Characterization of Surface Properties of $BaTiO_3$ Powder by XPS

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Abstract

The effects of particle size on the surface properties of hydro-thermally synthesized barium titanate powders were investigated by means of particle size analysis, specific surface area, SEM, zeta potential and XPS. Particle sizes were measured by laser light scattering and are in the range of 150 to 1100nm. Zeta potential increased with increasing particle size and it was large minus value in the range of particle size from 500 to 900nm, which seems to be related with the dissolution of Ba^{2+} ion in these particle sizes from the analysis of surface properties by XPS.

Keywords : Barium titanate, Particle size, Specific surface area, Zeta potential, XPS

1. Introduction

Barium titanate ($BaTiO_3$) has been used as capacitor, positive termperature coefficient of resistivity (PTCR)[1,2] and electro-optic materials due to its excellent dielectric, semi-conducting and transparent properties.

Ultra-fine powders should be used for improving the reliability of multilayer ceramic capacitor (MLCC) with high capacitance. Meanwhile, it is easy to agglomerate for the ultra-fine powders and difficult to disperse them in solvent such as water.

In this work, zeta potential, XPS, XRD and SEM were investigated for the ultra-fine $BaTiO_3$ suspensions in aqueous medium. The variation of zeta potential as a function of particle was described in terms of surface properties measured by XPS.

2. Experimental and Results

Ultra-fine barium titanate (Sakai chem., JPN) powders of BT01, BT02, BT03, BT04 and BT05 were used in this study. Table.1 shows the particle size measured by laser light scattering. The particle size is in the range of 150 to 1100 nm. Most of powders are agglomerated as shown in Fig.1. In particular, BT03 powders seem to be agglomerated strongly.

Table 1. Particle size measured by Laser scattering

Sample	BT01	BT02	BT03	BT04	BT05
Particle size (D50) nm	150	500	900	900	1100

It appears that the morphology of powders changes from round to facet surface with increasing particle size.

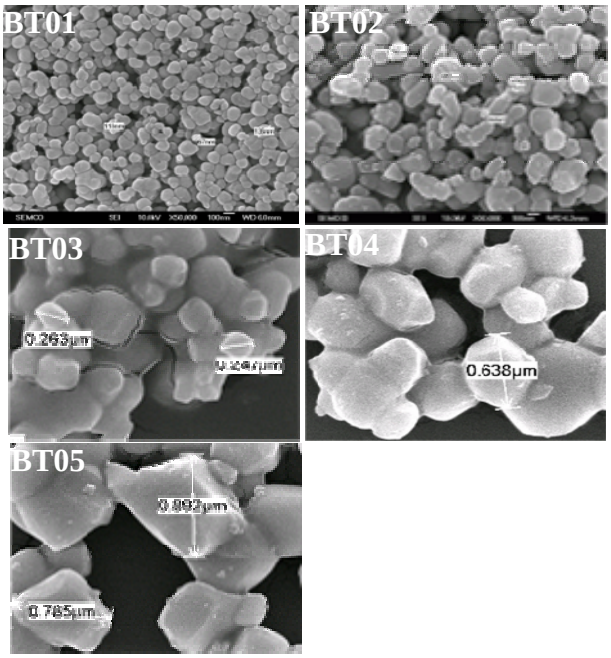

Fig. 1. SEM micrographs of $BaTiO_3$ samples.

Table 2. shows the specific surface area of BaTiO3 powders measured by BET (Brunauer-Emmett-Teller) method. Its specific surface area decreases slowly with increasing particle size of powders.

Table 2. Specific surface area of $BaTiO_3$ powders measured by BET method

Sample	BT01	BT02	BT03	BT04	BT05
BET (cm^2/g)	13.5	7.3	3.8	3.0	2.4

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Fig.2 shows the zeta potential of $BaTiO_3$ powders as a function of particle size. As the particle size decreases, zeta potential shows linear decrease relatively except BT03 powder. BT03 powder has a large minus value of zeta potential in comparison with other powders, which means that the surface of BT03 powder has larger minus charge than others. Ba^{+2} from $BaTiO_3$ powder tends to dissolve in water in terms of reaction as follows.

$$BaTiO_3(s) + H_2O(l) = Ba^{2+}(aq.) + TiO_2(rutile) + OH^-(aq.)$$

It is considered that a large minus value and deviation from the linearity of zeta potential for BT03 powder is related with more dissolution of Ba^{2+}.

