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FERROELASTIC MECHANICAL BEHAVIOR OF POROUS $LA_{0.6}SR_{0.4}CO_{0.2}FE_{0.8}O_{3-\Delta}$ PREPARED USING CORN STARCH AS A PORE FORMER

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Abstract— The present study investigates the deformation behavior of porous $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) under uniaxial compression. The porous LSCF samples were prepared using corn starch as a pore former with porosity ranging from 16.5% to 51%. XRD patterns of prepared sample showed the reflection of LSCF perovskite with rhombohedral crystal structure. Uniaxial compression tests were performed at room temperature and a loading rate of 1.0 MPa/s. All samples exhibited non-elastic stress-strain behavior during loading-unloading cycles owing to ferroelasticity. The slope of the stress-strain curves decreased with increasing the porosity and during unloading remnant strain was stored in the material due to domain switching. Ferroelastic parameters like as initial modulus, loading modulus and critical stress of the prepared material decreasing with increasing porosity.

Key Words- Porous LSCF, Corn starch, Ferroelasticity.

I. INTRODUCTION

Porous ceramics are frequently used for a variety of application such as solid oxide fuel cell (SOFC), filters, gas sensors, catalyst supports etc [1]-[11]. Considering the porous structure of ceramics, the combination of mechanical properties with controlled porosity and microstructure are of great importance. Specifically, controlled porosity are required for functional ceramics like as solid oxide membrane, piezoceramic hydrophone etc [12], [13]. Many different preparation procedure and use of various pore forming agents to prepare porous ceramic have been studied [14]-[17].

Isobe et al. prepared the porous alumina ceramics containing the porosity ranging from 15% to 43% by the use of polymethyl methacrylate (PMMA) as a pore forming agent [18]. Chen and Miyamoto presented the result of porous ceramics prepared using carbon as a pore formering agent [19]. Considering cost effective pore forming agent and due to its favorable chemical composition which is easily burnt out without leaving residue in the final body, starch is one of the biopolymer pore former used in ceramic technology [20]-[23]. The porosity effect on the mechanical behavior of porous ceramics prepared with different pore forming agents like as PMMA, carbon, graphite, starch etc. has been investigated for compressive strength, bending strength, elastic modulus [24]–[27]. Generally, with increasing porosity, a decrease in value of these properties was observed. The present work evaluates the ferroelastic mechanical behavior of porous LSCF prepared using corn starch as a pore forming agent.

II. EXPERIMENTAL

$La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) powder (AGC Seimi Chemical, Japan) and corn starch (Hansa Corn Starch Industries, Dhaka, Bangladesh) was used to prepare LSCF samples with varying porosity. The scanning electron microscope (SEM) image of raw powder of corn starch is shown in Fig.1. Three different weight percent of corn starch namely 10%, 20%, and 30% were mixed with LSCF. The mixing powders were uniaxially pressed at 100 MPa into circular discs and then sintered at 1473 K for 6 hr with heating and cooling rates nearly 1 K/min and 2 K/min respectively. Rectangular (3.0 mm × 3.0 mm × 10.0 mm) shaped specimens were prepared from sintered discs by low speed diamond wheel cutter. In order to remove residual stress, cut specimens were annealed at 1273 K for 1 hr.
In order to confirm the crystal structure, X-ray diffraction analysis (XRD; XRD-6100, Shimadzu, Japan) was performed with Cu Kα radiation at 40 kV and 30 mA at room temperature of the prepared samples at a rate of 2°/min over a 2θ range of 20° to 80°. One side of each sample was mirror-polished using a polishing machine (IM-P2, IMT). A scanning electron microscope (JSM-5600, JEOL) was used to observe the polished surface in order to confirm the ferroelastic domain. Archimedes´ principle was adopted to measure the porosity p of each sample after sintering. Table 1 summarized the sample preparation conditions with lattice parameter. Uniaxial compression tests were performed at room temperature using a universal material testing machine (AGS-X, Shimadzu) and strain gauges (FLA, Tokyo Sokki) in order to evaluate the mechanical behavior of the samples.

