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Synthesis and Characterizations of $\text{Ba}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$ Powder

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1. Introduction

A microwave communication system is important applications in the communication industry such as global communication satellites, cellular phones, radar detectors and wireless communication of the demands are rapidly rising in the present. Microwave dielectric ceramics were applied to the operation of filters and oscillators in several microwave systems as a small ceramic component. The general formula $\text{Ba}(\text{B}'_{1/3}\text{B}''_{2/3})\text{O}_3$ ($\text{B}' = \text{Mg, Zn, Ni or Co}$; $\text{B}'' = \text{Ta or Nb}$) ceramics are one of microwave dielectric which have attracted a great deal of attention and are currently being used for microwave devices due to its very high quality factors. Barium magnesium niobate ($\text{Ba}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$: BMN) compound is one of candidate materials of relative cheap and exhibits high performance microwave dielectric. Firstly, a high dielectric constant ($\epsilon_r = 32$) is needed, so that the materials can be miniaturized (because the size of a dielectric resonator $\propto 1/\epsilon_r^{1/2}$). Secondly, a high quality factor ($Q = 5600$) is very important for radio frequency system to keep a better selectivity and noise reduction, and a small temperature coefficient of resonance frequency ($\tau_f = 33 \text{ ppm}/^\circ\text{C}$) is also required so that the microwave circuits remain stable (Dias et al., 2001; Lin et al., 2006; Lian et al., 2004; Lian et al., 2005; Chen et al., 2006; Zhong-qing et al., 2004; Tian et al., 2009). So far, there have been only a few studies on BMN system and most of them prepared by chemical route due to its high purity and small particle size. However, it's expensive and complicated technique. Therefore, in this paper, BMN were prepared by conventional mixed-oxide technique which is the most economical and very simply consists of wet milling the individual oxides or other compounds that decompose to the oxides during calcining (Haertling, 1999). The characterizations of the phase formation, particle shape and particle size of all powders were investigated and experimental results are then discussed.

2. Experimental

The BMN powder was prepared by the convention mixed-oxide method. The reagent grades of BaCO_3 (Fluka, >98.5% purity), MgO and Nb_2O_5 (Aldrich, >99% purity) were used as raw materials in this system. The raw materials were weighed and mixed by ball milling technique with alumina balls in ethanol for 24 h. The mixtures were then dried into mixed powder. The powder processing was shown schematically in Fig. 1.

