

# Synthesizing Functional Ceramic Powders for Solid Oxide Cells in Minutes through Thermal Shock

Weiwei Fan,<sup>||</sup> Zhichu Ren,<sup>||</sup> Zhu Sun, Xiahui Yao, Bilge Yildiz,\* and Ju Li\*Cite This: *ACS Energy Lett.* 2022, 7, 1223–1229

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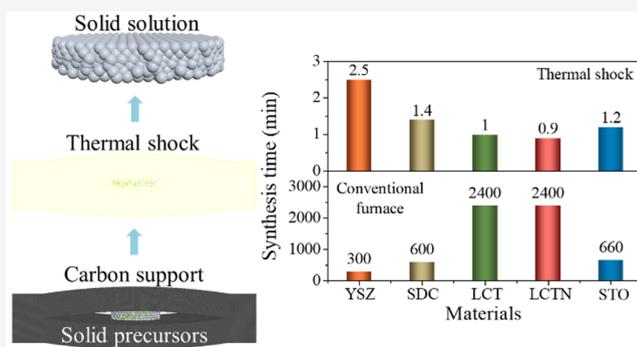


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Supporting Information

**ABSTRACT:** Ceramic fuel/electrolysis cells are key energy/material conversion devices. Here, we report that, by thermal shock, mixed ionic and electronic conducting electrode powders with a perovskite structure can be successfully synthesized within 2 min, while this procedure requires a two-step calcination and almost 40 h when a conventional furnace is used. This benefits from the high temperature supplied to the system, greatly enhancing the reaction kinetics among the raw materials. Moreover, typical electrolyte powders with a fluorite structure, such as  $(Y_2O_3)_{0.08}(ZrO_2)_{0.92}$  (YSZ) and  $Sm_{0.2}Ce_{0.8}O_{1.95}$  (SDC), are also synthesized in several minutes through a thermal shock, which significantly reduces the fabrication time of a solid oxide cell. Also, one can easily prepare multiple samples at one time via tailoring the carbon support size. The availability of fast synthesis of the thermal-shock technique enables the development of new functional ceramic powders for solid-oxide cells in a high-throughput and economical manner.



All-solid electrochemical cells are an important class of energy conversion devices,<sup>1–4</sup> benefiting from their high efficiency and environmentally friendly characteristics. They consist of open porous ceramic electrodes and a dense (airtight) ceramic electrolyte membrane.<sup>5</sup> The employed powders are generally prepared by a solid-state reaction (Figure S1) in a furnace.<sup>6,7</sup> Different solids cannot react with each other at room temperature on a normal time scale (Figure 1a). The diffusion length  $L$  of a diffusing species is  $(2Dt)^{1/2}$ , while the diffusion coefficient ( $D$ ) is temperature ( $T$ ) dependent ( $\propto \exp(-E_a/RT)$ ); thus, a higher diffusion rate could be expected at a higher temperature. A furnace is conventionally used as the heating source. For furnace-based calcination and/or sintering, generally many hours of treatment is needed to obtain the target phase due to the kinetic limitation (Figure S2).<sup>8–11</sup> Moreover, the heating and cooling rates are low (generally less than  $20\text{ }^\circ\text{C min}^{-1}$ ) due to the thermal inertia of the furnace. Thus, one needs to wait for several to many hours to obtain the target powders.

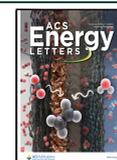
Extensive efforts have been made in the past to reduce the ceramic synthesis time and cost, such as spark plasma synthesis, microwave synthesis, and flash synthesis. Even though these methods can enhance the reaction kinetics and thus reduce the synthesis time, it is still difficult to experiment with multiple

