Journal of the European Ceramic Society xxx (2016) xxx–xxx

Contents lists available at www.sciencedirect.com

Journal of the European Ceramic Society

journal homepage: www.elsevier.com/locate/jeurceramsoc

Full Length Article

Mechanical behavior of ferroelastic $LaAlO_3$

M.N. Islam[a,b], W. Araki[b,*], Y. Arai[b]

[a] Department of Mechanical Engineering, Rajshahi University of Engineering & Technology, Rajshahi, 6204, Bangladesh
[b] Department of Mechanical Engineering, Saitama University, 255 Shimo-okubo, Sakura-ku, Saitama 338-8570, Japan

ARTICLE INFO

Article history:
Received 21 October 2016
Received in revised form
21 November 2016
Accepted 22 November 2016
Available online xxx

Keywords:
Mechanical behavior
Lanthanum aluminate
Ferroelasticity
Domain switching
Stress-strain relationship

ABSTRACT

The present study investigates the mechanical deformation response of lanthanum aluminate ($LaAlO_3$) under various loading rates and temperatures. Ferroelastic domains were observed in samples of different porosity. Uniaxial compression tests were performed at room temperature and different loading rates ranging from 0.03 to 5.75 MPa/s. Temperature variation experiments were performed at 93 K, 193 K, 293 K, 393 K, and 553 K. $LaAlO_3$ shows non-elastic stress-strain behavior in which hysteresis loops are observed during loading–unloading cycles owing to ferroelasticity. The slope of the stress-strain curve became steeper with increasing loading rate and temperature. After unloading, remnant strain was stored in the material owing to ferroelastic domain switching.

1. Introduction

Lanthanum-based perovskite-related materials are of research interest owing to their wide applications in advanced technologies such as solid oxide fuel cells (SOFCs). A large amount of research work on the electrochemical characterization and ionic conductivity of lanthanum oxides, such as doped $LaCoO_3$, $LaAlO_3$ and $LaGaO_3$ have been carried out aiming at their possible use as cathodes and electrolyte materials for SOFCs [1–5].

Higher oxygen-ion conductivity is essential for solid electrolytes in high temperature electrochemical devices. Several studies have been conducted on processes for enhancing the conductivity of these materials [6–8]. Manganese (Mg)-doped $LaGaO_3$ possesses higher conductivity than typical zirconia-based materials [9–13]. However, owing to their high cost and low mechanical stability at high temperature [14], $LaGaO_3$-based perovskites are not yet applicable despite their higher oxygen ionic conductivity. To avoid the high cost of Ga-based compounds, $LaAlO_3$-based perovskites may be a more cost effective solid electrolyte candidate [7,8,15,16].

The few studies that have been done on the mechanical behavior of lanthanum-based metal oxides have reported atypical mechanical behavior [17–21]. This unusual behavior can be attributed to ferroelasticity [22]. Lanthanum cobaltite-based perovskites, such as $LaCoO_3$ (LCO), La-Sr-Co-Fe-O (LSCF), and La-Sr-Co-O (LSCO) [23–26], are well known to show ferroelastic characteristics, which are ferroic effects found in mechanical behavior [22]. Ferroelastic domain switching has been observed for these perovskites under compressive loading at room temperature [25,27]. The ferroelastic mechanical behavior of $LaAlO_3$ at room temperature has been reported recently [28]. Its non-linear stress-strain behavior is attributed to its ferroelastic nature [25,29–32].

A number of works have been carried out to investigate the mechanical properties such as flexural strength and Young's and shear moduli of Sr-doped lanthanum manganite (LSM) [33], Sr- and Mg-doped lanthanum gallate (LSGM) [14,34], and $La_{0.6}Sr_{0.4}Co_{1-y}Fe_yO_{3-d}$ (LSCF, LSC, LSF) [18,35] under different temperatures. The loading rate effect has also been investigated for LSCF and LCO [19,21]. In the present study, the mechanical behavior of $LaAlO_3$ under various loading rate and temperature conditions is investigated.

2. Experimental

$LaAlO_3$ (LAO) samples were prepared using a solid state reaction method. La_2O_3 (99.99%) and Al_2O_3 (99.99%) starting powders were mixed with ethanol in a ball mill for 48 h. The mixture was then dried at 423 K for 1 h and calcined at 1473 K for 10 h. The calcined powder was ground using mortar and pestle made of alumina. For finer grinding, the ground powder was then mixed with ethanol and zirconia balls and operated in a ball mill for 48 h. The mixture was then dried at 423 K for 1 h, which was pressed uniaxially into circular discs, and sintered at 1773 K for 10 h (hereafter called sample A).

* Corresponding author.
E-mail address: araki@mech.saitama-u.ac.jp (W. Araki).

http://dx.doi.org/10.1016/j.jeurceramsoc.2016.11.036
0955-2219/

Table 1
Sintering conditions, lattice constant, rhombohedral angle, grain diameter and porosity of prepared samples.