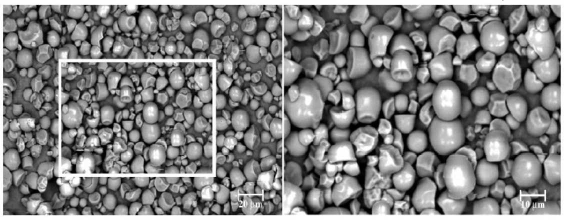

Fig.1 SEM image of raw powder of corn starch

Table 1 Sample preparation conditions

Sample	LSCF	Corn starch/LSCF		
Weight percentage	100	10/90	20/80	30/70
Porosity, *p* (%)	1.5	16.5	28	51
Lattice constant (Å)	5.48	5.50	5.48	5.47
Rhombohedral angle (°)	60.43	60.2	60.14	60.17
Sintering Temp., K	1473	1473	1473	1473

Two strain gauges were used in order to minimize the effect of bending stress. Compressive stress was applied to the specimens at room temperature (293 K) with at loading rates of 1.0 MPa/s to observe the stress-strain behavior. For 10%, 20%, and 30% pore former samples, a stress increment of 40 MPa, 20 MPa, and 10 MPa was used respectively.

III. RESULTS AND DISCUSSION

Fig. 2 shows XRD patterns of dense LSCF and porous LSCF sample prepared using 20% corn starch. The results showed that the samples had a rhombohedral (R-3c) crystal structure. Fig. 3(a)–(c) shows the SEM images of the polished surfaces of the LSCF samples prepared with different percentage of pore former and Fig. 3(a´)–(c´) shows the enlarged images of rectangular mark region of Fig. 3(a)–(c). The stress–strain curves of dense LSCF and corn starch/LSCF samples subjected to loading–unloading cycles were shown in Fig. 4 and Fig.5 respectively. To avoid material failure during experiment, cyclic stress–strain experiments were performed in number of steps. The uniaxial compression tests as shown in Fig. 4 and Fig. 5 proves the ferroelastic mechanical behavior of the dense LSCF and porous LSCF prepared with corn starch as a pore forming agent. The stress–strain curves showed a nonlinear behavior. All stress–strain curves showed a hysteresis, which is usual behavior for ferroelastic materials [28]. Fig. 6 shows clear comparison of the stress–strain curves of dense LSCF and the LSCF samples prepared with different percentage of corn starch keeping the stress level at ~40 MPa and found that remnant strain increased with increasing the porosity.

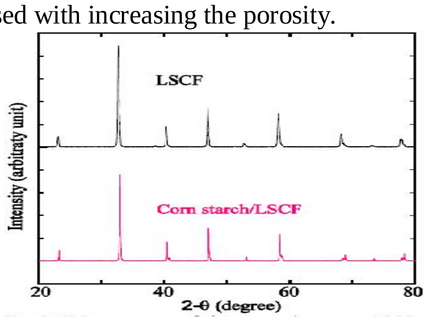

Fig. 2 XRD patterns of dense and porous LSCF.

This is simply explained that deformation increased with increase of porosity due to lowering the actual cross-sectional area. Fig. 7 shows the relationship between porosity and compressive fracture stress of porous LSCF. The average fracture stress decreased from 207 MPa to 42 MPa with increasing the porosity from 16.5% to 51%. The solid line represents the approximation results of fracture stress. The approximation is followed by the modification of well known Knudsen [29] general equation: $\sigma_f = \sigma_0' \exp(-bp)$ where $\sigma_0' = 0.8\sigma_0$, $b = 4.5$, and $\sigma_0 = 550$ MPa, is the compressive fracture stress of dense (no addition of pore former) LSCF sample and p is the porosity. Eom et al. reported that with increasing the porosity from 40% to 70% in porous SiC ceramics, the compressive strength decreased from 240 MPa to 20 MPa [26].