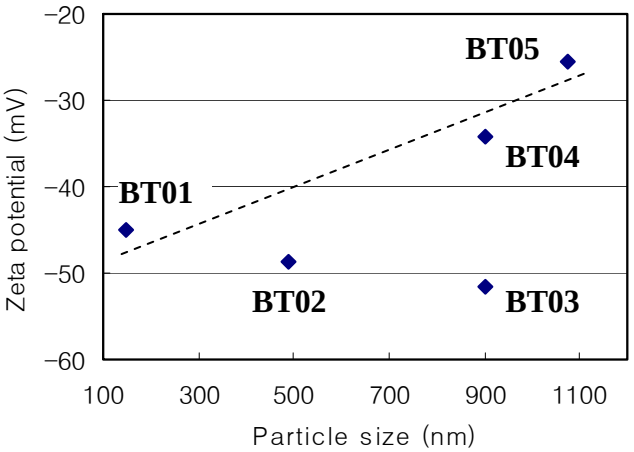

Fig. 2. Zeta potential of $BaTiO_3$ powders as a function of particle size.

The variation of zeta potential as a function of particle size has a great dependence of the chemical state of particles. In order to understand the chemical states of $BaTiO_3$ particles, the data of XPS for $BaTiO_3$ powders were obtained. Fig.3 shows XPS emission spectra (O 1s) of BaTiO3 powders. Their overall shapes of other powders are nearly symmetric except that emission spectra of BT02 and BT03 are asymmetric, have hump in the side of higher binding energy of peak point. It was reported that there are two spectra of 530.08 eV (Ti^{4+} - O) and 532.36 eV (OH^-) for O 1s spectra of TiO_2. Therefore, it is considered that the asymmetry and hump of O 1s spectra for BT02 and BT03 powders result from the adsorption of OH^- on their surface after dissolution of Ba^{2+} in water.

Fig.4 shows the peak positions of XPS O 1s spectra for BaTiO3 powders. The peak position of binding energy of BT02 and BT03 powders, 530~531 eV is lower than that of BT01, BT04, and BT05, 532~531. On the basis of some report concerning with surface reduction of particle, Lower binding energy of BT02 and BT03 seems to be related with higher concentration of O^{2-} ion on the surface of BaTiO3 powder in comparision with the other powders.

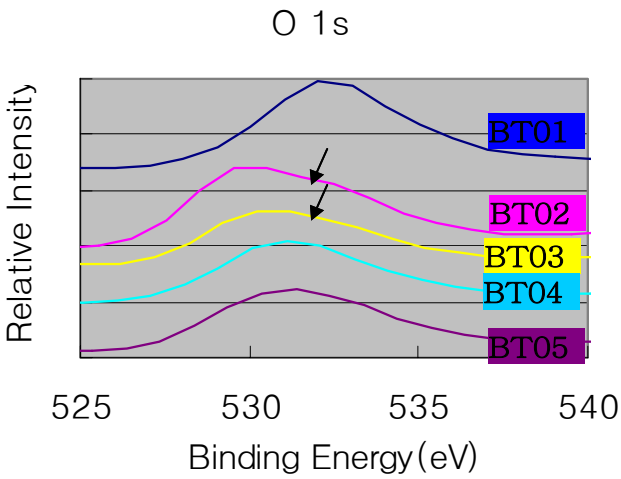

Fig. 3. XPS emission spectra (O 1s) of $BaTiO_3$ powders.

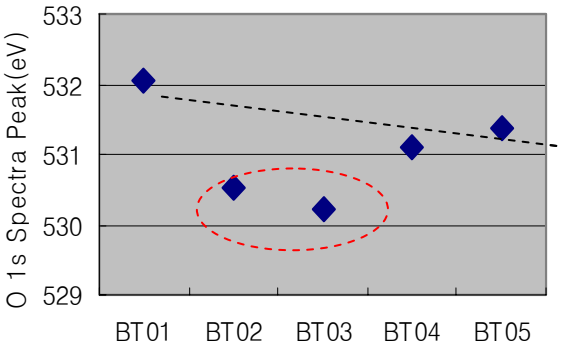

Fig. 4. Peak position of XPS spectrum of O 1s for $BaTiO_3$ powders.

3. Summary

The effects of particle size on the surface properties of hydro-thermally synthesized barium titanate powders were investigated by means of particle size analysis, specific surface area, SEM, zeta potential and XPS. The variation of zeta potential as a function of particle size can be described by the surface properties from XPS.

4. References

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