

EFFECT OF GRAIN SIZE OF PRECURSOR POWDER ON THERMOELECTRIC PROPERTIES OF TEXTURED $\text{Ca}_3\text{Co}_4\text{O}_9$ COMPOUNDS

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ABSTRACT

Partially Bi substituted $[\text{Ca}_2\text{CoO}_3]_p\text{CoO}_2$ ceramics were synthesized by a hot-forging technique and the effect of the Bi-substitution on the microstructure and thermoelectric properties evaluated. The average grain size of the precursor powder prepared by a solid-state reaction increased with the increase of the Bi content, and the electrical resistivity (ρ) of the hot-forged sample was reduced accordingly. Since the Seebeck coefficient (S) was slightly increased by the Bi-substitution and less affected by the grain size, the power factor ($PF=S^2/\rho$) was improved. On the other hand, the ρ value of the hot-forged sample was reduced by using large grained precursor powder prepared by a solution growth method. Unlike in the case of the former samples made from as-sintered powder, the ρ value of the sample made from large grained powder was slightly increased with the increase of the Bi content. These results indicated that the main advantage of the Bi-substitution for the reduction of ρ value of hot-forged sample is the enhancement of the grain growth during the heat treatment processes.

INTRODUCTION

The recent discovery of high thermopower coexisting with low electrical resistivity in layered cobaltites has opened the route to the exploration of thermoelectric materials in oxides [1-5]. Due to their high thermal stability, high tolerance to oxidation, and low toxicity, these oxides are promising candidates for practical use in thermoelectric power generation in air at high temperatures. The conversion efficiency of a thermoelectric material is evaluated by the dimensionless figure-of-merit $ZT=(S^2/\rho\kappa)T$, where S is the Seebeck coefficient, ρ is the electrical resistivity, κ is the thermal conductivity, and T is the absolute temperature. ZT values of single crystals of Na_xCoO_2 and $[\text{Ca}_2\text{CoO}_3]_p\text{CoO}_2$ (CCO) along the ab -plane exceed 1 at 1000 K [2, 4, 5],

the estimated criterion for practical application.

Some attempts to improve their thermoelectric efficiency by element partial-substitution have been reported [6-10]. Especially in the CCO system, it has been reported that the Bi-substitution for a part of the Ca site enhances its thermoelectric properties in sintered polycrystalline material [9]. Our recent investigation of the single-crystal CCO of mm size [11] indicated that the Bi-substitution had effect on the stability of the misfit CCO structure consisting of alternate stacking of a rocksalt-type $[\text{Ca}_2\text{CoO}_3]$ layer and a CdI_2 -type $[\text{CoO}_2]$ layer [12-14]. More to the point, the Bi-substitution can moderate the misfit relation of these two subsystems and eliminate stacking faults and defects, resulting in the enhancement of electrical conductivity without degradation of S . However, it is still unclear that this Bi-substitution effect on the crystallographic structure has an influence on the polycrystalline bulk material, which is indispensable for the practical application as it is difficult to grow single crystals large enough for the construction of a thermoelectric device.

In this study, partially Bi-substituted $[\text{Ca}_2\text{CoO}_3]_p\text{CoO}_2$ polycrystalline materials were synthesized and the effect of the Bi-substitution on the microstructure and thermoelectric properties evaluated. Due to the layered structure of the CCO phase, ρ along the c -axis is considerably higher than in the ab -plane [12]. Therefore, the samples were fabricated by means of hot forging technique in order to obtain the highly textured CCO bulk material. Since the grain size of the precursor CCO powder has significant implication for the grain alignment and ρ parallel to the pressed plane of the hot-forged sample [15], precursor powders of various sizes were used in order to investigate the effect of the microstructure on thermoelectric properties.