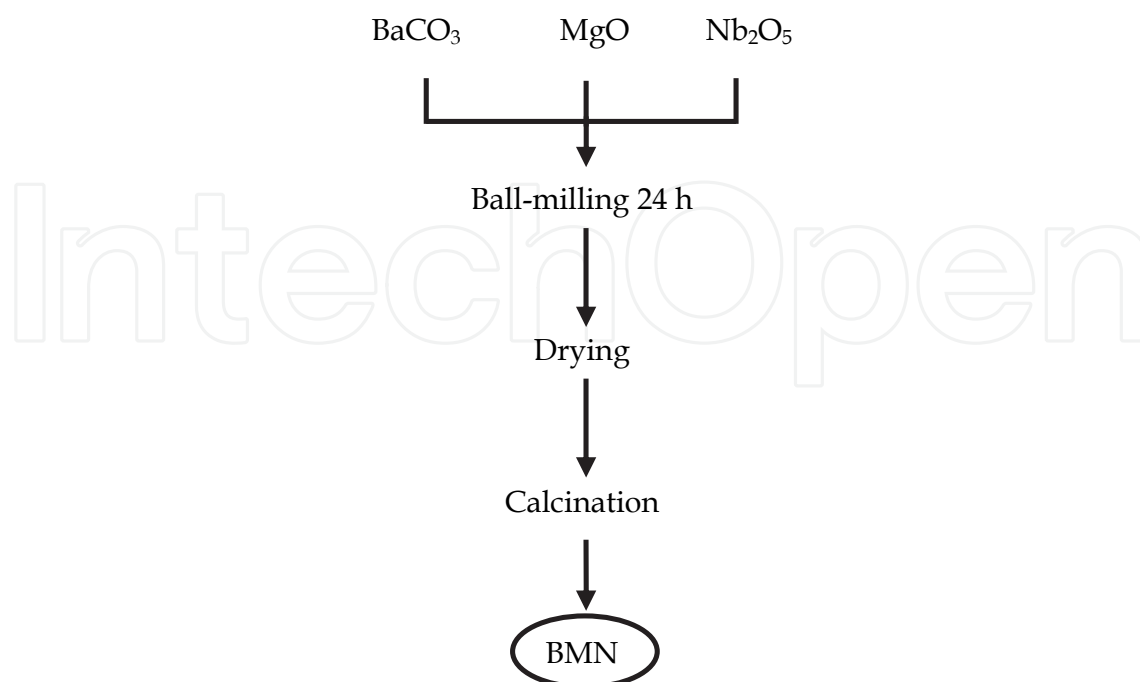


Fig. 1. Preparation route for the BMN powders.

The phase characterization process for all samples was examined using X-ray diffraction analysis (XRD) at room temperature in order to monitor phase evolution obtains calcination conditions that result in single phase BMN powder. The particle shape and particle size of powders were also observed using scanning electron microscopy (SEM).

3. Results and discussion

The TG-DTA curves of the BMN powders prepared by mixed-oxide method are illustrated in Fig. 2. In the temperature rang from room temperature to $\sim 1250^\circ\text{C}$. The TG curve shows two distinct weight losses. The first weight loss occurs at $\sim 300^\circ\text{C}$ and the second one between $600\text{--}1000^\circ\text{C}$. The both small endothermic and exothermic peaks are observed in the DTA curve which is related to the first weight loss ($\sim 1.0\%$). These DTA peaks can be attributed to the decomposition of the organic species from the milling process (Wongmaneerung et al., 2006; Ananta, 2004). After the first weight loss demonstrate a much sharper fall in specimen weight with increasing temperature from $\sim 600\text{--}1000^\circ\text{C}$. This precursor also exhibits a significantly larger over all weight loss ($\sim 14.2\%$). Corresponding to the second fall in specimen weight by increasing the temperature up to $\sim 1000^\circ\text{C}$ the solid-state reaction occurs the formation of some crystalline phase associated with BMN. A moderate exothermic peak at 930°C could be related to the crystallization of BMN phase as indicated in XRD patterns shown in Fig. 3. The broad exothermic characteristic in the DTA curve is found at the temperature range of $1000\text{--}1250^\circ\text{C}$, which has a maximum at $\sim 1018^\circ\text{C}$. It has been considered as the process of further crystallization in BMN phase (Lian et al., 2004). No further significant weight loss was observed for the temperatures above 1000°C in the TG curve, indication that the minimum firing temperature

to obtain BMN compound is in good agreement with XRD results (Fig. 3). These data were used to define the range of calcination temperatures for XRD investigation.

