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EVALUATION OF FERROELASTIC MECHANICAL BEHAVIOR OF LANTHANUM GALLATE, LaGaO₃

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Abstract-The mechanical deformation behavior of lanthanum gallate $(LaGaO_3)$ was investigated under uniaxial compression. XRD patterns showed the characteristics reflection of orthorhombic crystal structure. Ferroelastic domains were observed in sample as a striped pattern. Uniaxial compression tests were carried out at temperature of 93K, 193K, 293K, 393K, and 553K, at a constant loading rate of 1.0 MPa/s. LaGaO₃ shows non-elastic stress-strain curve related to its ferroelastic behavior. The initial modulus and critical stress were decreased with increasing the temperature. Additionally thermo-mechanical analysis was performed to measure the thermal expansion co-efficient of prepared sample.

Keywords: Mechanical behavior, lanthanum gallate, ferroelasticity.

1. INTRODUCTION

Lanthanum based perovskite-related materials are of research interest because of their wide variety of applications in advanced technology such as solid oxide fuel cells (SOFC), electrolyzers, oxygen pumps and oxygen sensors [1-4]. Higher oxide ionic conductivity and lower electronic conductivity are important as an electrolyte in these devices. Yttria-stabilized zirconia (YSZ) is generally used as an electrolyte. However at temperature lower than 800° C, resistivity of YSZ is a great problem which is considered as operating temperature of intermediate temperature of SOCF. In recent years, a new ionic conductor has been developed which shows higher oxygen ionic conductivity than zirconia. The new materials are considered as perovskites based on lanthanum gallate (LaGaO₃) [5-11] Several researcher reports about the investigation of ionic conductivity of doped lanthanum gallate [12-14], but a very few number research work on mechanical behavior of lanthanum gallate has been reported [15-17]. The present study investigates the ferroelastic mechanical behavior of LaGaO3 under different temperatures. Additionally thermo-mechanical analysis carried out to calculate the thermal expansion coefficient.

2. EXPERIMENTAL

LaGaO₃ (LGO) samples were prepared using the method of solid state reaction. Starting powder La_2O_3 (99.99%) and Ga_2O_3 (99.99%) were mixed for 48 hours in a ball mill mixing with ethanol. The mixed powders were then dried at 423 K for 1 hr. Obtaining powder was calcined at 1223K for 5 hr. The calcined mixture was ground and pressed uniaxially into circular discs and sintered at 1573K for 10 hrs. Low speed diamond saw was used to cut the sintered discs into rectangular shape

specimen of $3.0 \times 3.0 \times 15 \text{ mm}^3$. The cut specimens were annealed at 1273K for 1 hr to remove residual stresses. Fig.1 shows the calcining, sintering and annealing conditions.



Fig. 1: Calcining, sintering and annealing temperatures of LGO

For SEM observation annealed samples were embedded in moulding resin and one side of the sample was polished with SiC sandpaper, diamond paste of 3 µm and 1 µm and finally colloidal silica (~80nm) was used using (IM-P2, IMT, Japan) polishing machine. A mirror polished sample was examined under scanning electron microscope (JSM-5600, JEOL, Japan) to observe the © ICMERE2017 ferroelastic domain. In order to observe the crystal structure sintered samples were analyzed by a X-ray diffraction (XRD) system (XRD-6100, Shimadzu, Japan). The CuK α radiation was used. The scanning was performed at a rate of 1°/min at a 2 θ range between 20° to 80° operates under the current of 30mA and a voltage of 40kV. For thermal expansion co-efficient measurement of prepared samples, S-II EXSTAR-6000, Seiko Instrument, Japan was used. During experiment heating of 2°C/min was used.

To evaluate the mechanical behavior, uniaxial compression tests were performed using a universal material testing machine (AGS-X, Shimadzu, Japan). A dynamic strain meter (DC-204R, Tokyo Sokki, Japan) and strain gauges (FLA for room temperature, CEFLA for low temperature and ZFLK for high temperature, Tokyo Sokki, Japan) were used. To avoid the bending effects, two strain gauges were used on opposite faces of the specimen. Compressive stress up to 100 MPa was applied at a loading rate 1.0 MPa/s. For temperature dependence experiment, the tests were performed at 93K, 193K, 293K, 393K, and 553K.

3. EXPERIMENTAL RESULTS AND DISCUSSION

Fig. 2 shows the scanning electron micrographs of polished sample. In Fig. 2 ferroelastic domain was observed as striped pattern inside circular mark region. XRD patterns of LaGaO₃ sample prepared in this study with reference data [18] is shown in Fig.3. XRD result shows the characteristics reflection of orthorhombic crystal structure. Fig. 4 shows the relationship between deformation and thermal expansion co-efficient with temperature. It is found that deformation start to decrease at a temperature of 152°C, as a result the thermal expansion coefficient decrease suddenly. This is due to because of phase transition temperature from orthorhombic to rhombohedral structure of LGO is 145°C [19, 20]. The average value of thermal expansion coefficient is 7.5×10^{-6} °C⁻¹ before and after the phase transition where as H. Morkog [21] reported the value of LGO is $8.5 \times 10^{-6} \text{ °C}^{-1}$.



Fig. 2: Scanning electron microscope image of polished LGO sample.



Fig. 3: XRD patterns of LGO sample with reference data.



Fig. 4: Deformation and thermal expansion coefficient of LGO.

The cyclic stress-strain behavior is shown in Fig. 5 at different temperatures. The SS curve shows a distinctive nonlinear characteristic which is a typical ferroelastic material behavior. This is similar to ferroelastic behavior of cobaltite lanthanum perovskites such as. [22], La-Sr-Co-O [23]. Basically La-Sr-Co-Fe-O ferroelastic material shows non-elastic stress-strain behavior which was observed by uniaxial mechanical testing [24]. At the very beginning of loading cycle, stress increases linearly with strain and then stress plateau region (referred as critical stress) was observed due to domain switching phenomenon. The stress starts increasing after finishing the domain switching process. During unloading, also nonlinearity observed due to back-switching which results hysteresis loop. It is shown in Fig. 5 that with increasing the temperature, the slope of the curve is falling down which shows the ferroelastic behavior clearly.



Fig. 5: SS curve of LGO at different temperatures

Fig.6 shows the relationships between initial modulus and loading modulus to temperature, which are derived from Fig.5 according to the previous study [25]. It is shown that the initial modulus and loading modulus monotonically decreases with temperature, which indicates that at high temperature number of switchable domain start to increase.



Fig. 6: Ferroelastic parameters of LGO

4. CONCLUSION

In the present investigation, uniaxial compression test of LGO was performed over a temperature range of 93K to 553K in order to observe the ferroelastic characteristics. The stress-strain behavior under uniaxial compression exhibited nonlinear characteristics due to ferroelastic domain switching. The initial modulus and loading modulus decreased monotonically with increasing temperature. Measured thermal expansion coefficient of prepared sample was 7.5×10^{-6} °C⁻¹ before and after the phase transition from orthorhombic to rhombohedral phase.

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