Three processing routes have been used to prepare barium titanate powders, namely conventional coprecipitation, single-microemulsion coprecipitation using diether oxalate as the precipitant, and double-microemulsion coprecipitation using oxalic acid as the precipitant. A single-phase perovskite barium titanate was obtained when the double-microemulsion-derived oxalate precursor was calcined for 2 hr at a temperature of as low as 550°C, compared to 600°C required by the single-microemulsion-derived precursor. A calcination for 2 hr at >700°C was required for the conventionally coprecipitated precursor in order to develop a predominant barium titanate phase. It was, however, impossible to eliminate the residual TiO₂ impurity phase by raising the calcination temperature, up to 1000°C. The microemulsion-derived barium titanate powders also demonstrated much better powder characteristics, such as more refined crystallite and particle sizes and a much lower degree of particle agglomeration, than those of the conventionally coprecipitated powder, although they contained ~0.2 wt% BaCO₃ as the impurity phase.

I. Introduction

Barium Titanate (BaTiO₃) is among the most important electroceramics for applications in electronics and microelectronics owing to its excellent ferroelectric, piezoelectric, and dielectric properties.1,2 It is widely used as the main constituent in many types of electroceramic devices such as multilayer capacitors and positive temperature coefficient resistors (PTCR). Solid-state reaction between BaCO₃ and TiO₂ in an equimolar ratio at temperatures >1200°C has often been used to prepare BaTiO₃ powders.3 Unfortunately, the conventional solid reaction is associated with many disadvantages including the high impurity and poor powder characteristics, represented by a coarse particle size, wide particle size distribution, irregular particle morphology, and a high degree of particle agglomeration. It is therefore not surprising to note that a large number of chemistry-based novel processing routes have been developed for the production of fine and homogeneous BaTiO₃ powders. These include coprecipitation,4–6 sol–gel processing,7–9 hydrothermal synthesis,10,11 reactions in molten salts,12,13 processing from polymeric precursors,14,15 and oxalate16–18 and citrate19–21 routes, as have been reviewed by Phule and Risbud4 and Chaput et al.22 Some of these novel processing routes have demonstrated many apparent advantages over the conventional solid reaction in producing a fine and homogeneous BaTiO₃ powder, although the degree of success varies considerably from one technique to another.

Several technologically important ceramic systems have recently been synthesized from water-in-oil microemulsions.23,24 The microemulsion-derived ceramic powders are much finer in particle size, narrower in particle size distribution, and higher in both composition homogeneity and sinterability than those prepared via many other chemistry-based processing routes.24,25 A water-in-oil microemulsion, which consists of an oil phase, a surfactant, and an aqueous phase, is a thermodynamically stable isotropic dispersion of the aqueous phase in the continuous oil phase.26 The size of the aqueous droplets is in the range of 5 to 20 nm, rendering the microemulsions optically transparent. A precipitation/coprecipitation reaction will be brought about in the nanosized aqueous domains when droplets containing appropriate reactants collide with each other. Each of these aqueous droplets will be acting as a nanosized reactor for forming nanosized precursor particles.

It is both scientifically interesting and technologically challenging to synthesize an ultrafine, preferably nanosized, barium titanate powder. Microemulsions offer the feasibility of refining the particle sizes to nanometer scale, although they are associated with such disadvantages as a low production yield and high production cost when the oil and surfactant phases are washed off. It is, however, possible to recycle them when the microemulsion processing technique is fully developed and matured for industrial applications. Schlag and co-workers27 have recently tried without success to synthesize fine barium titanate particles of high purity from an inverse microemulsion consisting of decane (oil phase), a nonionic surfactant (Gempol OX30, Hoechst, Switzerland), and an aqueous phase containing barium and titanium chlorides. Oxalate precipitates were formed in the nanosized microemulsion domains; however, they were unable to obtain a single-phase BaTiO₃ when the precursor was calcined at various temperatures ranging from 400° to 1200°C. They attributed the failure to the homogeneous dispersion of titanium in microemulsion droplets and the adverse effects of residual surfactant and chlorine counterions left in the precursor. To study the feasibility of deriving ultrafine BaTiO₃ powders from microemulsions containing no chlorine ions in the aqueous phase, both singlemicroemulsion and double-microemulsion processing routes were employed in the present work. In the double-microemulsion processing route, oxalic acid was used as the precipitant. Diethyl oxalate is sparingly soluble in water (aqueous droplets) and slowly releases oxalic acid when decomposed. It was chosen as the precipitant in the single-microemulsion route, in order to avoid a very rapid coprecipitation reaction which may result in the formation of a highly agglomerated and chemically heterogeneous BaTiO₃ powder. The microemulsion-derived BaTiO₃ powders were characterized in a close comparison with the one derived from conventional coprecipitation of oxalates.
II. Experimental Procedure

