FULL PAPER

Growth and characterization of $\text{Ca}_{2-x}\text{Ae}_x\text{Al}_2\text{SiO}_7$ ($\text{Ae} = \text{Sr}, \text{Ba}$) piezoelectric single crystalsTakuto OSHIMA¹, Takuya HOSHINA¹, Takaaki TSURUMI¹,
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We report the effects of Sr or Ba substitution on the crystal growth and electrical and mechanical properties of $\text{Ca}_2\text{Al}_2\text{SiO}_7$ (CAS) piezoelectric single crystals, which are suitable for use in high-temperature pressure sensors. Ba was scarcely incorporated in the crystal lattice of CAS. Strontium-substituted CAS single crystal with a nominal chemical composition $\text{Ca}_{0.75}\text{Sr}_{1.25}\text{Al}_2\text{SiO}_7$ (CSAS125) was grown by the Czochralski method. Although the piezoelectric modulus d'_{31} of CSAS125 is a bit lower than that of the CAS crystal, the rupture strength of the (XY)45° rotation cut substrate of CSAS125 crystal was 170 MPa, which was 1.2 times higher than that of CAS crystal. By considering the rupture strength and the apparent piezoelectric d^*_{31} coefficient calculated for the crystal rotation cut of CAS crystal, the CSAS125 crystal substrate with a rotation cut of (XY)25° was found to indicate a rupture strength of 220 MPa and a piezoelectric d'_{31} coefficient of approximately 2 pC/N.

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Key-words : Silicate, Solid solution, Crystal growth, Piezoelectricity, Mechanical strength

[Received December 4, 2017; Accepted February 5, 2018]

1. Introduction

High-temperature piezoelectric materials are in high demand currently. Their potential applications include gas sensors, gas injectors, and combustion pressure sensors. Promising candidates are langasite $\text{La}_3\text{Ga}_5\text{SiO}_{14}$ (LGS)-type crystals,^{(1)–(5)} rare-earth calcium oxoborate $\text{ReCa}_4\text{O}(\text{BO}_3)_3$ (Re = rare earth elements, ReCOB) crystals,^{(6),(7)} gallium phosphate GaPO_4 ,^{(8),(9)} and AlN and its modified thick films,^{(10),(11)} which have been intensively investigated. In fact, these materials have one or more weaknesses in terms of actual application, as mentioned in Ref. 12). Under this situation, melilite group crystals such as $\text{Ca}_2\text{Al}_2\text{SiO}_7$ (CAS)^{(12),(13)} and $\text{SrLaGa}_3\text{O}_7$ ⁽¹⁴⁾ are attracting attention. Among them, the CAS single crystal exhibits both stable resistivity at high temperatures and good piezoelectric properties. Melilite crystals are characterized by a distinct cleavage plane, however, which is normal to the crystallographic c -axis, that is, corresponding to the crystallographic {001} plane. This cleavage plane may cause low mechanical strength and thus narrow the range of the crystals' potential target applications (including combus-

tion pressure sensors). In our previous report, a successful improvement of mechanical strength was demonstrated by Sr substitution to CAS single crystal.⁽¹⁵⁾ Based on the crystal structure analysis, it was considered that this improvement was caused by relaxation of the distorted Ca decahedrons of the CAS lattice by replacement of Ca^{2+} ($r^{\text{VIII}} = 1.12 \text{ \AA}$)⁽¹⁶⁾ with Sr^{2+} ($1.26 \text{ \AA}$). This report gave us some hints for improving the mechanical strength of CAS crystal. One simple way is the possible substitution of a larger alkali-earth cation, Ba^{2+} ($1.42 \text{ \AA}$) rather than Sr^{2+} for Ca^{2+} . Another way is to increase the Sr content incorporated into the CAS crystal lattice. In Ref. 15), the composition of the Sr-substituted CAS crystal was $\text{CaSrAl}_2\text{SiO}_7$. Hence, increasing the Sr content in the starting materials for crystal growth can be assumed to be a reasonable solution.