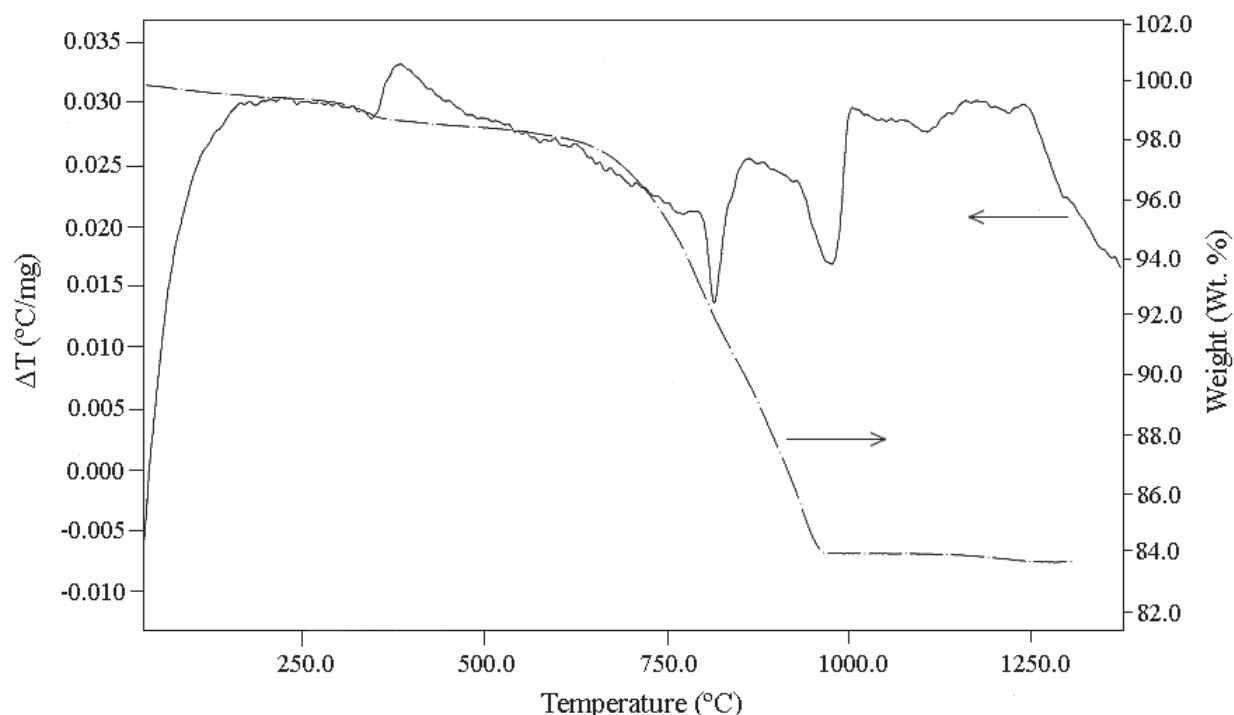


Fig. 2. TG-DTA curves for the mixture of BMN powders.

The XRD patterns of the BMN powder calcined in the temperature range of 800-1400°C for 4 h in air are shown in Fig. 2. The uncalcined powder show only X-ray peaks of BaCO₃, MgO and Nb₂O₅ precursors, which could be matched with JCPDS file no. 05-0378 (Powder Diffraction, 2000) 71-1176 (Powder Diffraction, 2000) and 80-2493 (Powder Diffraction, 2000), respectively. This result confirmed that no reaction had been initiated during the milling process. After calcination at 800°C, the crystalline phase of Ba(Mg_{1/3}Nb_{2/3})O₃ was developed accompanying with BaCO₃, MgO and Nb₂O₅ as separated phases. This observation agrees well with those derived from the TG-DTA results. As the temperature increased to 900°C, the intensity of the BMN peaks was further enhanced. Whereas the traces of minor phases of unreacted Nb₂O₅ could not be completely eliminated at 1100°C. This could be attributed to the poor reactivity of niobium species (Ananta et al., 1999). The peak of precursors gradually disappeared with increasing calcination temperature and reached to single BMN phase after calcination at 1200°C. This perovskite BMN powder can be matched exactly with JCPDS file no. 17-0173 for the hexagonal phase, in space group P3m1 with cell parameters of $a = 5.77$ pm and $c = 7.08$ pm (Powder Diffraction, 2000).

The morphological evolution of the calcined BMN powders was also revealed by SEM and showed Fig. 3. In general, the particles are agglomerated and basically irregular in shape with a substantial variation in particle size. The smallest and the biggest particle size of powders were estimated from SEM micrographs and listed in Table 1. From the results, it is seen that average particle size increases with increasing calcination temperature of BMN which can be attributed to the occurrence of hard agglomeration with strong inter-particle bond within each aggregates resulting from firing process.

