step hot-press sintering. This is because both the use of two-step hot-pressing and the Zener pinning effect between the two phases act as obstacles to mass transport [28].

Based on the obtained grain size and fractional density of the consolidated Y$_2$O$_3$-MgO nanocomposite, for the first time, a kinetic window for two-step hot-pressing to achieve full density without grain coarsening is displayed in Fig. 4. The indicated solid squares show the conditions to achieve densification while retaining grain size. The open squares above the upper boundary of the kinetic window indicate the grain growth, which follows the arrows during consolidation because high sintering temperature offers energy beyond the activation energy of grain boundary mobility. With the increase of the initial grain size, the upper temperature rises. One can note that the large initial grains require high thermal energy to move the grain boundary during two-step sintering [16]. The triangles indicate the exhausted densification at specific conditions of temperatures and initial grain size due to low thermal energy. The kinetic windows of the two-step hot-pressed Y$_2$O$_3$-MgO nanocomposite differ from those of normal two-step sintering in that, when the initial grain size is fine, exhaustion of densification happens even at the upper boundary. The boundary between the region of exhausted densification and grain growth is represented by a dashed line. As mentioned before, exhaustion occurs because there are to go with both insufficient grain boundary and volume diffusion due to the

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**Fig. 2.** (a) Relative density and grain size curves of Y$_2$O$_3$-MgO nanocomposite ceramics in two-step and conventional hot-press sintering conducted for 1 h. (b) Relative density and grain size curves of Y$_2$O$_3$-MgO nanocomposites sintered by two-step hot-pressing with different maximum temperatures. Open squares are values obtained by holding at maximum temperature for 0 h. Solid squares are values achieved after two-step hot-pressing for 1 h. The inset shows that full densification can be achieved without grain growth after two-step hot-pressing.
excessively low pre-firing and holding temperatures, as well as the short holding time. So, when the nanocomposite is sintered in two steps at 1300 °C to 1150 °C, there is no obvious densification. The presences of a grain boundary, residual pores and second phases as grain junctions may act as hindrances to grain growth at the lower temperature, such that grain size does not change. In addition, the slope of the upper boundary of the two-step hot-pressed Y2O3-MgO nanocomposite is much steeper than those of previously sintered ceramics studied using normal two-step sintering [29]. When the two nanocomposites were held for 1 h, it was possible to densify them to 90.1 and 99.0%, respectively. However, the fractional density achieved using the two sets of sintering conditions was not apparently improved, even when the holding time was increased to 10 h. This points out the hindrance of further densification, a zero-densification-rate period, with increasing of incubation time to 10 h. In two-step hot-pressing, the rate of densification is so fast that the plateau of densification is reached in a short time [25]. It is notable that the low incubation temperature of 1050 °C retards the volume and grain boundary diffusion that densification stops [30]. These results are in agreement with the requirement of high initial density, necessary to obtain full density and minimized grain size in the two-step hot-press sintering paths.