In this study, we report the effect of Sr and Ba substitution on the crystal growth and electrical and mechanical properties of CAS piezoelectric single crystals. The solid solubility of Ba and Sr in the Ca-site of CAS grown by the Czochralski method is addressed. Taken into account with potential pressure sensor applications, a crystal orientation cut with both higher mechanical strength and sufficient piezoelectric coefficient is proposed and demonstrated for CAS crystals.

2. Experimental

Sr- and Ba-substituted CAS fiber crystals were grown via a laser-heated pedestal growth (LHPG) method.⁽¹⁷⁾ For the chemical composition, in which the crystals grown by

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†† Preface for this article: DOI <http://doi.org/10.2109/jcersj2.126.P5-1>

††† Publication of this international collaborative article is supported by JSPS Grants-in-Aid for Scientific Research (KAKENHI), Grant Number 252016

the LHPG method had no impurity phase or small amount of secondary phases, we applied the Czochralski (Cz) method as the bulk crystal growth technique. Since several samples can be grown by the LHPG method in one day, it allows rapid production of a large variety of materials and determination of their optimum compositions. A detailed description of the LHPG method is seen in Ref. 17). The growth speed and atmosphere were 0.3 mm/min and air, respectively. Ceramic fibers were used for the seed and feed. A ceramic system with the chemical formula $\text{Ca}_{2-x}\text{Sr}_x\text{Al}_2\text{SiO}_7$ ($x = 0.00\text{--}2.00$: CSAS100 x) and $\text{Ca}_{2-x}\text{Ba}_x\text{Al}_2\text{SiO}_7$ ($x = 0.00\text{--}0.20$: CBAS100 x) was synthesized using a conventional solid-state ceramic fabrication technique. 99.99% pure CaCO_3 , SrCO_3 , BaCO_3 , Al_2O_3 , and SiO_2 powders were used as the starting materials. Stoichiometric amounts of oxides and carbonates corresponding to CSAS100 x and CBAS100 x were weighed, mixed with ethanol, and dried and calcined at 1350–1390°C for 4 h. The calcined powders were enclosed in rubber tubes and pressed under hydrostatic conditions, and then formed into a cylindrical shape with a 6 mm diameter and 40–50 mm length. The resulting rods were sintered at 1420–1500°C for 4 h in air and cut into the form of a rectangular parallelepipeds with dimensions of $1.0 \times 1.0 \times 30\text{--}40 \text{ mm}^3$. The fiber rods were used as charge and seed rods for the LHPG method. The as-grown crystals were polished, and their chemical composition was then determined by electron probe micro analyzer (EPMA).

The obtained LHPG data were used in an attempt to grow CSAS100 x and CBAS100 x single crystals subsequently by a conventional radio frequency heating Cz method inside an Ir crucible with a diameter and height equal to 50 mm. The raw materials were the same powders as those used for the LHPG method. They were mixed in air, calcined at 1350°C, and then charged into the crucible. The growth was conducted under an Ar gas flow at a rate of $10^{-3} \text{ m}^3/\text{min}$. The pulling rate and rotation rate were 1.0 mm/h and 10 rpm, respectively. Phase identification of the as-grown crystals was performed by powder X-ray diffraction (XRD), while their density was measured by the Archimedes method using distilled water at room temperature (23°C). A part of the grown crystals was pulverized and dissolved in hydrochloric acid. The solutions were used for bulk composition analysis by inductive coupled plasma-atomic emission spectrometry (ICP-AES).