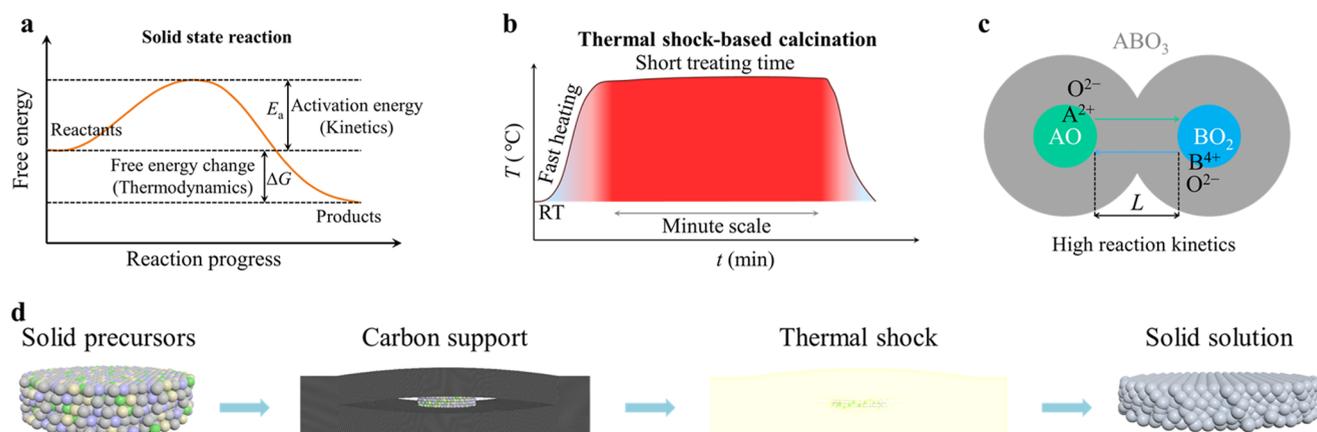
compositions in parallel in a high-throughput way.<sup>12–16</sup> With the aims of rapidly synthesizing and screening multiple functional ceramic powders for solid oxide cells, in this work, a low-cost carbon felt was selected and used as a disposable heating source. To load the as-prepared solid precursors, a cavity was easily manufactured according to the required size of the sample. To increase the contact area of the raw materials and conveniently collect the product(s), the mixtures were compressed into a pellet and then placed into the “mini disposable furnace” surrounded by carbon felt. When a high electronic current was applied across the carbon felt, a large amount of Joule heating can be generated around the sample to drive the solid reaction to take place, thus rapidly forming the target phase (Figure 1b–d). The beauty of this technique includes (1) superhigh heating and cooling rates, (2) flexible size, and (3) low cost. According to the desired dimension of the target sample(s), one can conveniently tailor the size of the carbon