EXPERIMENTAL

The $[(\text{Ca}_{1-2x}\text{Bi}_{2x})_2(\text{Co}_{1-x}\text{Bi}_x)\text{O}_3]_p[\text{CoO}_2]$ ($x=0, 0.01, 0.02, 0.03$ and $0.04, p=0.62$ [16]) powders were prepared by solid-state reaction. Appropriate amounts of Co_3O_4 (99.9 %), CaCO_3 (99.5 %) and Bi_2O_3 (99.999 %) powders were mixed with an agate mortar. The mixture was calcined at 1073 K for 10 h in air, pulverized, and pressed into pellets, which were sintered at 1153 K for 20 h in an oxygen gas flow and ground into fine powder. In order to increase the grain size, the prepared powders were heated at 1123 K for 20 h in a solvent consisting of K_2CO_3 and KCl at a molar ratio of 4:1 [17]. The grains were grown at different weight ratios of solvent to CCO powder between 1wt% to 40wt%. The powder was collected from the solidified materials by dissolving the solvent in distilled water in an ultrasonic washing machine. The powders were cold-pressed into 20 thin-disks, which were finally stacked under uniaxial pressure of 190 MPa into a pellet of about 6 mm thickness and 20 mm diameter. The layered pellets were then hot-forged at 1153 K for 20 h in air under uniaxial pressure of 10 MPa. The obtained samples, with a diameter of about 22-25 mm and a thickness of about 3-4 mm, were cut into a bar shape

with a typical size of $3 \times 3 \times 15 \text{ mm}^3$ for measurement of thermoelectric properties.

Crystallographic structure and constituent analyses were performed with powder X-ray diffraction (XRD) using Cu $K\alpha$ radiation and an energy dispersive X-ray spectrometer (EDX), respectively. The microstructure was observed with a scanning electron microscope (SEM). The grain size of the CCO powders was measured using a HORIBA particle-size analyzer model LA-920. ρ was measured in air in the range of 300-1100 K using a conventional four-probe dc technique in the direction parallel to the pressed plane. S was calculated from a plot of thermoelectric voltage against temperature difference using an instrument designed by our laboratory. Two Pt-Pt/Rh (R-type) thermocouples were attached to both ends of the sample using silver paste and the Pt wires of the thermocouples used as voltage terminals. Measured S values were corrected by thermopower of the Pt wires to obtain the net S values of the samples.

RESULTS and DISCUSSION

The observation of the microstructure of prepared powders was conducted because our previous investigation has demonstrated that the size and the shape of the grains have an important effect on the grain alignment and the magnitude of ρ in the hot-forged CCO sample [15]. SEM images of the typical example of the prepared powders are shown in Fig. 1. Powders annealed in the mixture of K_2CO_3 and KCl consist of large and thick grains (Fig. 1(b) and 1(d)), while as-sintered CCO powders contain fine and thin particles (Fig. 1(a) and 1(c)). Thus, it is obvious that the annealing in the solvent is effective to grow CCO grains. The grains annealed in the solvent tend to form a platelet-shape derived from the layered crystallographic structure of CCO phase. Moreover, it can be seen that the grains of Bi-substituted CCO powder (Fig. 1(d)) are much larger than the non-substituted CCO (Fig. 1(b)) powder. Therefore, the grain size of CCO powder appeared to be significantly enhanced by the Bi-substitution.

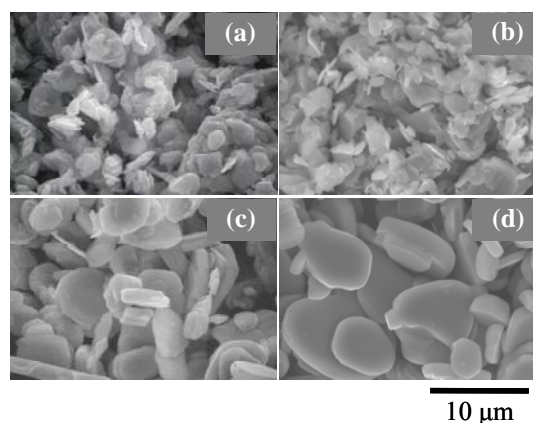


Fig. 1 SEM images of (a) as-sintered non and (b) Bi substituted CCO powder, (c) non and (d) Bi substituted powders heat-treated in a solvent mixture of K_2CO_3 and KCl with 10wt%.

The measurements of average grain size, represented in Fig. 2 and Fig. 3, confirmed this observation. As shown in Fig. 2, the average grain size increases with the increase of the amount of the solvent. This clearly proves the efficiency of solvent addition in improving the grain growth of the CCO phase. Since the solute of CCO was not entirely dissolved in the solvent even at 40wt%, the grains were grown by the recrystallization of the dissolved CCO on the residual solid particle. Therefore, the increase of the grain size is caused by the increase of the amount of the dissolved part of CCO powder. In addition, a small amount of Bi-substitution, about 1 mol% of sum total of Ca and Co cations, has a significant influence on the grain size.