As mentioned later, CSAS125 crystals with the nominal chemical composition $\text{Ca}_{0.75}\text{Sr}_{1.25}\text{Al}_2\text{SiO}_7$ were successfully grown by the Cz method. These CSAS125 crystals were used for further investigation. CAS-based crystal belongs to the tetragonal space group $P4_21m$, therefore, possess two independent piezoelectric moduli, d_{14} and d_{36} . This study is focused on the apparent piezoelectric coefficient d'_{31} expressed by the equation $d'_{31} = d_{14} \sin 2\theta/2$ using the piezoelectric modulus d_{14} and the rotation angle θ , because d_{14} values are higher than d_{36} values and are also discussed in the previous report.¹⁵⁾ The d'_{31} coefficients were evaluated by measuring resonant and anti-resonant frequencies of the resonators in the length-

extensional mode. The relative permittivity $\epsilon_{11}^T/\epsilon_0$ and apparent elastic compliance s'_{11}^E were estimated according to the IEEE standard¹⁸⁾ with the magnitude of the apparent d'_{31} coefficients taken into account. The dielectric coefficients ϵ_{11}^T were determined by measuring the resonator capacitances (corrected for the parasitic capacitance). Sample electrodes were produced by sputtering Au metal on the main surfaces. These coefficients of the CSAS125 crystal were compared to those of the CAS crystal.

Compression tests were conducted in the same way as we reported in Ref. 15) to determine the rupture strength of the crystal substrates by using a texture analyzer (TA.XT.PLUS). The sample size and the load speed in this study were different, however, from those in Ref. 15). In this study, they were set according to JIS R1608.¹⁹⁾ Several samples were fabricated in the form of rectangular parallelepipeds with dimensions of $1 \times 1 \times 2.5 \text{ mm}^3$ and a $(XY)\theta$ cut. The load speed was 1 mm/min. The rupture strength of the CSAS125 crystal substrates was also compared to those of the CAS substrates.

3. Results and discussion

As the starting materials for the LHPG method, the prepared chemical compositions were $\text{Ca}_{2-x}\text{Sr}_x\text{Al}_2\text{SiO}_7$ (CSAS100 x) with $x = 0.50, 1.00, 1.25, 1.50, 1.75, 2.00$, and $\text{Ca}_{2-x}\text{Ba}_x\text{Al}_2\text{SiO}_7$ (CBAS100 x) with $x = 0.01, 0.05$. **Figure 1** shows some of the as-grown CSAS100 x and CBAS100 x crystals obtained by the LHPG method. All the crystals were colorless and almost transparent. These crystals were polished and investigated by EPMA to obtain detailed internal observations. **Figure 2** shows reflected electron images of the polished surfaces of the CSAS125, CSAS175, and CBAS5 crystals grown by the LHPG method. There is no secondary phase in the CSAS125 crystal, as shown in Fig. 2(a). Furthermore, it was also found that the CSAS100 x crystals grown with $x \leq 1.50$ do not contain any inclusions. On the other hand, the CSAS175 and CBAS5 crystals have many inclusions,

Fig. 1. Photographs of $\text{Ca}_{2-x}\text{Sr}_x\text{Al}_2\text{SiO}_7$ (CSAS100 x) and $\text{Ca}_{2-x}\text{Ba}_x\text{Al}_2\text{SiO}_7$ (CBAS100 x) crystals grown by the LHPG method.

Fig. 2. Reflected electron images of polished surfaces of (a) CSAS125, (b) CSAS175, and (c) CBAS5 crystals grown by the LHPG method.

which were found to be SrAl_2O_4 [dark lines in Fig. 2(b)] and $(\text{Ba},\text{Ca})(\text{Al},\text{Si})_4\text{O}_8$ phases [bright areas in Fig. 2(c)], respectively. In particular, a very low amount of Ba on the order of <500 ppm is included in the dark area corresponding to the gehlenite phase. These observations suggest that $\text{Sr}_2\text{Al}_2\text{SiO}_7$ ²⁰⁾ melted incongruently and that Ba atoms were hardly incorporated into the gehlenite crystal lattice. Since the growth of bulk CSAS100 crystals ($\text{CaSrAl}_2\text{SiO}_7$) was already reported,¹⁵⁾ therefore, we started to grow CSAS150 crystal by the Cz method in this study. For CBAS100x crystal, we also attempted to grow CBAS1 crystal.