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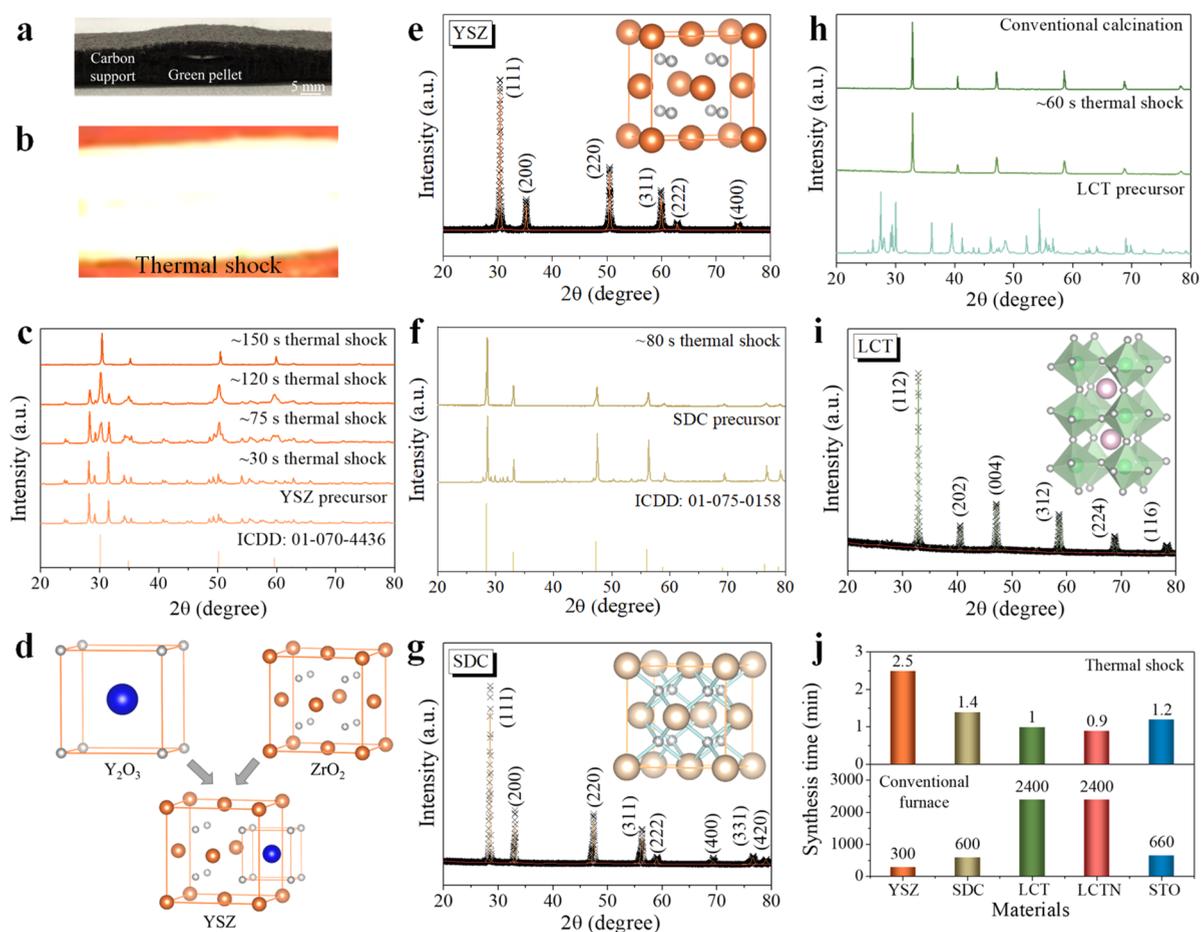
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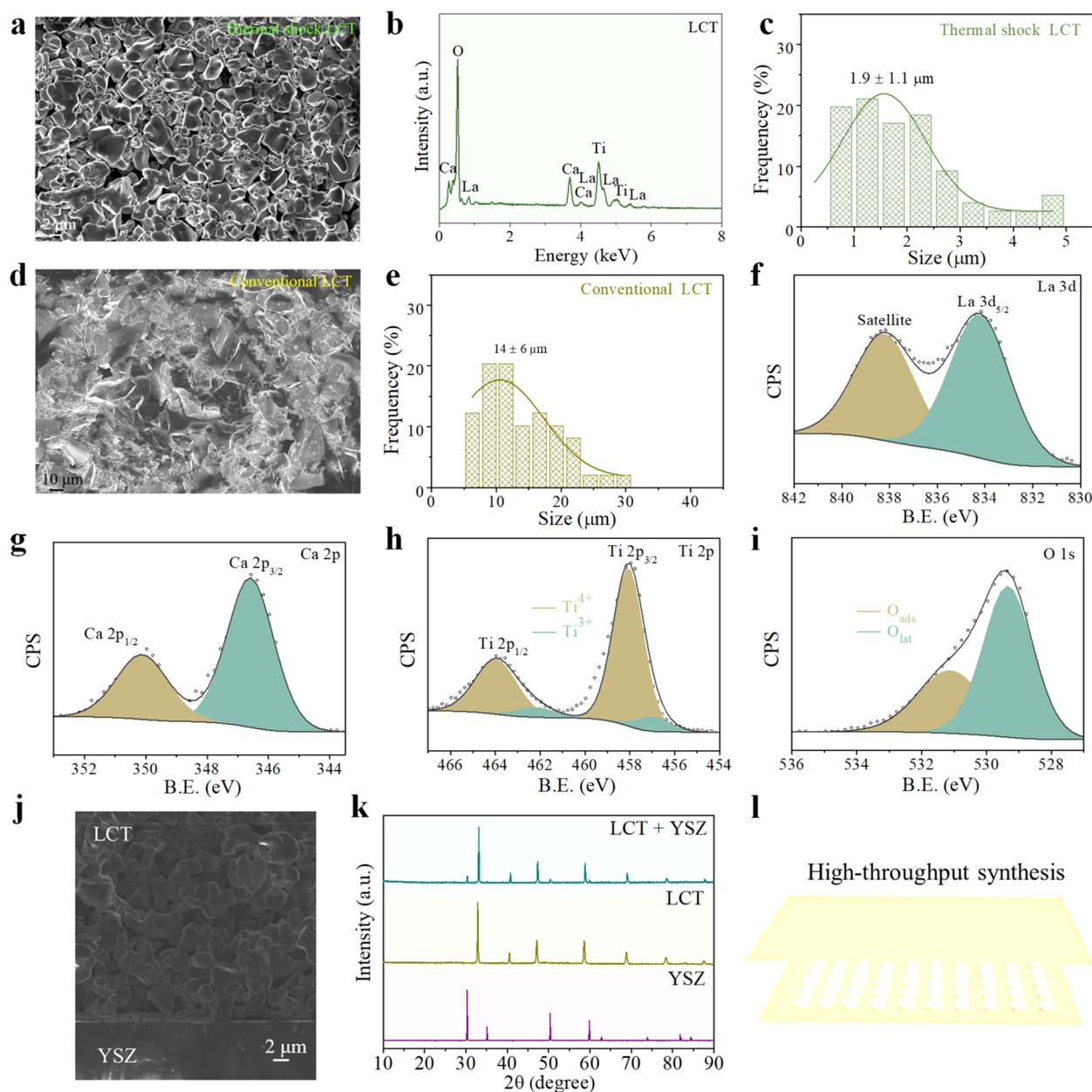




**Figure 1.** (a) Diagram of the thermodynamics and kinetics of the solid-state reaction. (b) Temperature profile as a function of time for calcining solid precursors using a thermal-shock approach. (c) Illustration of a solid reaction between raw materials. High reaction kinetics can be obtained by thermal shock. (d) Schematic of the fabrication of a solid solution by the thermal-shock technique. Each sphere represents a grain. During thermal shock, a pellet form was adopted to increase the contact area among raw materials and thus to enhance the solid-state reaction kinetics. Also, this was beneficial for the collection of the product.