(1) Starting Materials

The starting materials used in the present work included barium nitrate (>99.0%, Merk, Germany), titanium(IV) chloride (>99.0%, Hayashi Pure Chemical Industries Ltd., Japan), a high-purity cyclohexane (AJAX Chemicals, Australia), a nonionic surfactant consisting of poly(oxyethylene)₉ nonyl phenol ether (NP5) and poly(oxyethylene)₁₀ nonyl phenol ether (NP9) (NP5:NP9 weight ratio: 2:1, Albright and Wilson Asia Pte Ltd, Singapore), oxalic acid dehydrate (>99.9%, J. T. Baker Int., USA), together with an ammonia solution (concentration: 28.0–30.0 wt%, J. T. Baker Inc., USA) and nitric acid (Hetalab Chemical Corp., USA).

(2) Aqueous Solution Containing 0.12M Ba(NO₃)₂ + 0.12M TiO(NO₃)₂

An aqueous solution of titanium oxynitrate was prepared by following the procedures of Kudaka et al.²³ For this, an appropriate amount of deionized water was added slowly to preweighed titanium tetrachloride (TiCl₄) which was kept cool (−0°C) and was constantly stirred. A cold ammonia solution (12 wt%) was then added to the aqueous solution, resulting in the formation of titanium hydroxide hydrates. In order to remove the chloride ions, the gelatinous precipitates were filtered and washed repeatedly using deionized water until the pH of the filtrate was close to 7.0 and no trace of chloride was detected by AgNO₃. Titanium oxynitrate in aqueous solution was then prepared by dissolving the white precipitates in an appropriate amount of 3.0M HNO₃, immediately followed by the concentration determination of Ti⁴⁺ using ICP (inductively coupled plasma, Thermo Jarrell Ash, IRIS/AP). The concentration of TiO(NO₃)₂ in the solution was adjusted to 0.24M by adding an appropriate amount of deionized water. To prepare the aqueous solution containing 0.12M Ba(NO₃)₂ + 0.12M TiO(NO₃)₂ with an equimolar ratio of Ba²⁺/Ti⁴⁺, an equal volume of 0.24M Ba(NO₃)₂ solution was combined with the aqueous solution of titanium oxynitrate.

(3) Phase Diagrams

The procedure of establishing a partial phase diagram at room temperature for the ternary system consisting of cyclohexane, NP5 + NP9, and an aqueous solution has been detailed elsewhere.²³,²⁴ To locate the demarcation between the microemulsion and nonmicroemulsion regions, the aqueous phase was titrated into a mixture of given cyclohexane-to-surfactant ratio. Partial phase diagrams at room temperature for two ternary systems were established. They consisted of cyclohexane, NP5 + NP9, and an aqueous phase containing 0.12M Ba(NO₃)₂ + 0.12M TiO(NO₃)₂ and 0.34M H₂C₂O₄ solutions, respectively. Precipitation reaction was made to take place when the two microemulsions were mixed together by vigorously stirring the mixture for 3 h at room temperature. The resulting precipitate was recovered and dried by following the same procedures as those in the SMC route.

(4) Preparation of BaTiO₃ Powders

As shown in Fig. 1, three processing routes were used to prepare BaTiO₃ powders in this work, namely, the conventional coprecipitation reaction (CCR), single-microemulsion coprecipitation (SMC) using diether oxalate as the precipitant, and double-microemulsion coprecipitation (DMC) using oxalic acid as the precipitant.

For the CCR, 200 mL of the aqueous solution containing 0.12M Ba(NO₃)₂ + 0.12M TiO(NO₃)₂ was titrated dropwise into 200 mL of 0.38M oxalic acid (H₂C₂O₄) solution while being vigorously stirred. The resulting coprecipitates were washed repeatedly using deionized water and recovered by centrifugation, followed by vacuum-drying at room temperature for 48 h.