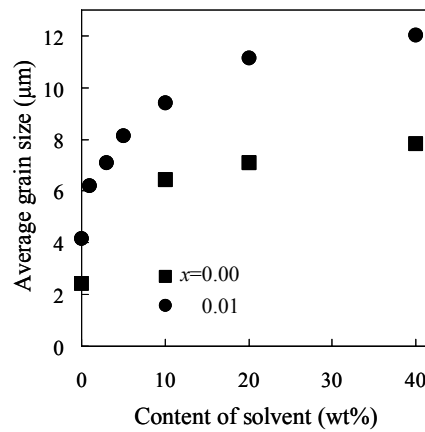


Fig. 2 Relationship between the weight ratio of solvent and average grain size of (■) CCO and (●) $[(Ca_{0.98}Bi_{0.02})_2(Co_{0.99}Bi_{0.01})O_3]_{0.62}[CoO_2]$ powders.

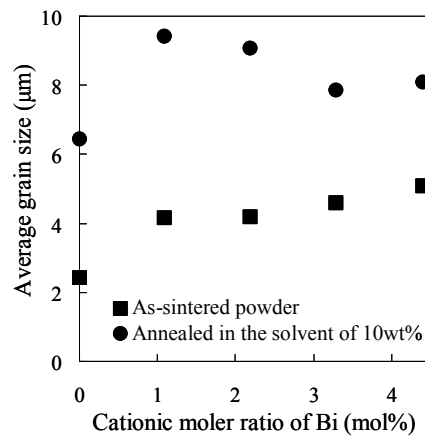


Fig. 3 Relationship between Bi composition and average grain size of $[(Ca_{1-2x}Bi_{2x})_2(Co_{1-x}Bi_x)O_3]_{0.62}[CoO_2]$ ($x=0, 0.01, 0.02, 0.03$ and 0.04) powders. (■) As-sintered and (●) annealed in a solvent mixture of K_2CO_3 and KCl with 10wt%.

The average grain sizes of CCO powder with the different Bi content were plotted in Fig. 3 in order to clarify the effect of the Bi-substitution on the grain growth. The grain size of as-sintered Bi substituted CCO powder is about twice as large as the non-substituted CCO and is increased with the increase of the Bi content. This result indicates that the Bi-addition can enhance the grain growth at the first solid-state reaction of raw powders. We propose that the liquid phase of Bi_2O_3 , which melts at around 1100 K, at the sintering temperature of 1153 K facilitates the chemical reaction and the grain growth. In addition, since it has been reported that the Bi-substitution affects the modulation of misfit structural CCO phase [16], the alteration of crystallographic structure might affect the growth rate. On the other hand, the grain size of the powders annealed in the solvent of 10wt% decreases with the increase of the Bi content, although their gain size is much larger than the non-substituted powder. The increment ratio of grain size decreases with the increase with the Bi content. This result means that the Bi-substitution interferes with the growth of CCO grains in the solvent. Therefore, the Bi-substitution might reduce the solubility and the amount of the dissolved part of CCO.

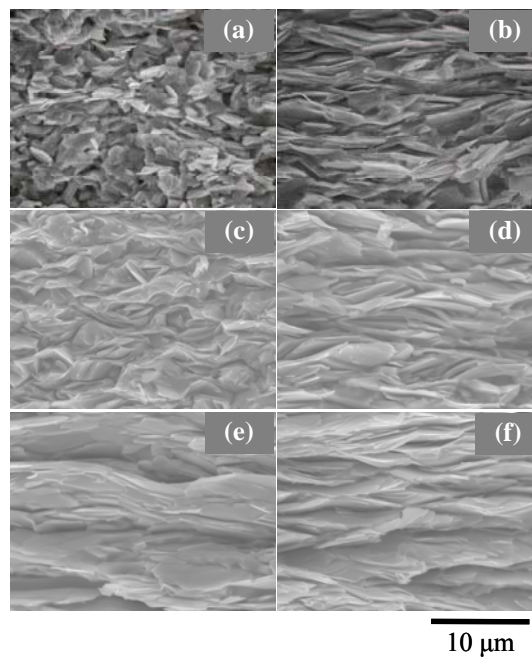


Fig. 4 SEM images of fracture transverse section of samples made from (a) as-sintered and (b) large grained CCO, (c) as-sintered and (d) large grained $[(\text{Ca}_{0.98}\text{Bi}_{0.02})_2(\text{Co}_{0.99}\text{Bi}_{0.01})\text{O}_3]_{0.62}[\text{CoO}_2]$, (e) as-sintered and (f) large grained $[(\text{Ca}_{0.92}\text{Bi}_{0.08})_2(\text{Co}_{0.96}\text{Bi}_{0.04})\text{O}_3]_{0.62}[\text{CoO}_2]$ powder.

