BT1-30vol%Ni

BT1 $10.5\,\mu\,\mathrm{m}$ $30 \,\mu \mathrm{m}$ Ni $+10 \,\mu \text{m}$

Fig. 1. TEM and SEM photographs of starting BaTiO₃ powders (BT1, BT2 and BT3) and Ni powder.

thick. The green density was 51-70% which increased with an increase in Ni content from 0 to 50 vol%. The powder compacts were set in a platinum boat and heated under atmospheres of purified Ar, unpurified Ar and air with an electric furnace. The gas flow rate was 100 ml/min. The heating rate was 10°C/min. The samples were kept at 1300°C for 4 h and cooled in the furnace. The mixture of BaTiO3 and NiO powders was sintered in the same process to investigate the solubility of NiO.

2.3 Evaluation of sintered bodies

The bulk density was determined by the Archimedes method in water. Crystal phases were identified by X-ray powder diffraction (Cu K α radiation) and the lattice parameters of BaTiO3 were measured using MgO powder as internal standard. Sintered bodies were cut with a diamond cutting machine. The section surface was polished with diamond paste. etched with HCl-HF mixed acid, and observed by SEM. The dielectric properties were measured at 10 kHz and 1 Vrms using LCZ meter (YHP, 4277 A). For the electrical measurement, both sides of sintered disk were polished and plated with gold electrodes by sputtering deposition.

3. Results and discussion

3.1 Sintering behavior and microstructure

BaTiO₃-Ni composites were obtained by sintering in Ar. The microstructures are shown in Fig. 2. Although Ni particles were dispersed in BaTiO₃ matrix when the content was small, they became to coalesce with increasing Ni content. The coales-

 $0 \mu m$ 10BT3-30vol%Ni BT3-29vol%NiO $+10 \mu m$ $+10 \,\mu \text{m}$

BT2-30vol%Ni

Fig. 2. SEM photographs of etched section surfaces of BaTiO₃-Ni and BaTiO₃-NiO sintered composites.

cence of Ni particles was remarkable for the composites of BT1 and BT2, whereas Ni particles were better dispersed in the composite of BT3 at the same Ni content. As seen in the BT3-composite, Ni particles were spherodized during sintering. When the sintering was performed in air, BaTiO3-NiO composites were produced because Ni was totally oxidized. NiO particles produced were still irregular-shaped as shown in Fig. 2. The grain size of BaTiO₃ was small as 1–3 μ m for BT1- and BT2-composites and 0.5–1 μm for BT3-composites.

The sintering data and the lattice parameters of BaTiO₃ are summarized in Table 1. The bulk density of sintered bodies was 95-99% of theoretical. The densification is thought in the present system to proceed by sintering of BaTiO3 particles because BaTiO₃ formed the matrix. BT1 consisted of large agglomerates but exhibited a good sinterability as well as fine particles of BT2 and BT3. The agglomerates may be crushed on powder mixing and press forming. Inclusion of Ni and NiO particles did not necessarily retard the sintering. After sintering, the crystal phase of BaTiO₃ was tetragonal. The lattice constant ratio, c/a, has been used to evaluate the tetragonality: c/a > 1 for tetragonal BaTiO₃. Sintered bodies of BaTiO₃ alone produced in the present work had the c/a ratios of 1.009–1.011. When Ni and NiO were included, the tetragonality decreased, suggesting the dissolution of NiO into BaTiO₃ as described later.