Sample	1st Sintering temp. (K)	2nd Sintering temp. (K)	Lattice constant (Å)	Rhombohedral angle (α_r)	Grain diameter (μm)	Porosity (%)
A	1773	–	5.37	60.10	1.7 ± 0.3	29
B	1773	1773	5.36	60.08	2.9 ± 0.8	20
C	1773	1873	5.36	60.05	6.8 ± 1.9	2

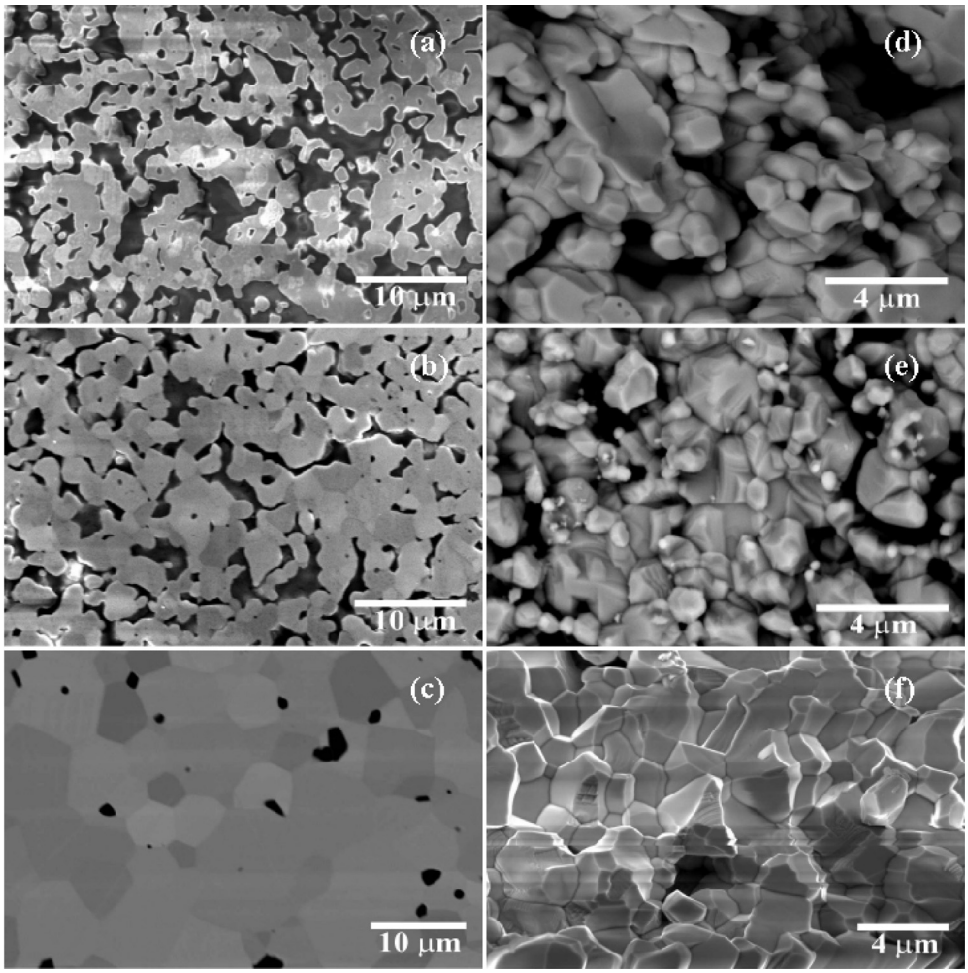

Fig. 1. Scanning electron microscope images of polished surfaces (a–c) and fractured surfaces (d–f).

The sintered discs were cut into rectangular shaped specimens of $3.0 \times 3.0 \times 15\ mm^3$ with a low-speed diamond saw. The cut specimens were annealed at 1073 K for 1 h to remove residual stresses. After the subsequent evaluations were completed, the test specimens were ground once again following the procedure described above and sintered at 1773 K for 10 h (hereafter called sample B) or at 1873 K for 10 h (called sample C hereafter), and then test samples were prepared for evaluation as described above. Table 1 summarized the sintering conditions of the prepared specimens.

For scanning electron microscope (SEM) observations, annealed samples were embedded in molding resin and one side of the sample was polished with SiC sandpaper, 3 μm and 1 μm diamond paste, and finally colloidal silica (~80 nm) using a polishing machine (IM-P2, IMT). The mirror polished surface of each sample was examined under a scanning electron microscope (JSM-5600, JEOL) to observe its grain and ferroelastic domain structure. Grain diameters were measured from SEM micrographs using the linear intercept method. The pristine surface and fracture surface of as-sintered specimens were also examined using SEM.

The Archimedes method was used for porosity measurements. After weighing the dry sample (W_{dry}), it was placed in water for 24 h and then weighed as the saturated sample (W_{sat}). The saturated sample was then weighed while immersed in water (W_{wet}). From these three weights, the porosity was calculated according to $\phi = (W_{sat} - W_{dry})/(W_{sat} - W_{wet})$.

To examine their crystal structure, the sintered samples were analyzed using a X-ray diffraction (XRD) system (XRD-6100, Shimadzu) operated at a current of 30 mA and voltage of 40 kV with CuKα radiation. Scanning was performed at a rate of 2°/min in a 2θ range of 20° to 70°.

To evaluate the mechanical behavior of the samples, uniaxial compression tests were performed using a universal material testing machine (AGS-X, Shimadzu). A dynamic strain meter (DC-204R, Tokyo Sokki) and strain gauges (FLA for 293 K, CEFLA for 93 K, 193 K, and 393 K, and ZFLK for 553 K, Tokyo Sokki) were used. To avoid bending effects, two strain gauges were used on opposite faces of the specimen. Compressive stress up to 100 MPa was applied to the specimens at room temperature (293 K) with three different loading rates from 0.03 to 5.75 MPa/s. For temperature dependence

Please cite this article in press as: M.N. Islam, et al., Mechanical behavior of ferroelastic $LaAlO_3$, *J Eur Ceram Soc* (2016), http://dx.doi.org/10.1016/j.jeurceramsoc.2016.11.036