Figure 3 shows as-grown CSAS100x and CBAS100x crystals fabricated by the Cz technique. CSAS150 crystal is completely opaque as shown in Fig. 3(a), while CBAS1 crystal is slightly opaque as shown in Fig. 3(b). Many inclusions were observed in both the crystals. In a similarity to the LHPG growth results mentioned in the previous paragraph, SrAl_2O_4 and $(\text{Ba},\text{Ca})(\text{Al},\text{Si})_4\text{O}_8$ phases were detected by EPMA in the CSAS150 and CBAS1 crystals, respectively. It was confirmed that Ba atoms were scarcely incorporated in the gehlenite crystal lattice. It was

Fig. 3. Photographs of side views of (a) CSAS150, (b) CBAS1 and (c) CSAS125 crystals grown by the Cz technique.

reported that single crystals with a high concentration of dopant can be grown by the LHPG method as compared to the Cz method.¹⁷⁾ After the Sr content in the raw materials with the composition of CSAS100x was reduced, therefore, CSAS125 single crystals with no inclusions were successfully grown by the Cz method, as shown in Fig. 3(c). The CSAS125 crystals grown in this study were transparent and colorless. The size was 30 mm in maximum width, 60 mm in length, and 10 mm in thickness. The obtained boule contained two developed flat surfaces consisting of planes parallel to the $\langle 100 \rangle$ growth direction. Backscattering Laue X-ray analysis showed that the flat surfaces corresponded to $\{001\}$ planes. Large cracks can be seen in Fig. 3(c). This was due to intentional increments of the volume of the crystal. All the peaks of the crystal powder XRD patterns agreed with those of the melilite-type structure. We carefully eliminated these macro defects via proper sample preparation, such as cutting and polishing. The solidification fraction, $g = W_{\text{crystal}}/W_{\text{initial}}$, where W_{crystal} and W_{initial} are the weight of the grown crystal and the starting melt, respectively, was 0.30. The Sr/Ca ratio of the CSAS125 crystal was 1.64, which was in very good agreement with that for the starting raw materials of 1.67, taking of the accuracy of ICP-AES analysis conducted in this study into account. This suggested that the chemical composition of the CSAS125 crystal is close to the congruent melt composition, therefore, further bulk crystal growth is in progress.

Electromechanical properties were characterized using the $(XYI)\theta$ substrates of the CAS and CSAS125 crystals. **Table 1** lists electromechanical coupling factors, and elastic, piezoelectric, and dielectric coefficients for the

Table 1. Electromechanical coupling factors, and piezoelectric, dielectric and elastic coefficients for CAS and CSAS125 crystals measured at 23°C

Composition (Sample)		Ca ₂ Al ₂ SiO ₇ (CAS)	Ca _{0.75} Sr _{1.25} Al ₂ SiO ₇ (CSAS125)	
Substrate		(XYt)45°	(XYt)45°	(XYt)25°
Electromechanical coupling factors	k'_{31}	0.12	0.098	0.074
Elastic coefficient [10 ⁻¹² m ₂ /N]	s'_{11}^E	9.36	8.35	7.79
Piezoelectric coefficient [pC/N]	d'_{31}	3.59	2.55	1.93
Relative dielectric coefficient	$\varepsilon_{11}^T/\varepsilon_0$	10.6	9.1	9.91
	[-]

Fig. 4. Stress-strain curves for the (XYt)45° substrates of CAS and CSAS125 single crystals.