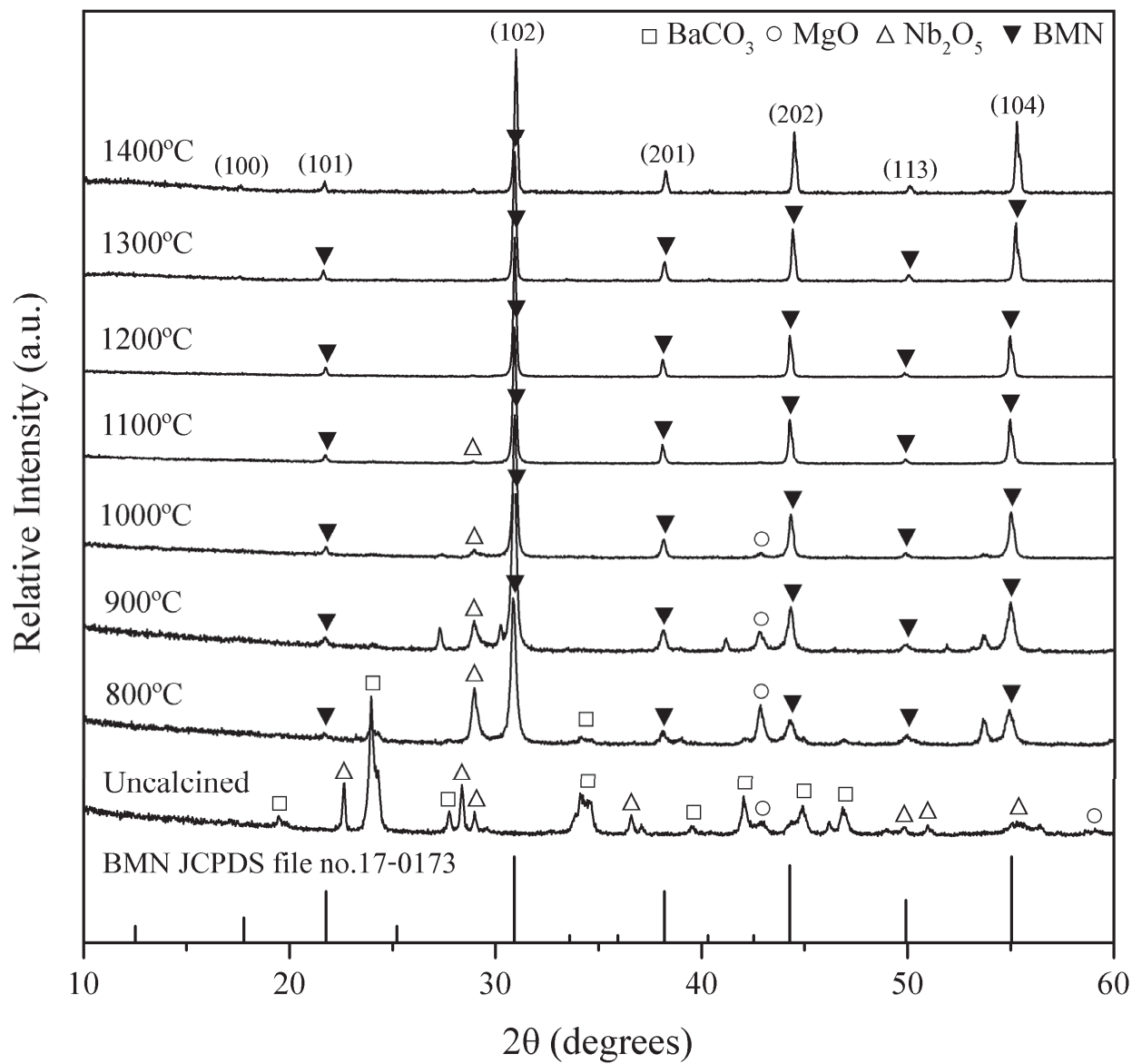