**Figure 2.** Pictures of the carbon support (a) before and (b) during the thermal shock. (c) XRD profiles of YSZ after thermal shocks for different times. (d) Illustration of the reaction to form YSZ. (e) Rietveld-refined XRD spectrum of the YSZ. The inset shows the crystal structure of YSZ. Orange spheres denote Y/Zr, and gray spheres denote O. (f) XRD profiles of SDC after thermal shocks for different times. (g) Rietveld-refined XRD spectrum of the SDC. The inset shows the crystal structure of SDC. Brown spheres denote Sm/Ce, and gray spheres denote O. (h) XRD profiles of LCT after calcination by thermal shock and a conventional furnace. (i) Rietveld-refined XRD spectrum of LCT. The inset shows the crystal structure of LCT. Pink spheres denote La/Ca, green spheres denote Ti, and gray spheres denote O. (j) Comparison of the synthesis times of ceramic materials by thermal-shock-based and conventional furnace-based approaches.



**Figure 3.** (a) SEM image of the LCT powders after cracking and grinding a pellet with the desired phase synthesized by thermal shock. (b) EDX pattern of LCT synthesized by thermal shock. (c) Particle size distribution of (a). (d) SEM image of the LCT powders after cracking and grinding a pellet fabricated by the conventional method. (e) Particle size distribution of the (d). XPS spectra of (f) La 3d, (g) Ca 2p, (h) Ti 2p, and (i) O 1s of the thermal-shock-synthesized LCT. (j) SEM image of the cross-section view of an LCT electrode printed on a commercial dense YSZ electrolyte. (k) XRD profile of the LCT working electrode on the YSZ electrolyte after sintering at 1200 °C for 3 h. (l) Schematic of the high-throughput synthesis of functional ceramics by the thermal-shock technique.

support. Due to the rapid calcination (usually on a scale of minutes), one can synthesize and screen the functional ceramic powders in a high-throughput manner.

In this work, we showed that a functional ceramic electrode and electrolyte powders with different crystal structures (such as perovskite and fluorite) for solid-oxide cells (SOC) were successfully synthesized within 3 min, while generally, the process requires tens of hours with the conventional furnace-based fabrication. As a demonstration, the thermal-shock-fabricated pellets with the desired phase were also converted into an ink followed by assembly into a solid oxide cell. Subsequently, the electrochemical performance was investi-

gated. Thermal shock opens up a new avenue to rapidly and economically prepare functional ceramic powders for SOCs.

**Crystal Structure of the Thermal-Shock-Fabricated SOC Powders.** To investigate the feasibility of the rapid synthesis of functional ceramic powders by the thermal-shock technique, we first prepared the  $(Y_2O_3)_{0.08}(ZrO_2)_{0.92}$  (YSZ) precursor, which is a popular oxygen-ion conductor material in solid oxide cells. Subsequently, the as-prepared sample was sandwiched in the carbon support followed by a thermal shock (Figure 2a,b). The results showed that no obvious change occurred in the XRD profile after thermal shock for ~30 s (Figure 2c), suggesting that nearly no reaction took place among the raw materials during the calcination. A change was observed

in the XRD profile after thermal shock for  $\sim 75$  s, implying a partial solid-state reaction took place and more calcination time was needed to acquire the desired phase. As expected, with the increase of shock time to  $\sim 120$  s, the extent of the solid reaction proceeded further, and the main phase appeared. Surprisingly, after a thermal shock of around 150 s, a single phase was acquired, suggesting that the yttrium was incorporated into the lattice (Figure 2d) and a solid-solution oxide was formed during the thermal shock. Using a conventional furnace, generally several hours are required to obtain the YSZ product.<sup>17</sup> In addition, the results showed that the as-synthesized YSZ could be well indexed on the basis of a cubic fluorite structure belonging to the space group  $Fm\bar{3}m$  (No. 225) (Figure 2e), and the lattice parameter was  $a = b = c = 5.1411(1)$  Å (Table S1). To further investigate the feasibility of synthesizing the fluorite structure ceramic by the thermal-shock approach, subsequently, the precursor of  $\text{Sm}_{0.2}\text{Ce}_{0.8}\text{O}_{1.95}$  (SDC), another broadly used Ce-based ionic conductor in solid oxide cells,<sup>18</sup> was prepared. It was found that after a thermal shock of  $\sim 80$  s, a single SDC phase was acquired (Figure 2f,g and Table S2). Thus, the fabrication period of SDC-based solid oxide cells can be significantly reduced by the thermal-shock technique.