XRD patterns of powders were in agreement with data from the literature on CCO phase [12]. There is no secondary phase. The diffraction peaks from the Bi-substituted samples tend to shift toward lower 2θ angle compared to the non-substituted one and the amount of the shift

increases with the increase of the Bi content. This tendency indicates that the crystallographic structure is enlarged by the Bi-substitution. This result is consistent with the reported result of the precise structural analysis of the Bi substituted CCO [16]. The EDX analysis performed on the powders heated in the solvent showed the grain growth treatment causes no change of the CCO composition and no incorporation of K^+ or Cl^- ions contained in the solvent. These results indicate that the present grain-growth method has no contamination effects on CCO powders.

SEM images of the typical example of the fracture transverse section of the hot-forged samples are shown in Fig. 4. It can be seen that samples made from large grained powders (Fig. 4(b), 4(d) and 4(f)) are constructed from well-oriented grains with a size of several μm . The alignment of the grains is obviously improved when compared to samples made from as-sintered powders (Fig. 4(a) and 4(c)), where small misalignment grains can be observed. The effect of the grain size of the precursor powder on the grain alignment of hot-forged bulk has been demonstrated by means of the quantitative texture analysis in our previous report [15]. The grain size of the Bi-substituted hot-forged sample is obviously larger than the non-substituted one resulting from the size difference of the precursor powder as mentioned above. Interestingly, the grains in the sample made from as-sintered powders with a nominal composition of $[(Ca_{0.92}Bi_{0.08})_2(Co_{0.96}Bi_{0.04})O_3]_{0.62}[CoO_2]$ (Fig. 4(e)) seem to be well connected and aligned when compared to the non-substituted sample, although we could not evaluate the connectivity of the grains from a quantitative point of view. This change of the microstructure by the Bi-substitution might affect the magnitude of ρ described below.

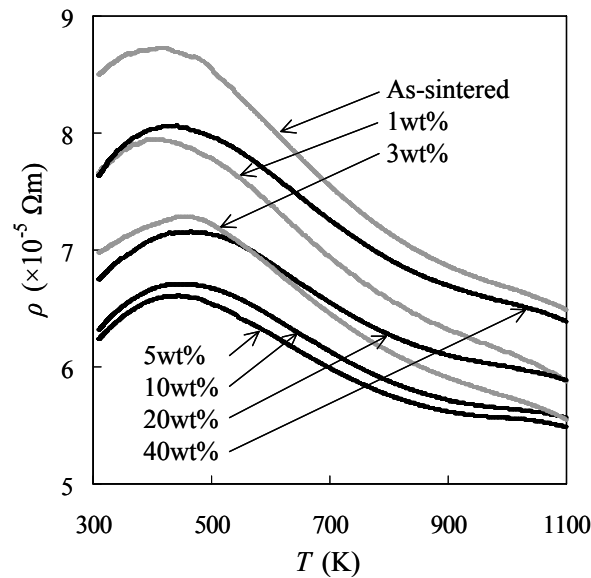


Fig. 5 T -dependence of ρ along the pressed plane of $[(Ca_{0.98}Bi_{0.02})_2(Co_{0.99}Bi_{0.01})O_3]_{0.62}[CoO_2]$ sample made from powders annealed in a mixture of K_2CO_3 and KCl with different weight ratio.

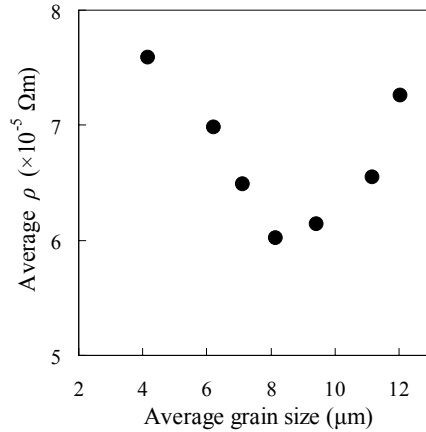


Fig. 6 Relationship between average ρ (from 373 K to 1073 K) and the average grain size of precursor powder of $[(\text{Ca}_{0.98}\text{Bi}_{0.02})_2(\text{Co}_{0.99}\text{Bi}_{0.01})\text{O}_3]_{0.62}[\text{CoO}_2]$.