3.3. Optical properties

Due to the poor optical properties resulting from residual pores, no previous studies on transparent polycrystalline ceramics fabricated by two-step sintering have considered the effect of two-step consolidation on the transmittance. In this study, to verify the relationship between the microstructure and the near-infrared transmittance of the two-step hot-pressed Y2O3-MgO nanocomposite, in-line transmittance values according to a variety of hot-pressing conditions were determined and are shown in Fig. 5a–d. Results differ only in terms of sintering technique and temperature; the sintering time and applied pressure were maintained at 1 h and 50 MPa. To compare results obtained under identical conditions, the thickness of the polished samples was fixed at 0.85 mm. With different sintering conditions, due to the strong hydrophilic characteristics of the MgO phase in the nanocomposite, there was no apparent difference in the absorption peak assigned to H2O at 3 μm [9]. The near-infrared transmittance of the conventional hot-pressed Y2O3-MgO nanocomposite is illustrated in Fig. 5a. As the sintering temperature increased from 1150 to 1300 °C, the transparency in the near and mid-infrared wavelength regions improved, such that the transparency is in agreement with the enhanced relative density. With sintering at 1300 °C, the mid-infrared transmittance at 3 μm has a maximum value of 77.8%; the transmittance at 1.5 μm has a value of 68.2%. When the sintering temperature was increased to 1400 °C to
further eliminate residual pores in the microstructure, outstanding mid-infrared transmittance of 80% was achieved at 3 μm. However, the transmittance declines dramatically at 1.5 μm, and shows a value of 38.3%. It can be seen that the cut-off wavelength of the sample, indicating a transmittance of 10%, shifts from 914 to 1269 nm when the sintering temperature rises from 1300 °C to 1400 °C. Notwithstanding the sufficient elimination of residual pores with increasing sintering temperature, excessive grain coalescence leads to severe scattering of short wavelength incident beams at the grain boundary, such that near-infrared transmittance is considerably degraded [8]. It is notable that there are many limitations that prohibit these materials from being employed in various near-infrared applications. When two-step hot-pressing is carried out with different maximum and holding temperatures, the measured near-infrared transmittance is as shown in Fig. 5b–d. As can be seen in Fig. 5b, when the temperature is increased to 1300 °C and held at this low temperature for 1 h, the near-infrared transmittance obtained by two-step hot-pressing is much lower than the values obtained by normal hot-pressing. This is because, as shown in the kinetic window (Fig. 4), the sintering temperature is not high enough to cause densification. So, at the low maximum temperature, conventional hot-pressing is more efficient and can be used to obtain high transmittance in the near- and mid-infrared regions. When the maximum temperature is increased to 1350 °C, the mid-infrared transmittance can be improved (Fig. 5c). The near-infrared transmittance near 1500 nm is still slightly low; this is attributed to the presence of fine residual pores at the grain boundary, although the density can be improved to over 99% by two-step hot-pressing. By minimizing the grain size compared with that of the conventional hot-pressed sample, the cut-off wavelength can be further extended from 1073 nm to 803 nm in the short wavelength region. The transmittance values of the two-step hot-pressed Y2O3-MgO nanocomposite, obtained by raising the samples to 1400 °C and cooling them to low temperatures, are shown in Fig. 5d. It is notable that the infrared transmittance of the specimens, 80%, close to Stefanik’s theoretical value, obtained by two-step sintering is superior to that which can be obtained by conventional hot-pressing [31]. Compared with the near-infrared transmittance, a maximum transmittance of 74.4% at 1500 nm is obtained despite the lower holding temperature. In the case of conventional hot-pressing, after sintering at high temperature of 1400 °C, the domain size excessively increases, thereby sharply decreasing the near-infrared transmittance due to grain boundary scattering. The cut-off wavelength appears at 1269 nm. However, the cut-off wavelength is at 849 nm when the Y2O3-MgO nanocomposite is heated to 1400 °C and held at 1200 °C for 1 h. The slope of the transmittance does not decrease sharply in the near-infrared region, which is a result of overcoming the drawbacks of the Y2O3-MgO nanocomposite itself. The transmittance and scattering mechanism of the sintered Y2O3-MgO nanocomposite for the different sintering steps are represented in Fig. 6. In Fig. 6a, when there is no
soaking of the Y$_2$O$_3$-MgO nanocomposite at 1400 °C, the sample has poor infrared transmittance due to its low relative density, which resulted from insufficient densification; however, the cut-off wavelength of this sample is similar to the value of the two-step sintered sample. As illustrated in Fig. 6b, the large residual pores scatter the incident beam in a wide range of wavelengths. On the other hand, when conventional hot-pressing (1-step) is employed, the large grain size induces scattering of light of short wavelengths at the boundaries. These results show that two-step hot-pressing at optimized temperature can achieve densification and grain growth inhibition at the same time, thus making it possible to use translucent materials to develop new applications, including a tunable laser gain medium and opto-electronics that function in the near-infrared [32,33].