In SMC, a microemulsion composition consisting of 64.0 wt% cyclohexane, 16.0 wt% NP5 + NP9, and 20.0 wt% aqueous phase containing 0.12M Ba(NO₃)₂ + 0.12M TiO(NO₃)₂ was first prepared. The concentration of Ba(NO₃)₂ and TiO(NO₃)₂ was 0.12M in the overall microemulsion composition. The coprecipitation was brought about by mixing 780 g of microemulsion composition with 21.7 g of diether oxalate via vigorously stirring the mixture for 24 h. At the same time, the mixture was heated slowly to 40°C. To retrieve the coprecipitates formed in the microemulsion, the oil and surfactant phases were washed off using distilled ethanol and the precipitants were then recovered by centrifugation, followed by washing using deionized water and vacuum-drying at room temperature for 48 h.

For DMC, two microemulsion compositions, both consisting of 64.0 wt% cyclohexane, 16.0 wt% NP5 + NP9, and 20.0 wt% aqueous phase, were prepared. The aqueous phases in the two microemulsions were 0.12M Ba(NO₃)₂ + 0.12M TiO(NO₃)₂ and 0.34M H₂C₂O₄ solutions, respectively. Coprecipitation reaction was made to take place when the two microemulsions were mixed together by vigorously stirring the mixture for 3 h at room temperature. The resulting precipitate was recovered and dried by following the same procedures as those in the SMC route.

(5) Precursor and Powder Characterizations

The as-dried precipitants from the above three processing routes were characterized using thermogravimetric analysis (TGA2950, Du Pont Instruments, Wilmington, DE) and differential thermal analysis (DTA1600, Du Pont Instruments) with alumina as the reference at a heating rate of 10°C/min in air from room temperature up to 950°C. They were then calcined in air at various temperatures, up to 800°C, followed by phase identification performed at room temperature using a (CuKα) X-ray diffractometer (PW1729, Philips, 7602 EA Almelo, The Netherlands) and a Raman scattering spectrometer (Ramascop...
A transmission electron microscope (100CX, JEOL, Japan) and a scanning electron microscope (JSM-35CF, JEOL, Japan) were employed to analyze the specific surface area and particle/agglomerate morphology of these powders, respectively. The analysis of carbon content was performed on the powders calcined at 800°C using a Perkin-Elmer elemental analyzer (2400 CHN) and the Ba/Ti ratios in these powders were determined using ICP (IRIS/AP, Thermo Jarrell Ash, Ton Beach, FL), a transmission electron microscope (100CX, JEOL, Japan) and a scanning electron microscope (JSM-35CF, JEOL, Japan). A gas sorption analyzer (Nova 2000, Quantachrome Corp., Boynton Beach, FL), a transmission electron microscope (100CX, JEOL, Japan) and a near-infrared laser (4782 nm) as the exciting source. They were also characterized using a FTIR spectrometer (FTS135, BIO-RAD Laboratories, Inc., Cambridge, MA) over the spectrum range 4000–400 cm⁻¹ and the spectra were averaged out from 64 scans with a nominal resolution of 2 cm⁻¹ (KBr pellet). On the basis of XRD line broadening at half-maximum of the (110, 101) peaks, crystallite sizes in the calcined BaTiO₃ powders at various temperatures were estimated using the Scherrer equation. The particle/agglomerate size distribution was measured using a laser scattering particle size analyzer (LA-910, Horiba, Miyanohigashi Minami-Ku, Kyoto, Japan). A gas sorption analyzer (Nova 2000, Quantachrome Corp., Boynton Beach, FL), a transmission electron microscope (100CX, JEOL, Japan) and a scanning electron microscope (JSM-35CF, JEOL, Japan) were employed to analyze the specific surface area and particle/agglomerate morphology of these powders, respectively. The analysis of carbon content was performed on the powders calcined at 800°C using a Perkin-Elmer elemental analyzer (2400 CHN) and the Ba/Ti ratios in these powders were determined using ICP (IRIS/AP, Thermo Jarrell Ash, USA).

III. Results and Discussion

Figure 2(a) shows the partial phase diagram established at room temperature for the ternary system consisting of cyclohexane, NP5 + NP9, and aqueous solution containing 0.12M Ba(NO₃)₂ + 0.12M Ti(OH)₅O₂. Similarly, the partial phase diagram for the ternary system containing 0.34M H₂C₂O₄ as the aqueous phase is shown in Fig. 2(b). In both systems, the microemulsion region widens with increasing NP5 + NP9 to cyclohexane ratio, although the system containing 0.12M Ba(NO₃)₂ + 0.12M Ti(OH)₅O₂ exhibits a wider microemulsion region than that containing 0.34M H₂C₂O₄. The surfactant-rich compositions are highly viscous, which poses problems in obtaining a homogeneous mixture. In both ternary systems, the composition of 64.0 wt% cyclohexane, 16.0 wt% NP5 + NP9, and 20.0 wt% aqueous phase is within the microemulsion region.