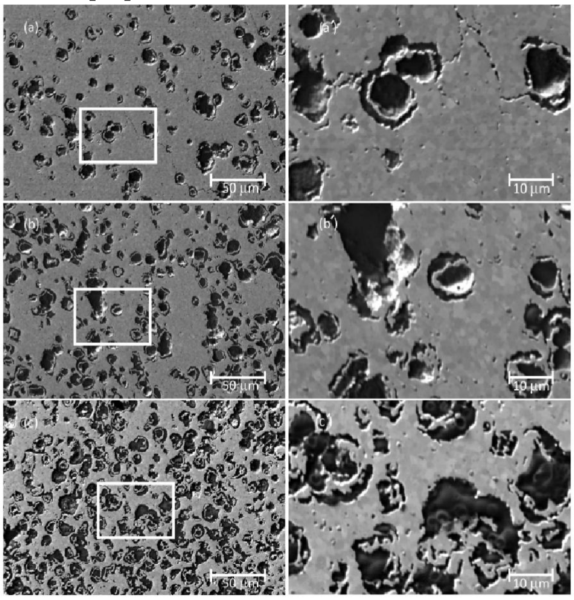

Fig. 3 SEM images of porous LSCF prepared by corn starch as a pore forming agent: (a) and (a´)—10/90 (Corn starch/LSCF), (b) and (b´)—20/80 (Corn starch/LSCF), (c) and (c´)—30/70 (Corn starch/LSCF).

The relationships between the porosity and ferroelastic parameters i.e. initial modulus, loading modulus and critical stress are shown in Fig. 8. The ferroelastic parameters are calculated from Fig. 5. The principle used for calculation of these parameters can be found in our previous study [30]. With increasing porosity, the initial modulus of the porous samples decreases gradually. Similar results reported by Chen et al. [31] that the Young's modulus of porous LSCF is decreased with increasing porosity from 5% to 45%. The loading modulus also decreases with increasing porosity. With increasing porosity, the critical stress monotonically decreases indicating that much lower stress is required for domain switching of samples have higher percentage of porosity. The present results show little bit higher value of ferroelastic parameter than porous sample prepared by PMMA, carbon and graphite as a pore former [32] because present samples contained the pore size variation from 2.5

μm to 15 μm, having homogeneous distribution as shown in Fig. 3.

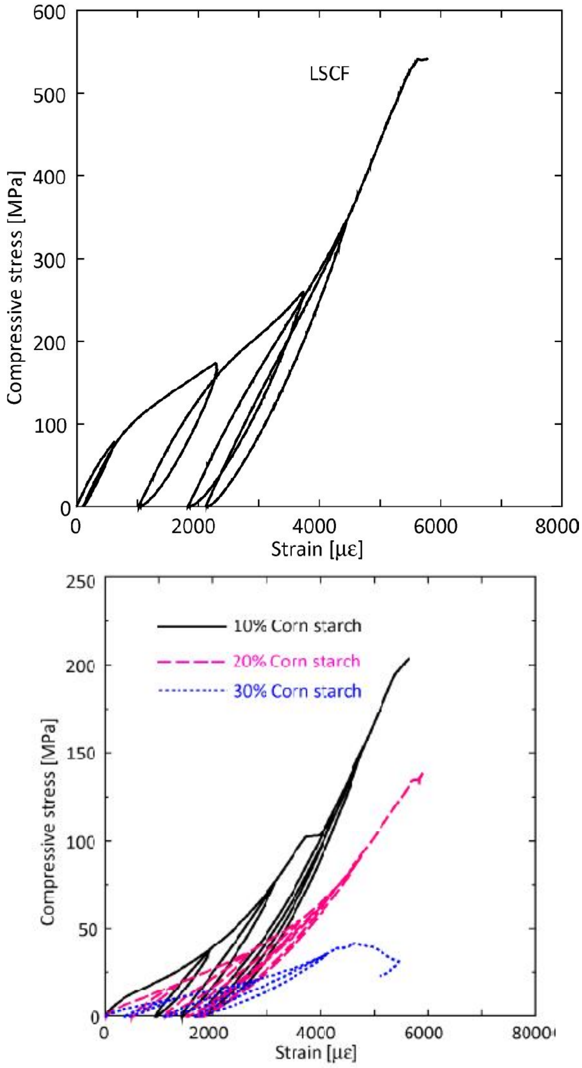

Fig. 5 Stress-strain relationship of porous LSCF.