On consideration that the perovskite oxides, which generally need tens of hours to synthesize by a conventional furnace, are good candidates for solid-oxide cells due to the mixed ionic and electronic conducting (MIEC) properties, the Ti-based oxide  $\text{La}_{0.4}\text{Ca}_{0.4}\text{TiO}_{3-\delta}$  (LCT) was subsequently investigated. It was found that a solid solution was formed after a thermal shock of  $\sim 60$  s (Figure 2h). Moreover, the results showed that the as-synthesized LCT presented a perovskite orthorhombic structure belonging to space group  $Pbnm$  (No. 62) (Figure 2i and Table S3). The LCT crystal structure can be represented by a 3D network of  $\text{BO}_6$  octahedra filled with the A-site cations among them. For comparison, an LCT precursor was also calcined in a conventional furnace; a single perovskite phase was acquired after treatment for  $\sim 40$  h.

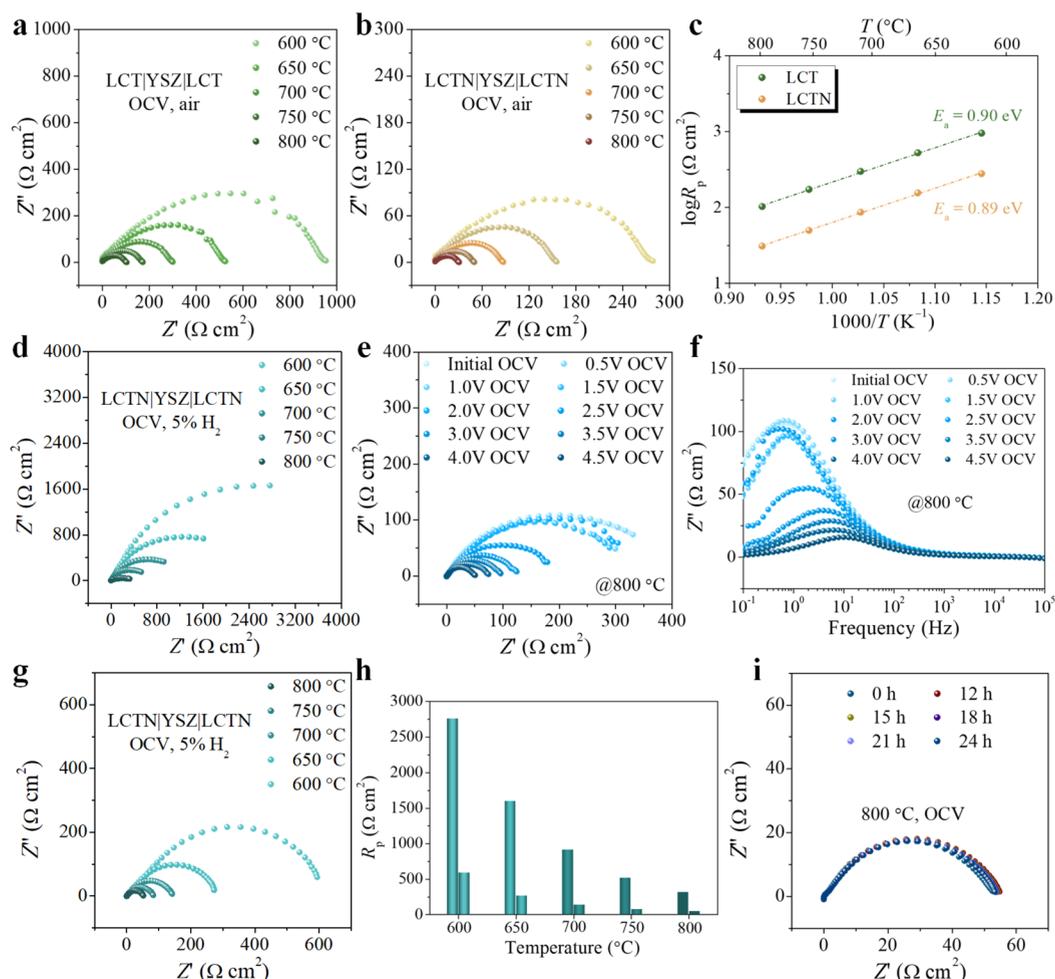
Generally, for perovskite oxides, the introduction of another transition metal possessing lower valence and superb catalytic activity can tune the electrochemical properties of the host oxide; therefore, nickel was introduced and the  $\text{La}_{0.4}\text{Ca}_{0.4}\text{Ti}_{0.94}\text{Ni}_{0.06}\text{O}_{3-\delta}$  (LCTN) precursor was thermally shocked for different times. Similarly, the results showed that with an increase of shock time (Figure S3), the reaction took place and the primary phase formed after an  $\sim 30$  s shock. After a thermal shock of around 55 s, a perovskite phase was achieved (Table S4). In addition, the cubic structure  $\text{SrTiO}_3$  (STO, a good candidate for an electrode backbone for infiltration) within space group  $Pm\bar{3}m$  (No. 221) was also successfully synthesized after thermal shock for  $\sim 70$  s (Figure S4 and Tables S5 and S6). By comparison, it was found that the functional ceramic oxides generally can be synthesized in several minutes by a thermal shock (Figure 2j), while these oxides require several or tens of hours with the traditional furnace-based approach,<sup>17–19</sup> indicating that the thermal-shock technique can greatly enhance the solid reaction kinetics. The fast synthesis ability of this technique can not only greatly decrease the fabrication cost and time of electrode and electrolyte powders for solid oxide cells but also facilitate the development of more advanced ceramics.