(XYt)45° substrates of the CAS and CSAS125 crystals measured at room temperature. The magnitude of the apparent piezoelectric d'_{31} coefficient for CSAS125 was lower than that for CAS. Reduction by Sr substitution was also reported.¹⁵⁾ The reason for this is under consideration and will be ascertained by combining detailed crystal structure analysis and characterization of all the electro-mechanical coefficients. The apparent d'_{31} coefficient was still higher than the d_{11} values for quartz (2.30 pC/N²¹⁾, which has been used as a combustion sensor on a laboratory scale for practical applications with a large-scale cooling water system attached. The elastic compliance magnitude s'_{11}^E was lower for CSAS125 than for CAS, indicating that the CAS crystal lattice was hardened due to Sr substitution. This observation differs from that reported in Ref. 15). The reason for this is also under investigation using the plane-wave ultrasonic material characterization system.^{22,23)} The results will appear elsewhere.

Figure 4 shows the stress-strain curves for the (XYt)45° substrates of the CAS and CSAS125 single crystals. The rupture strength of the CSAS125 crystal was 170 MPa, which was 1.2 times higher than that 140 MPa of the CAS crystal. In the previous report,¹⁵⁾ we observed that the rupture strength of the CAS crystal was 190 MPa. The difference between the results of this study and the previous report was due to the sample size and load speed.

The load speed adopted in Ref. 15) was 6.0 mm/min, which is six times higher than the 1.0 mm/min used in this study, according to JIS R1608.¹⁹⁾ Since it is well known that higher load speeds produce higher rupture strength, it is meaningless to discuss the difference. Considering the safety requirements of combustion pressure sensors, higher rupture strength is more suitable, and we therefore attempted to find a substrate cut orientation showing both higher rupture strength and an apparent d'_{31} coefficient of 2.0 pC/N, which is comparable to the piezoelectric d_{12} constant of quartz.²¹⁾

There is a trade-off relationship between rupture strength and the apparent piezoelectric d'_{31} coefficient in the (XYt)θ cut of CAS crystals. This is because the cleavage eventually becomes the origin of fracture. A share stress T with respect to the cleavage plane {001} is calculated using the following equation:

$$T = \frac{F}{2S} \sin 2\theta \quad (1)$$

where F , θ and S correspond to the applied force, the rotation angle and the area of the substrate sample normal to the applied force direction, respectively. It is necessary to decrease the T value to attain higher rupture strength. The apparent piezoelectric coefficient d'_{31} is expressed by the equation $d'_{31} = d_{14} \sin 2\theta/2$ using the piezoelectric modulus d_{14} and the rotation angle θ . Both the T and d'_{31} values are the maximum at $\theta = 45^\circ$ and both decrease when θ separates from 45° . In this study, we calculated the apparent d^*_{31} coefficient appearing in triple-rotated coordinates based on the Euler angles.²⁴⁾ Considering the symmetry $\bar{4}2m$ of the CAS crystal based on Refs. 18), 25), 26), after the rotation of angle α around the z -axis in the original coordinate (x, y, z), followed by the rotation of angle β around the x' -axis in the generated coordinate (x', y', z'), and, finally, by the rotation of angle γ around the z'' -axis in the second generated coordinate (x'', y'', z''), the apparent piezoelectric d^*_{31} coefficient in the new coordinate can be calculated using the following formula:

$$\begin{aligned} d^*_{31} = & d_{14} \sin^2 \beta \sin \gamma [2 \sin \alpha \cos \alpha \cos \beta \sin \gamma \\ & + \cos \gamma (\sin^2 \alpha - \cos^2 \alpha)] \\ & + d_{36} \cos \beta [\sin \alpha \cos \alpha (\cos^2 \gamma - \cos^2 \beta \sin^2 \gamma) \\ & + \cos \beta \cos \gamma (\cos^2 \alpha \sin \gamma - \sin^2 \alpha \sin \gamma)] \end{aligned} \quad (2)$$