Figure 3 shows the TGA and DTA traces at a heating rate of 10°C/min in air for precursors CCR, SMC, and DMC. When the conventionally coprecipitated precursor was heated from room temperature to 950°C, it exhibited three apparent falls in specimen weight over the temperature ranges from 50°C to 120°C, 330°C to 400°C, and 680°C to 710°C, respectively. Each of these falls in specimen weight corresponds to an endothermic reaction on the DTA curve. It is generally accepted that the formation of barium titanate from barium titanium oxalate precursors involves the following three steps with increasing calcination temperature, although there are strong arguments for the types of intermediate phases involved: (i) dehydration of the oxalate precursor, for example, the conversion of BaTiO(C₂O₄)₂·4H₂O to BaTiO(C₂O₄)₂; (ii) decomposition of BaTiO(C₂O₄)₂ to form intermediate phases, such as BaCO₃, TiO₂, BaTi₂O₅, and Ba₂Ti₃O₇CO₃; and (iii) formation of BaTiO₃ as a result of the reaction between the intermediate phases or the decomposition of the metastable Ba₂Ti₃O₇CO₃.

The three falls in specimen weight are therefore believed to correspond to the above three stages, respectively. There is a close agreement between the overall weight loss of 47.7% observed when the precursor is heated to 950°C and that expected on the basis of the conversion from BaTiO(C₂O₄)₂·4H₂O to BaTiO₃ (46.8%). To further support this, Fig. 4 shows the FTIR spectra for precursors CCR, SMC, and DMC calcined at various temperatures, respectively. The strong absorption band at around 1440 cm⁻¹, together with those at ~2450, 1752, 1060, and 859 cm⁻¹, detected in the conventionally coprecipitated powder calcined at 550°C indicates the presence of a carbonate phase. These absorption bands decrease in intensity with increasing calcination temperature and most of them will almost completely disappear from the spectrum when the calcination temperature is raised to 800°C. As will be discussed later, XRD phase analyses show that the carbonate phase is crystalline BaCO₃, which is therefore believed to be one of the major phases involved in the conversion of barium titanate.
oxalates to barium titanate with increasing calcination temperature. Most of the carbonate-related absorption bands were also observed in precursors SMC and DMC when they were calcined at low temperatures. As indicated in Fig. 4, however, both SMC and DMC exhibit a much weaker absorption band at around 1440 cm\(^{-1}\) than that for the conventionally coprecipitated powder when they are all calcined at 800°C for 2 h. It was estimated on the basis of these FTIR analyses that ~0.2 wt% BaCO\(_3\) existed in the two-microemulsion-derived barium titanate powders. The impurity level was also indicated using a
Perkin-Elmer elemental analyzer (2400 CHN) for carbon content in these powders.

Three steps in specimen weight, each corresponding to an endothermic reaction, are also observed in the precursor derived via the single-microemulsion route. The first two of the three occur over almost the same temperature ranges as those for the conventionally coprecipitated precursor. The remaining one, however, occurs at \( \sim 640°C \), which is almost 55°C lower than that in the conventionally coprecipitated precursor. Similarly, the first two of the major falls in specimen weight observed for the precursor derived via the double-microemulsion route occur over temperature ranges similar to those for the conventionally coprecipitated precursor. This is followed by a two-step fall in specimen weight over the temperature range of \( 480° \) to \( 595°C \). A minor and broadened exothermic peak correlates to the first step of the fall in specimen weight at around \( 505°C \), presumably due to the oxidation of organic residuals. The final fall in specimen weight at around \( 590°C \) is associated with an endothermic peak observed on the DTA curve. It is apparent that the weight loss in precursor DMC is completed at \( \sim 595°C \), which is much lower than that of the conventionally coprecipitated precursor. This is followed by a two-step fall in specimen weight over the temperature range of \( 480° \) to \( 595°C \). A minor and broadened exothermic peak correlates to the first step of the fall in specimen weight at around \( 505°C \), presumably due to the oxidation of organic residuals. The final fall in specimen weight at around \( 590°C \) is associated with an endothermic peak observed on the DTA curve. It is apparent that the weight loss in precursor DMC is completed at \( \sim 595°C \), which is much lower than that of the conventionally coprecipitated precursor.