**Microstructural and Chemical States of the Thermal Shock Fabricated Powders.** To investigate the micro-morphology, chemical states, and electrochemical performance of the thermal-shock-synthesized ceramics, the pellet products with the desired phase were cracked and ground into powders.

Subsequently, scanning electron microscopy (SEM) was carried out. It was observed that the LCT powder surfaces showed a terrace-like microstructure (Figure 3a and Figure S5), which is typical for an A-site-deficient perovskite oxide.<sup>11</sup> The energy-dispersive X-ray (EDX) result confirmed the composition of La, Ca, Ti, and O elements (Figure 3b). Moreover, an average particle size of  $1.9 \pm 1.1$   $\mu\text{m}$  was obtained for the thermal-shock-fabricated LCT powders (Figure 3c). For comparison, conventional furnace-prepared LCT was also investigated. Due to the long-term treatment, a coarsening phenomenon was very severe, resulting in a denser electrode (Figure 3d and Figure S6). The average particle size is  $14 \pm 6$   $\mu\text{m}$  (Figure 3d). Similarly, our YSZ product showed a particle size of  $\sim 500$  nm (Figure S7), which is relatively smaller than that of the product prepared by a conventional furnace (usually micrometer size). This is mainly due to the short treatment time (several minutes), limiting the particle coarsening after solid-state synthesis. In other words, one can easily acquire functional ceramic powders with a smaller particle size through the rapid synthesis technique of thermal shock. Small particle sizes are often beneficial for ceramic electrochemical cells due to better fracture toughness and thermal quench-cracking resistance. Additionally, EDX results also confirmed the corresponding compositions of SDC, STO, and LCTN powders (Figures S8–S11).

To verify the chemical states of the synthesized powders, X-ray photoelectron spectroscopy (XPS) was conducted on the samples. The C 1s peak (284.8 eV) was used as a reference to calibrate all the collected spectra, and a Shirley-type background was employed during fitting.<sup>20</sup> For LCT, the survey scan XPS result further revealed the composition of La, Ca, Ti, and O elements (Figure S12). Figure 3f shows the La 3d core spectrum of LCT, associated with  $\text{La}^{3+}$  in a perovskite lattice.<sup>21</sup> Similarly, the main peaks appearing in the Ca 2p spectrum (Figure 3g) correspond to the  $\text{Ca}^{2+}$  in LCT.<sup>22</sup> With regard to the Ti 2p core spectrum,<sup>23</sup> it presents a double excitation. The peaks appearing at 464 eV for Ti 2p<sub>1/2</sub> and 458.1 eV for Ti 2p<sub>3/2</sub> can be attributed to  $\text{Ti}^{4+}$ , while the peaks located at 462.2 eV for Ti 2p<sub>1/2</sub> and 456.9 eV for Ti 2p<sub>3/2</sub> can be attributed to  $\text{Ti}^{3+}$  (Figure 3h). For O 1s (Figure 3i), it could be fitted into two subpeaks. The peak with a lower binding energy of 529.4 eV was related to the structure  $\text{O}^{2-}$ , whereas the peak at 531.2 eV was attributed to the adsorbed oxygen from the ambient atmosphere.<sup>24</sup> In addition, for YSZ, the core spectra of Y 3d, Zr 3d, and O 1s further suggested the composition of the lattice  $\text{Y}^{3+}$ ,  $\text{Zr}^{4+}$ , and  $\text{O}^{2-}$  (Figure S13).