where d_{36} is another piezoelectric constant. **Fig. 5(a)** shows the orientation dependence of the piezoelectric coefficient, where the highest d^*_{31} value is equal to the apparent d'_{31} coefficient obtained in the (XYt)45° cut. This is because both d_{14} and d_{36} are positive^{27,28)} and the d_{14} value is seven times higher than the d_{36} value.²⁹⁾ This Eq. (2) should be useful for estimating the high apparent d'_{31} coefficient when new melilite-type crystals are grown, and their electromechanical coefficients are characterized. To find the substrate cut orientation showing both higher rupture strength and an apparent d'_{31} coefficient of 2.0 pC/N, the (XYt)25° cut was selected in this study. **Figure 5(b)** illustrates samples of the (XYt)45° cut and the (XYt)25°

Fig. 5. The piezoelectric d^*_{31} coefficient as a function of orientation for CSAS125 crystal.

Fig. 6. Stress-strain curves for the $(XYt)45^\circ$ - and $(XYt)25^\circ$ -cut substrates of CSAS125 single crystals.

cut. The electromechanical coefficients of the $(XYt)25^\circ$ cut substrate of CSAS125 crystal are also listed in Table 1. As expected, the apparent d^*_{31} coefficient of the $(XYt)25^\circ$ cut substrate is close to 2.0 pC/N.

Figure 6 shows the stress-strain curves for the $(XYt)25^\circ$ and $(XYt)45^\circ$ substrates of the CSAS125 single crystals. The rupture strength of the $(XYt)25^\circ$ substrate was 220 MPa, which was approximately 1.3 times higher than the 170 MPa of the $(XYt)45^\circ$ substrate. A comparison of the stress-strain curves before fracture reveals that the $(XYt)45^\circ$ substrate exhibits a linear inclination, but the $(XYt)25^\circ$ substrate shows two different inclinations. The reason for this is under investigation, and we will measure the rupture

strength of quartz, because only the 130–170 MPa tensile strength of AT-cut quartz³⁰⁾ has been reported as far as our research could discover. The rupture strength of the $(XYt)25^\circ$ substrate of the CSAS125 crystal is approximately 1.6 times higher than that of the $(XYt)45^\circ$ substrate of the CAS crystal. The obtained results, therefore, suggest that CSAS125 crystal can be a candidate material for manufacture of high-temperature pressure sensors.

4. Summary

Crystal growth of Sr- and Ba-substituted CAS, $\text{Ca}_{2-x}\text{Sr}_x\text{Al}_2\text{SiO}_7$ and $\text{Ca}_{2-x}\text{Ba}_x\text{Al}_2\text{SiO}_7$ was conducted using the LHPG and Cz methods. $\text{Ca}_{0.75}\text{Sr}_{1.25}\text{Al}_2\text{SiO}_7$ (CSAS125) single crystal with no inclusions was successfully grown. Crystal grown from a melt with the $\text{Ca}_{1.99}\text{Ba}_{0.01}\text{Al}_2\text{O}_7$ composition, meanwhile, included secondary phases throughout the interior, which prevented further sample characterization. The electromechanical coefficients of the CSAS125 crystal were slightly smaller than those of the CAS crystal, but the value were sufficient for application. The mechanical strength of CAS increased by a factor of about 1.6 after both Sr substitution and optimization of the crystal substrate orientation. These processes succeeded in overcoming the weakness of the CAS crystals, making CSAS125 an attractive candidate for pressure sensor applications at elevated temperatures.

Acknowledgements The authors wish to thank Associate Professor T. Ikoma of Tokyo Institute of Technology for his helpful suggestions regarding the mechanical measurements. They also thank Dr. M. Tada of the Center of Advanced Materials Analysis at the Tokyo Institute of Technology for EPMA analysis. A part of this study was financially supported by the Grants-in-Aid for Scientific Research program of the Japan Society for the Promotion of Science (JSPS KAKENHI, grant number 15KK0201, 16H04214), and the Kumagai research grant, Murata Science Foundation.

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