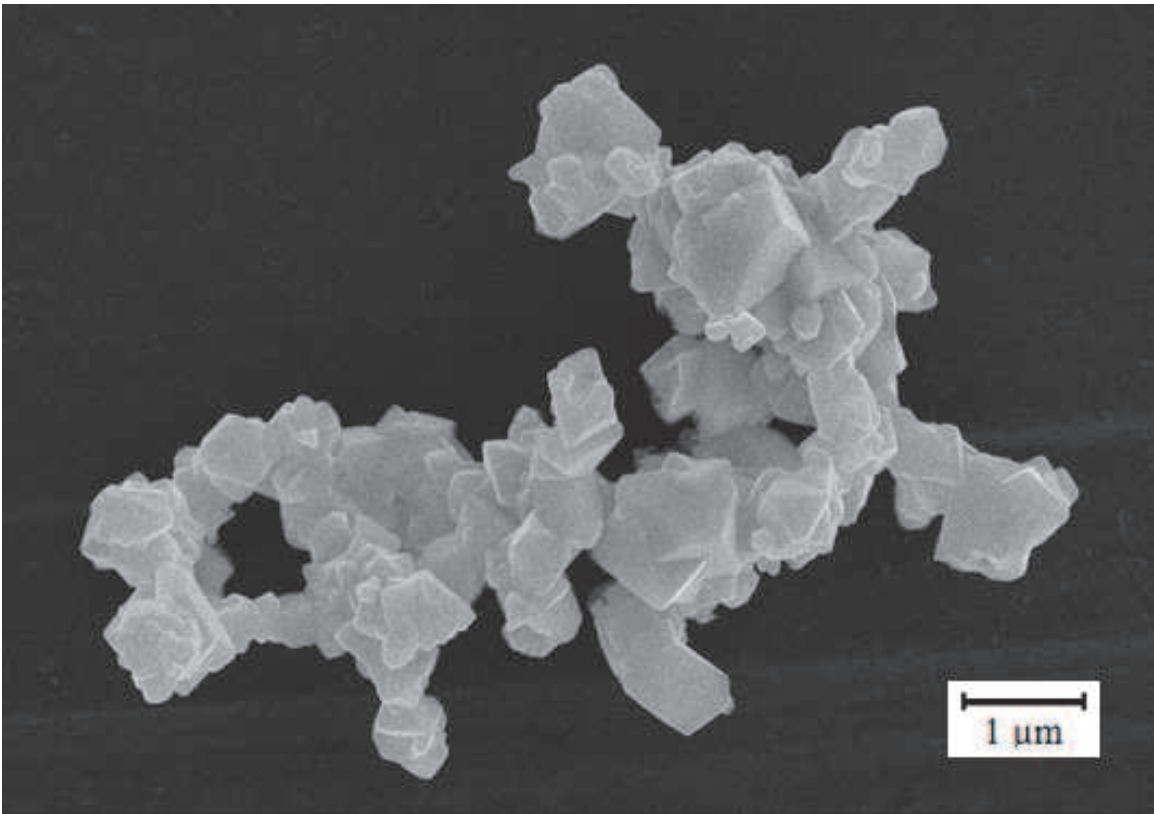