In the Y$_2$O$_3$-MgO nanocomposite, due to grain boundary scattering resulting from randomly oriented domains with large difference in refractive index, as previously mentioned, the transmittance in the near-infrared region is restricted. Therefore, it is essential to improve the near-infrared transparency by preventing grain boundary scattering [34]. To define the relation between the microstructure and the near-infrared transparency of the Y$_2$O$_3$-MgO nanocomposite, the cut-off wavelengths of all sintered samples are plotted in Fig. 7. It is apparent that there is the linear relationship between the average grain size and the cut-off wavelength, regardless of whether two-step hot-pressing is used or not. With the reduction of the grain size, the cut-off wavelength shifts to the short wavelength region. The minimum cut-off wavelength can be achieved at 790 nm when the Y$_2$O$_3$-MgO nanocomposite is sintered by pressure-assisted two-step sintering at steps of 1350 and 1150 °C. Apparently, since two-step hot-pressing can effectively reduce the grain size, the cut-off wavelength shifts to the short wavelength region. The cut-off wavelength of the conventional hot-pressed nano-composite moves to around 1300 nm due to severe grain interface scattering, which is associated with large grains in spite of the elimination of pores. Notably, the cut-off wavelength drastically rises at low sintering temperature, as indicated in the figure by triangles, although grain coalescence is prohibited. The cut-off wavelength rises because insufficient densification induces many residual pores in the microstructure, which impede light penetration in the whole infrared wavelength region. These results reveal that the finned-grained Y$_2$O$_3$-MgO nanocomposite obtained by pressure-assisted two-step sintering, with full density, can enlarge the high transmittance region to the short wavelength region.

### 3.4. Mechanical properties

The Vickers hardness values of the Y$_2$O$_3$-MgO nanocomposites fabricated by different sintering methods were measured (Fig. 8). Since the mechanical hardness of polycrystalline ceramics is strongly dependent on the average grain size, as described by the Hall-Petch relation:

\[ H = K \left( \frac{1}{d} \right)^m \]

where $H$ is the Vickers hardness, $d$ is the average grain size, $K$ and $m$ are constants. The experimental results show that the Vickers hardness values increase with decreasing grain size, as predicted by the Hall-Petch relation.

![Fig. 7. Relation of cut-off wavelength to average grain size for conventional and two-step hot-pressed Y$_2$O$_3$-MgO nanocomposite. Squares indicate conventional hot-pressing and circles indicate two-step hot-pressing. Triangles indicate that densification was exhausted during two-step hot-pressing.](image1)

![Fig. 8. Relation of Vickers hardness to average grain size for conventional and two-step hot-pressed Y$_2$O$_3$-MgO nanocomposite.](image2)
relation, the nanograins achieved through two-step hot-pressing method can improve the hardness through the dislocation pile-up mechanism [35,36]. Large numbers of grain boundaries act as physical obstacles to dislocation movement, leading to improved hardness [26,37]. The maximum hardness of 11.4 GPa is achieved in a two-step hot-press-sampled; this value is higher than that of a conventional sintered sample.

4. Conclusion

Pressure-assisted two-step sintering of Y2O3-MgO nanocomposite ceramics was investigated to achieve full density with extremely restricted grain coarsening. We successfully fabricated Y2O3-MgO nanocomposites with nano-grained microstructure. Compared with conventional two-step sintering, two-step hot-pressing can be performed at lower temperature. It is confirmed that densification without grain growth can occur only when the initial density is high. The kinetic windows required for two-step hot-pressed Y2O3-MgO nanocomposite to achieve densification without final stage grain coarsening are determined for the first time. The Y2O3-MgO nanocomposite with fine grains shows excellent transmittance in the near and mid-infrared wavelength regions. Especially, the influences of the maximum temperature in two-step hot-pressing on the microstructure and transmittance are demonstrated. We also show that the cut-off wavelength has a linear relationship with the average grain size. The Y2O3-MgO nanocomposite shows outstanding mechanical properties due to the effective inhibition of grain growth. Based on these results, we have demonstrated that a Y2O3-MgO nanocomposite fabricated by two-step hot-pressing can be a promising candidate for use in near-infrared optical materials. Pressure-assisted two-step sintering shows a meaningful possibility to restrict grain growth in a variety of nanoceramics, such that their optical and mechanical properties can be greatly improved through microstructure controls.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:10.1016/j.jeurceramsoc.2019.07.029.

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