To study the phase development with increasing calcination temperature in each of the above three precursors, they were calcined in air to various temperatures in the range from \( 550° \) to \( 1000°C \) at a heating rate of \( 10°C/min \), followed by phase and structure analyses using XRD and Raman spectrometer. Figure 5 shows the XRD patterns for precursors CCR, SMC, and DMC calcined at various temperatures over the range from \( 550° \) to \( 800°C \), respectively. BaCO\(_3\) was the predominant crystalline phase detected in the conventionally coprecipitated precursor calcined at \( 550°C \), indicating that it was one of the major intermediate phases involved during the conversion of the oxalate precursor into BaTiO\(_3\) with increasing calcination temperature. BaTiO\(_3\) became the predominant phase when the precursor was calcined at \( 600°C \). However, the carbonate phase was not completely eliminated until the calcination temperature was raised to \( 700°C \). In contrast, a high-purity perovskite BaTiO\(_3\) phase was obtained in the precursor derived via the single-microemulsion processing route at a calcination temperature of \( 600°C \). A further reduction in the formation temperature of a single-phase perovskite BaTiO\(_3\) powder was observed in the precursor derived via the double-microemulsion route (\( 550°C \) for 2 h). These XRD phase analysis results show that the three precursors are considerably different in the calcination temperature required to develop a high-purity BaTiO\(_3\) phase.

To further support the belief that the formation of BaTiO\(_3\) in the two-microemulsion-derived oxalate precursors is completed at a lower temperature than that in the conventionally coprecipitated precursor (more specifically, the formation temperature follows DMC < SMC < CCR), the powder precursors calcined at various temperatures ranging from \( 700° \) to \( 1000°C \) were characterized using a Raman spectroscopy. Figure 6 shows the Raman spectra of powders CCR, SMC, and DMC calcined at \( 700° \), \( 750° \), \( 800° \), and \( 1000°C \). They all exhibit two distinct bands at \( -310 \) and \( 720 \) cm\(^{-1} \), indicating the presence of tetragonal phase. The very strong resemblance of these spectra...
to that of a commercially available tetragonal barium titanate powder (99.9%, Johnson Matthey, USA) confirms that they are in the tetragonal form. Figure 6 also reveals two minor bands at ~640 and 1060 cm⁻¹ respectively, occurring in the conventionally coprecipitated barium titanate powder calcined at 700°C. They were not observed in the two-microemulsion-derived powders calcined at temperatures above 600°C. The 640 cm⁻¹ band corresponds to the principal band of TiO₂, which, as an impurity phase in the conventionally coprecipitated barium titanate powder, cannot be eliminated by calcination at temperatures up to 1000°C. The 1060 cm⁻¹ band is related to the carbonate, which was observed in the conventionally coprecipitated precursors calcined at temperatures below 700°C. This is consistent with the suggestion that the formation of BaTiO₃ involves BaCO₃ as an intermediate phase, which reacts with TiO₂ to form BaTiO₃.30–32

The BaTiO₃ powders derived via the above three processing routes are also different in crystallite and powder characteristics, such as the crystallite size, particle size, and particle morphology. Figure 7(a) plots the average crystallite size as a function of calcination temperature for powders CCR, SMC, and DMC. At each calcination temperature, the double-microemulsion-derived barium titanate powder exhibits the smallest crystallite size, followed by that derived via the single-microemulsion route. The conventionally coprecipitated barium titanate shows a much larger crystallite size than those of the other two. As expected, the crystallite size increases with increasing calcination temperature over the range from 550°C to 800°C and with increasing calcination time at 750°C for all three powders, although the increase rate varies with calcination time as shown in Fig. 7(b).

Table I shows the specific surface area (BET) of calcined BaTiO₃ powders at various temperatures for 2 h, derived via the CCR, SMC, and DMC routes, respectively. The equivalent discrete particle sizes were worked out by assuming that they all consisted of monosized particles. The conventionally coprecipitated powder shows the lowest specific surface area

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<th>Temp (°C)</th>
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<th>DMC</th>
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Fig. 7. (a) Average crystallite size estimated on the basis of peak (110) broadening as a function of temperature for the powders derived via the conventional coprecipitation reaction, and the single-microemulsion and double-microemulsion processing routes, respectively. (b) Average crystallite size estimated on the basis of peak (110) broadening as a function of annealing time at 750°C for the powders derived via the conventional coprecipitation reaction, and the single-microemulsion and double-microemulsion processing routes, respectively.