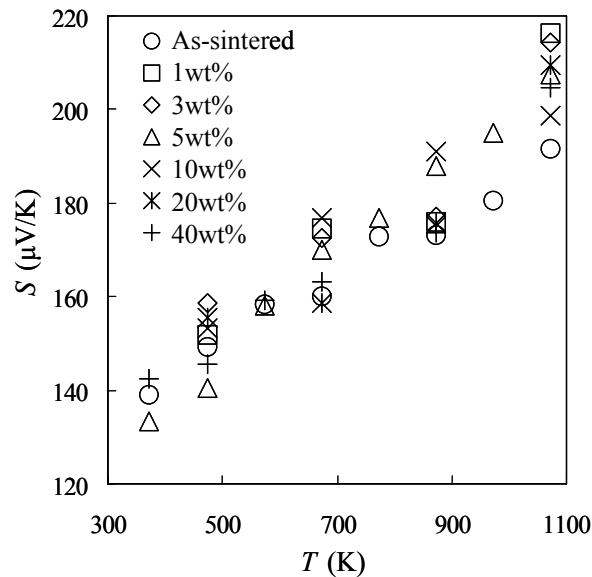


Fig. 7 T -dependence of S of $[(\text{Ca}_{0.98}\text{Bi}_{0.02})_2(\text{Co}_{0.99}\text{Bi}_{0.01})\text{O}_3]_{0.62}[\text{CoO}_2]$ sample made from powders annealed in a mixture of K_2CO_3 and KCl with different weight ratio.

In order to revalidate the influence of the grain size of precursor powder on the ρ value of the hot-forged sample [15], Bi-substituted samples were fabricated with the several powders of different grain sizes. The T -dependence of ρ along the direction parallel to the pressed plane of the samples with the nominal composition of $[(\text{Ca}_{0.98}\text{Bi}_{0.02})_2(\text{Co}_{0.99}\text{Bi}_{0.01})\text{O}_3]_{0.62}[\text{CoO}_2]$ is shown in Fig. 5. These samples were made from powders of different grain sizes as illustrated in Fig. 2. The inclination of ρ - T curve differs slightly between the sample made from powders heated in the solvent of 0wt% (as-sintered) to 3wt% and that in 5wt% to 40wt%, although the crystallographic

structure and composition of these samples are almost the same evaluating by XRD and EDX. We could not elucidate the origin of this difference with our present data. The relationship between the average grain size of the precursor powder and the average ρ value (from 373 K to 1073 K) is shown in Fig. 6. The clear correlation between the grain size of precursor powder and ρ value is observed. The ρ value reaches minimum at the grain size of 8 μm . The similar grain size dependence of ρ value was observed in non-substituted CCO and we have demonstrated that the origin of the reduction of ρ is derived from the improvement of the grain alignment [15]. We suppose that the same effect is obtained in Bi-substituted CCO sample by analogy. As shown in Fig. 7, S was less affected by the grain size, although there is some variation. The clear correlation between the grain size of precursor powder and S value is not observed. Thus, the power factor ($PF=S^2/\rho$) was improved by using precursor powder of around 8-9 μm .