For use in solid oxide cells, the electrode powders should have good compatibility with the electrolyte. Therefore, to investigate the chemical and thermal compatibility of the thermal-shock-fabricated electrode powders with the standard commercial YSZ electrolyte, the as-prepared powders were converted into an ink with the proper viscosity and printed on the surface of the YSZ, followed by heterophase diffusion bonding at 1200 °C for 3 h. The result showed that there was a good adhesion between our LCT porous electrode and the commercial YSZ dense electrolyte membrane, and no delamination was observed (Figure 3j). Moreover, the XRD result showed that no obvious additional peaks formed after the diffusion bonding (Figure 3k), implying a desirable chemical compatibility of the thermal-shock-fabricated electrode powders with the YSZ electrolyte. Due to the rapid synthesis ability of the thermal-shock technique, one can prepare and screen new ceramic powders in a high-throughput way. Multiple samples of different compositions can be synthesized in parallel by tailoring the



**Figure 4.** Nyquist plots of (a) LCT|YSZ|LCT and (b) LCTN|YSZ|LCTN symmetrical cells collected from 600 to 800 °C in air under OCV conditions. (c) Arrhenius plots of  $R_p$  for LCT and LCTN electrodes in air. (d) Nyquist plots of an LCTN|YSZ|LCTN symmetrical cell collected from 600 to 800 °C under a wet (3% H<sub>2</sub>O) 5% H<sub>2</sub>/95% N<sub>2</sub> atmosphere under OCV conditions. (e) Nyquist and (f) Bode plots of LCTN collected under OCV conditions after voltage shock at different voltages at 800 °C under a wet (3% H<sub>2</sub>O) 5% H<sub>2</sub>/95% N<sub>2</sub> atmosphere. (g) Nyquist plots of LCTN collected from 800 to 600 °C in wet (3% H<sub>2</sub>O) 5% H<sub>2</sub>/95% N<sub>2</sub> under OCV conditions after a voltage shock at 800 °C. (h) Comparison of  $R_p$  values of LCTN at different temperatures in wet (3% H<sub>2</sub>O) 5% H<sub>2</sub>/95% N<sub>2</sub> after voltage shock. (i)  $R_p$  collected at different times at 800 °C after a voltage shock.

carbon support to the desired size (Figure S1 and Figures S14 and S15). In this mode, the efficiency of developing new functional materials can be greatly enhanced, facilitating the discovery of new advanced ceramics for electrochemical energy devices.

**Electrochemical Performance of the Thermal-Shock-Synthesized Powders.** To investigate the electrochemical performance of the electrode powders of solid oxide cells, usually a symmetrical configuration is applied to benchmark their electrocatalytic activity.<sup>25</sup> On consideration of the desirable thermal and chemical compatibility between YSZ and LCT and LCTN (Figure S16), commercial YSZ electrolyte supported symmetrical cells were prepared and tested under open-circuit voltage (OCV) conditions using a homemade setup (Figure S17).

As shown in Figure 4a, with an increase in the temperature from 600 to 800 °C, the polarization resistance ( $R_p$ ) of the thermal-shock-synthesized LCT perovskite decreased from 954 to 103  $\Omega\text{ cm}^2$  in air, attributed to the thermal activation of the electrochemical reactions. For comparison, the electrochemical performance of the conventional-furnace-synthesized LCT

(Figure S18) was also investigated under the same conditions. The results showed that larger  $R_p$  values were obtained (Figure S19), suggesting an inferior electrocatalytic activity for the oxygen reduction reaction (ORR). This improved performance of the rapidly synthesized electrode powders is presumably related to the small particle size obtained by thermal shock, which can increase the gas diffusion area and shorten the transport path, thus accelerating the electrochemical reactions occurring on the electrode. Subsequently, the electrochemical impedance spectra (EIS) of the thermal-shock-synthesized LCTN electrode powders were collected. It was found that the  $R_p$  values varied between 279 and 31  $\Omega\text{ cm}^2$  in air during the measurement (Figure 4b), which were much lower than that of LCT. This was mainly due to the fact that, when an element with lower valence state was introduced at the B site of the perovskite oxide, to maintain the charge neutrality additional oxygen vacancies will be formed in the perovskite lattice, facilitating the migration of the oxygen ions. Moreover, a lower activation energy ( $E_a$ ) was obtained for the LCTN perovskite (Figure 4c), verifying the enhanced electrochemical kinetics for the ORR. In addition, further improved electrocatalytic activity was obtained

by using a LCTN:YSZ (50:50) composite as the electrode (Figure S20), ascribed to the increased concentration of oxygen vacancies and an extended three-phase boundary length for oxygen ion transfer.