Fig. 8. Particle/aggromerate size distribution of BaTiO₃ powders derived via the conventional coprecipitation reaction, the single-microemulsion and double-microemulsion processing routes, and calcined at 650°C, 700°C, and 750°C, respectively.
among the three at each calcination temperature and furthermore it demonstrates a dramatic fall in specific surface area when the calcination temperatures is raised from 650° to 700°C. In contrast, the double-microemulsion-derived powder exhibits the highest specific surface area and therefore the smallest discrete particle size at each calcination temperature, followed by the single-microemulsion-derived powder. This demonstrates the effectiveness of microemulsion processing in obtaining an ultrafine BaTiO₃ powder.

As shown in Fig. 8, the two-microemulsion-derived barium titanate powders were also smaller in average particle/agglomerate size than that of the conventionally coprecipitated powder, as measured using light scattering technique. To reduce the effect of particle agglomeration on the measured particle size distributions, each powder was dispersed in deionized water and ultrasonically stirred for 20 min before a test was carried out. It is apparent that the average particle/agglomerate size follows the order of CCR > SMC > DMC at each calcination temperature. To further demonstrate the particle/agglomerate size distribution and particle morphology, Figs. 9(a–c) and 10(a–c) are SEM micrographs showing the microstructure of the three barium titanate powders calcined at 650° and 800°C, respectively. Particle agglomerates of 5 to 10 μm in sizes dominate the conventionally coprecipitated powder.

Fig. 9. Three SEM micrographs showing the powders calcined at 650°C for 2 h, derived via (a) the conventional coprecipitation reaction, (b) the single-microemulsion and (c) double-microemulsion processing routes, respectively.

Fig. 10. Three SEM micrographs showing the powders calcined at 800°C for 2 h, derived via (a) the conventional coprecipitation reaction, (b) the single-microemulsion and (c) double-microemulsion processing routes, respectively.
although discrete particles are much smaller. The single-microemulsion-derived powder consists of loosely packed particle agglomerates 3 to 5 μm in size. In a remarkable contrast, the double-microemulsion-derived barium titanate powder consists of well-dispersed spherical barium titanate particles ~100 nm in diameter, which are almost agglomerate-free. Figures 11(a–c) are three TEM micrographs further showing the differences among the three barium titanate powders calcined at 800°C in crystallite size, particle morphology, and the degree of particle agglomeration.

As expected on the basis of the refinement in particle characteristics, the two-microemulsion-derived barium titanate powders exhibit better sintering behavior than that of the conventionally coprecipitated one. For example, the former two were sintered to ~96% theoretical density at 1250°C for 2 h, compared to ~92% for the latter at the same temperature. A sintered density of >99% theoretical was obtained for the two-microemulsion-derived barium titanates at 1325°C for 2 h, in contrast to <96% for the conventionally coprecipitated barium titanate at the same temperature. One of the concerns in employing microemulsions for synthesizing barium titanate is the Ba/Ti ratio in the resulting powders. As in many other wet chemical routes, there is a possibility of losing more barium than titanium because of their difference in solubility. However, the degree of Ba deficiency in the two-microemulsion-derived barium titanate powders prepared in this work is minimal, as supported by a Ba/Ti ratio of ~0.996 worked out for them using ICP.

IV. Conclusions

Barium titanate powders were prepared via three chemical-based processing routes, namely, conventional coprecipitation, single-microemulsion coprecipitation using diether oxalate as the precipitant, and double-microemulsion coprecipitation using oxalic acid as the precipitant. A single-phase perovskite barium titanate was obtained when the double-microemulsion-derived oxalate precursor was calcined for 2 h at 550°C, compared to 600°C required by the single-microemulsion-derived precursor. A calcination at >700°C for 2 h was required for the conventionally coprecipitated precursor in order to develop a predominant barium titanate phase. It was, however, impossible to eliminate the residual TiO2 phase in the coprecipitated BaTiO3 powder by raising calcination temperature up to 1000°C. All three barium titanate powders exhibited a tetragonal structure when calcined at 700°C and above. The microemulsion-derived barium titanate powders also demonstrated much better powder characteristics, such as more refined crystallite and particle sizes and a much lower degree of particle agglomeration, than those of the conventionally coprecipitated powder.

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