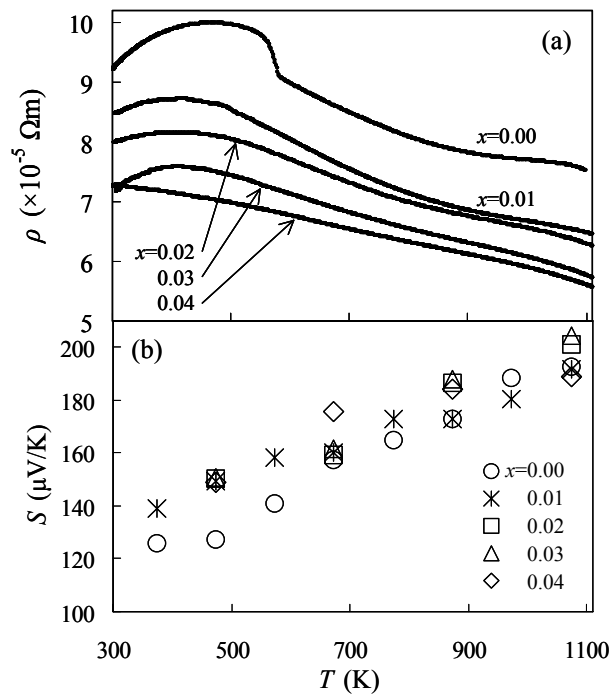


Fig. 8 T -dependence of (a) ρ and (b) S of hot-forged $[(\text{Ca}_{1-2x}\text{Bi}_{2x})_2(\text{Co}_{1-x}\text{Bi}_x)\text{O}_3]_{0.62}[\text{CoO}_2]$ ($x=0, 0.01, 0.02, 0.03$ and 0.04) sample made from as-sintered powders.

In order to examine the Bi-substitution effect, hot-forged samples with the different Bi content were synthesized and evaluated. The T -dependence of ρ along the direction parallel to the pressed plane of the samples made from as-sintered powder with the different Bi content is shown in Fig. 8(a). Above 600 K, ρ value in every sample decreases with the increase in T . On

the other hand, a broad maximum around 450 K and a steep decrease around 570 K is clearly observed in non-substituted CCO sample. However, in the Bi-substituted samples, the sharp decline at 570 K is not observed and the hump around 450 K is reduced as increase the Bi content. Although we could not elucidate the origin of these changes of the T -dependence of ρ within our present data, the Bi-substitution might have a significant influence on the electronic state of CCO phase. The magnitude of ρ is decreased with the increase in the Bi composition. The reduction of ρ is caused by the decrease of the number of grain boundaries derived from the increase of grain size as illustrated in Fig. 3 and in Fig. 4. We propose that the improvement of connectivity and alignment of grains also has influence on the ρ value as illustrated in Fig. 4. Since the Bi-substitution slightly increases the magnitude of S especially below 600 K (Fig. 8(b)), the PF was improved.

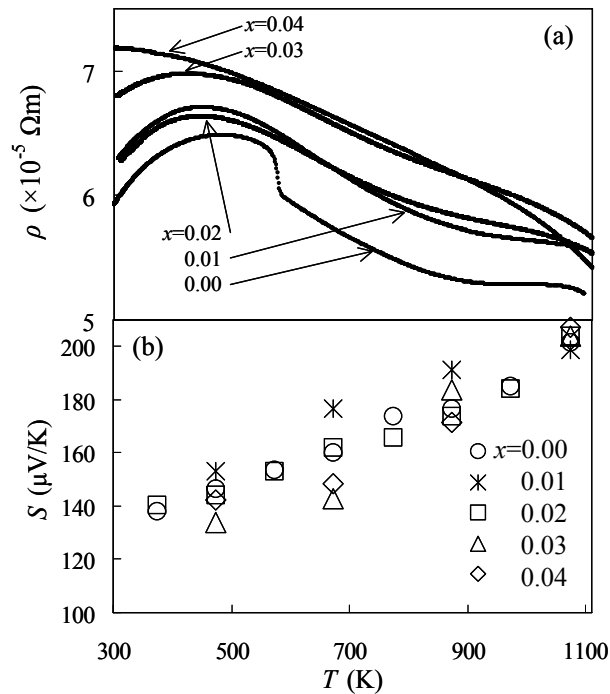


Fig. 9 T -dependence of (a) ρ and (b) S of hot-forged $[(\text{Ca}_{1-2x}\text{Bi}_{2x})_2(\text{Co}_{1-x}\text{Bi}_x)\text{O}_3]_{0.62}[\text{CoO}_2]$ ($x=0, 0.01, 0.02, 0.03$ and 0.04) samples made from powders annealed in the solvent mixture of K_2CO_3 and KCl with 10wt%.

As shown in Fig. 9(a), the effect of the Bi-substitution on the magnitude of ρ is completely changed when the large grained powders were used as a precursor. The ρ value is increased by the Bi-substitution despite the grain size is increased as mentioned above. This

result indicates that the Bi-substitution intrinsically increases the ρ value of CCO phase. Since it has been suggested that Bi^{3+} and Bi^{5+} substitute Ca^{2+} and Co^{3+} [16], the electrons would be injected. Thus, the Bi-substitution might reduce hole carrier concentration in the p-type CCO phase, resulting in the increase in ρ . However, a clear dependence of S on the Bi content was not observed as shown in Fig. 9(b). Additional measurements are required to elucidate the Bi-substitution effect on the electronic structure of CCO phase.