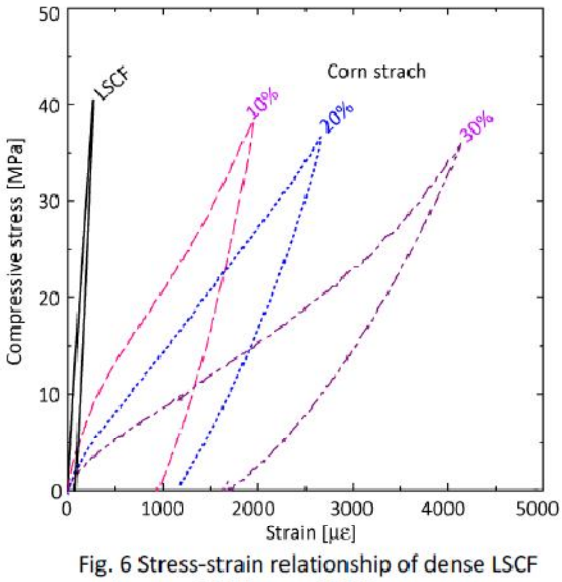

Fig. 6 Stress-strain relationship of dense LSCF and porous LSCF up to 40 MPa.

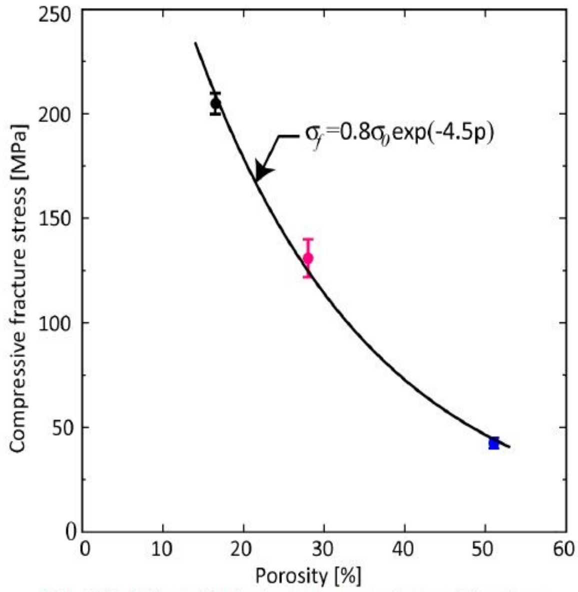

Fig. 7 Relationship between porosity and fracture stress.

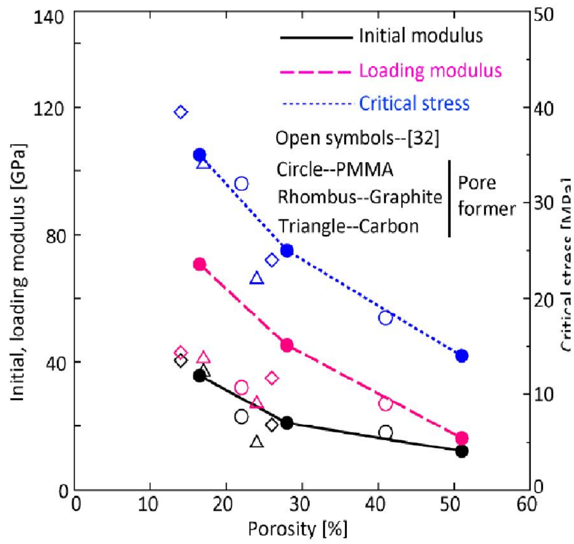

Fig. 8 Relationship between porosity and ferroelatic parameters.

CONCLUSION

The present contribution investigated the mechanical behavior of porous $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) under uniaxial compression prepared using corn starch (10%, 20%, and 30% in weight) as pore forming agents with different porous structures of porosity ranging from 16.5% to 51%. All samples exhibited ferroelastic mechanical behaviors. Compressive fracture stress is influenced significantly by the porous structure. The initial and loading moduli as well as the critical stress are calculated and found higher value than porous LSCF prepared by other pore forming agents in decreased behavior with increasing porosity. The effect of porous structure to fracture stress, ferroelastic parameters prepared by rice starch, chickpea starch will be investigated further.

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