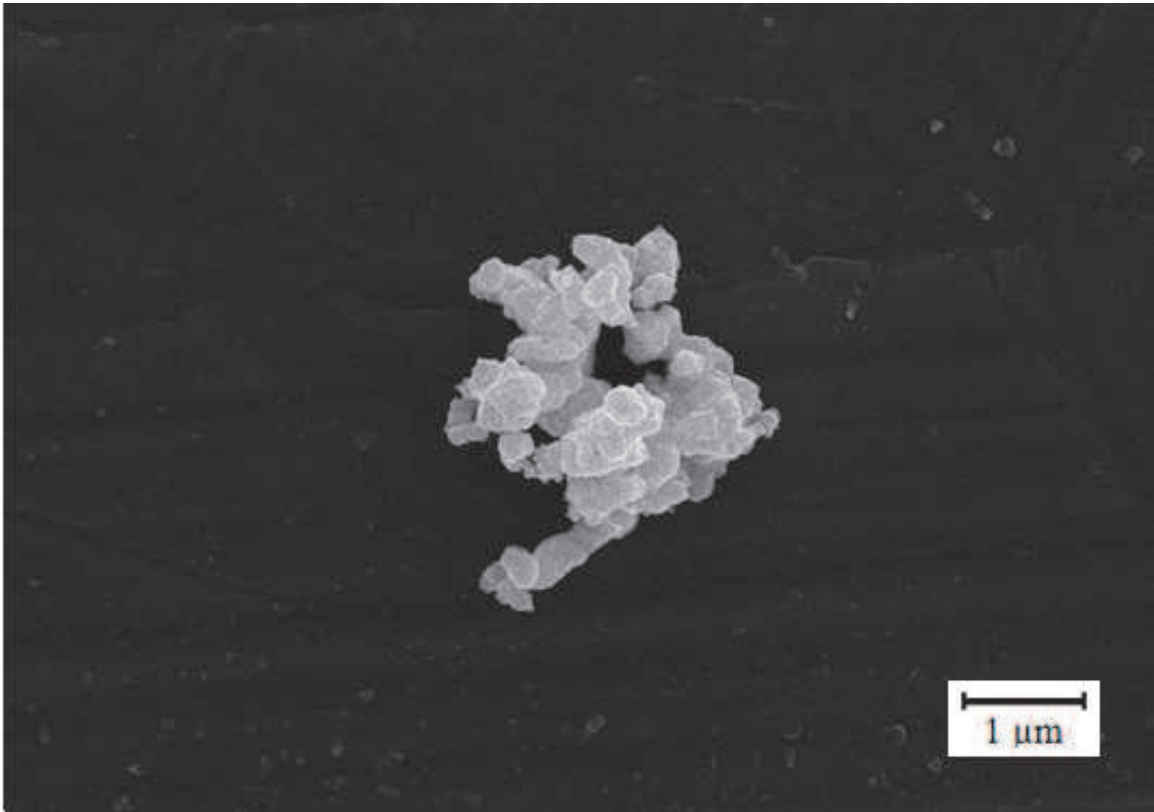