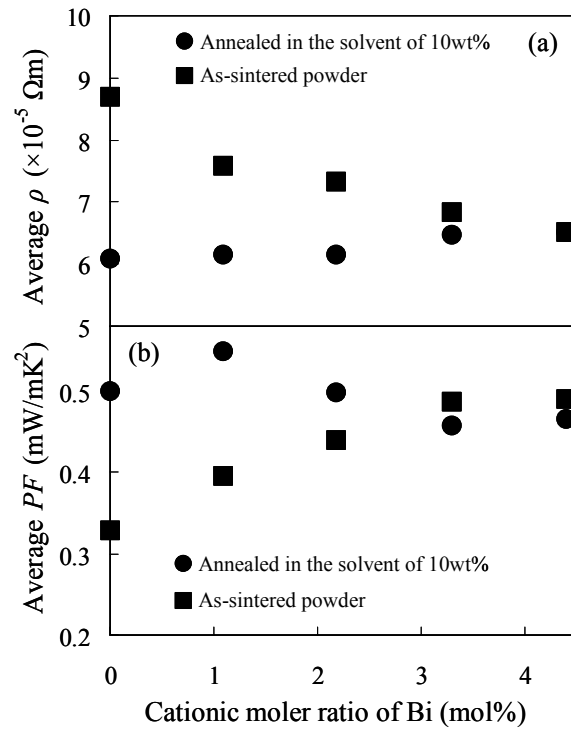


Fig. 10 The Bi-composition dependence of average ρ (from 373 K to 1073 K) and average PF (from 473 K to 1073 K). Hot-forged samples were made from (■) as-sintered and (●) large grained powders.

The relationship between the Bi content and the average ρ value (from 373 K to 1073 K) is shown in Fig. 10(a). This clearly shows the opposite influences of the Bi-substitution on the ρ value as mentioned above. Interestingly, ρ values of the sample made from the two kinds of precursor powder are almost the same at the nominal composition of $[(\text{Ca}_{0.92}\text{Bi}_{0.08})_2(\text{Co}_{0.96}\text{Bi}_{0.04})\text{O}_3]_{0.62}[\text{CoO}_2]$. This result indicates that the increase in grain size of precursor powder has little effect on the magnitude of ρ in the hot-forged sample. In other words, the grain size was sufficiently increased to fabricate the well-textured bulk at the first solid-state reaction of raw powders. This means that the Bi-substitution has almost the same contribution to the reduction of ρ as the increase in the grain size of the precursor powder, which has significant

influence on the ρ value in the non-substituted CCO phase. Since the Bi-substitution seems to intrinsically increase the ρ value of CCO phase as mentioned above, the reduction of ρ by the Bi-substitution in the sample made from as-sintered powder as illustrated in Fig. 8 might be caused by the change of the microstructure of hot-forged bulk, such as the reduction of the number of grain boundaries and the improvement of grain alignment. Since S value is less affected by the change of the microstructure, the Bi-substitution can contribute to the improvement of PF . The average PF value (from 473 K to 1073 K) was summarized in Fig. 10(b). This indicates that the PF of non-substituted CCO bulk can be enhanced by two kinds of methods. One is the increase of the grain size of the precursor powder. Second is the Bi-substitution enough to enhance the chemical reaction and the grain growth, which is about 4.5 mol% of sum total of Ca and Co cations in this study. Consequently, the development of microstructure is significantly effective to improve the PF of CCO bulk material.

CONCLUSION

The effect of the Bi-substitution on the electrical properties of hot-forged CCO bulk material was evaluated from the viewpoint of microstructure. The reduction of grain boundaries and the enhancement of grain alignment were achieved by using large grained powder as a precursor in the case of CCO and a small amount of Bi substituted CCO phase. The optimum grain size for the reduction of ρ was around 7-8 μm . On the other hand, the sufficient amount of Bi-substitution, which was 4.5 mol% of sum total of Ca and Co cations in this study, facilitates the grain growth during the sintering processes. The Bi-substitution effect on the microstructure has significant influence on the reduction of ρ , resulting in the improvement of PF in the hot-forged sample.

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