Fig. 3. XRD patterns of BMN powder calcined at various temperatures for 4 h with heating rates of 5°/min.



(a)



(b)

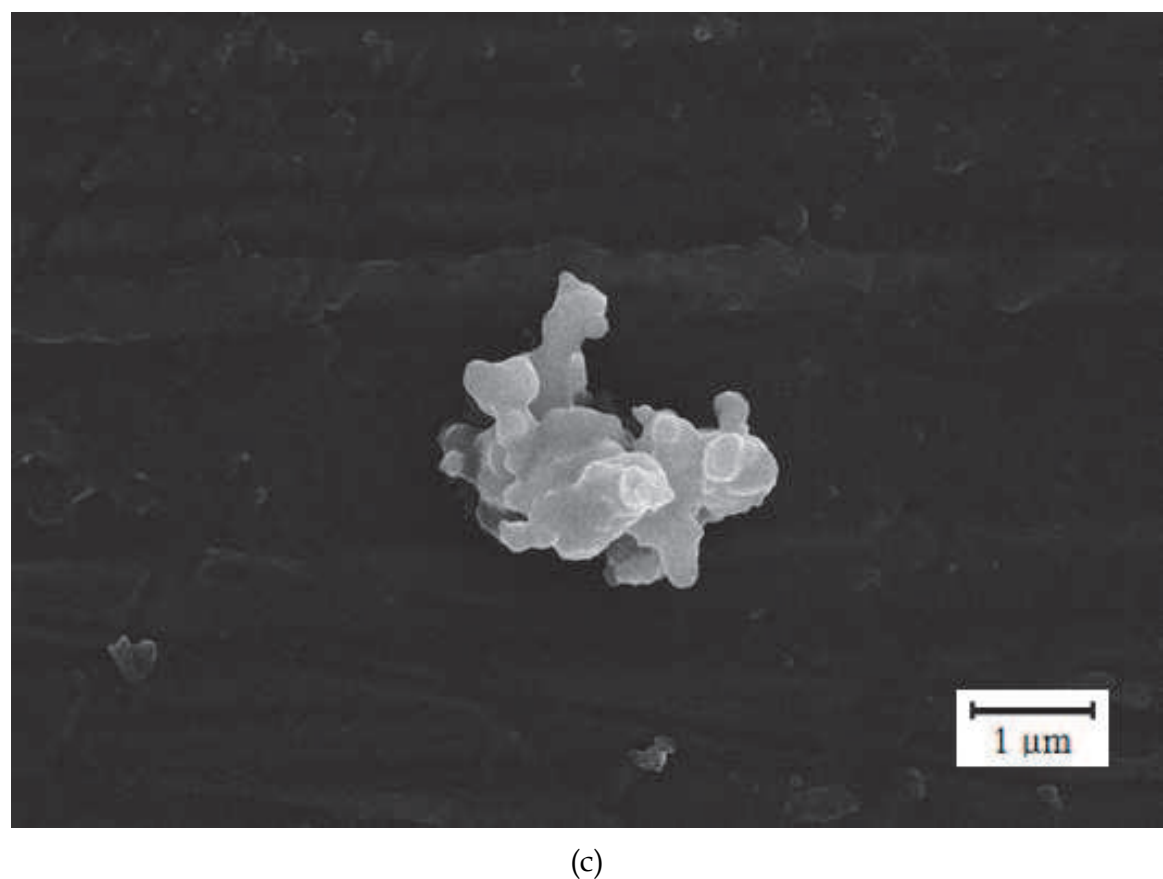


Fig. 4. SEM micrographs of BMN powder at different a calcination temperature of (a) 1200°C (b) 1300°C and (c) 1400°C for 4 h with heating rates of 5°/min.

Calcination temperature (°C)	Particle size rang (μm)	Average particle size (μm)
1200	0.21 - 1.45	0.65
1300	0.29 - 2.00	1.19
1400	0.35 - 4.00	1.56

Table 1. Particle size range and average particle size of BMN powder calcined at various temperatures for 4 h with heating rates of 5 °C/min.

4. Conclusion

The compound $\text{Ba}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$ powders were successfully prepared by the conventional mixed-oxide technique. The effect of calcination condition on the phase formation and microstructural evolution of this system was investigated via X-ray diffractometer (XRD) and scanning electron microscope (SEM), respectively. From the results, it can be concluded that single phase of $\text{Ba}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3$ powder has been obtained by using a calcination temperature of 1200°C for 4 h with heating rates of 5°C/min with particle size ranging from

0.21 to 1.45 μm . Moreover, average particle size increases with increasing calcination temperature.

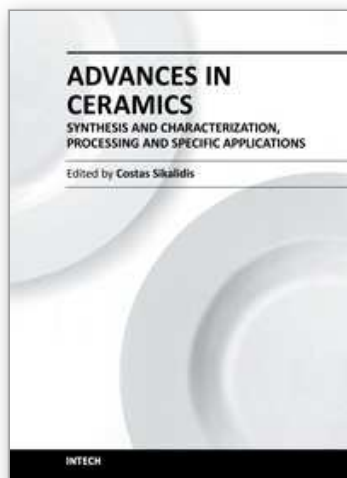
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