To obtain more information about the electrochemical performance of the LCTN perovskite electrode, wet (3% H<sub>2</sub>O) 5% H<sub>2</sub>/95% N<sub>2</sub> was introduced into the chamber, and then EIS was collected after stabilization. Results showed that the  $R_p$  value went from 2762 to 324  $\Omega$  cm<sup>2</sup> in the temperature range of 600–800 °C (Figure 4d). In comparison to those in air, larger  $R_p$  values were acquired in a reducing atmosphere. This can most probably be ascribed to the p-type conducting behavior of the LCTN (Figure S21). To improve the electrocatalytic activity of the LCTN perovskite oxide, an external voltage was applied to trigger the exsolution of nanoparticles with superb catalytic activity from the perovskite lattice. As shown in Figure 4e,  $R_p$  changed slightly after voltage shock before 1.5 V at 800 °C. In the range of 2–4 V,  $R_p$  gradually decreased from 179 to 74  $\Omega$  cm<sup>2</sup>. At 4.5 V, the  $R_p$  value reached 50  $\Omega$  cm<sup>2</sup>; an almost 6-fold enhancement was obtained in comparison to the  $R_p$  of LCTN before the voltage shock, suggesting that the electrocatalytic activity of LCTN was improved greatly. This was mainly due to the exsolved nanoparticles, which could provide sufficient active sites for the electrochemical oxidation of hydrogen, such as hydrogen adsorption ( $H_2 \leftrightarrow 2H_{ads}$ ), dissociation ( $H \leftrightarrow H^+ + e^-$ ), and water formation ( $2H^+ + O^{2-} \leftrightarrow H_2O$ ). Moreover, the results showed that, during the voltage shock, the impedance change primarily came from the low-frequency portion (Figure 4f), suggesting that the enhancement of the electrochemical kinetics is associated with the gas adsorption and dissociation processes.

To further investigate the electrocatalytic activity of LCTN after the voltage shock, the temperature was decreased programmatically to 600 °C. It was found that the  $R_p$  value increased from 50 to 594  $\Omega$  cm<sup>2</sup> with a decrease in the environmental temperature (Figure 4g). In comparison to those before the voltage shock, the  $R_p$  values were still promising at lower temperatures after the voltage shock (Figure 4h), further demonstrating that the electrocatalytic activity of LCTN was enhanced. In addition, no obvious change in  $R_p$  was detected during the measurement (Figure 4i), indicating a favorable stability of the product. Overall, one can rapidly synthesize the perovskite electrode powders by a thermal shock as well as tune the electrocatalytic activity through an electrochemical voltage shock, greatly enhancing the flexibility of the advanced ceramics in the application of electrochemical cells.

In summary, by rapid Joule heating by a thermal shock inside carbon felt, oxide electrode powders with a perovskite structure for solid-oxide cells can generally be synthesized in 2 min. In contrast, it usually takes tens of hours to acquire the desired phase by using a conventional furnace. This is mainly due to the remarkably enhanced reaction kinetics among the precursors during the thermal shock. We report that other popular electrolyte powders with a fluorite structure, i.e. YSZ and SDC, can also be synthesized in minutes by a thermal shock, implying that the fabrication time and the cost of electrode and electrolyte powders for solid-oxide cells can be greatly reduced. We also report that the thermal-shock-synthesized powders show reasonable electrochemical performance, which can be further improved by an *in situ* electrochemical voltage shock. The rapid synthesis ability of the thermal-shock approach facilitates the discovery of new functional ceramics in a high-throughput manner.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acseenergylett.1c02630>.

Experimental details, schematic of the solid-state reaction, XRD profiles, SEM images, EDX pattern, XPS spectrum, Nyquist plots, conductivity, Rietveld refined parameters, and thermal shock parameters (PDF)

## ■ AUTHOR INFORMATION

### Corresponding Authors

**Bilge Yildiz** – Department of Nuclear Science and Engineering and Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, United States; [orcid.org/0000-0002-2688-5666](https://orcid.org/0000-0002-2688-5666); Email: [byildiz@mit.edu](mailto:byildiz@mit.edu)

**Ju Li** – Department of Nuclear Science and Engineering and Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, United States; [orcid.org/0000-0002-7841-8058](https://orcid.org/0000-0002-7841-8058); Email: [liju@mit.edu](mailto:liju@mit.edu)

### Authors

**Weiwei Fan** – Department of Nuclear Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, United States; [orcid.org/0000-0001-6298-8964](https://orcid.org/0000-0001-6298-8964)

**Zhichu Ren** – Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, United States

**Zhu Sun** – State Key Laboratory of Electrical Insulation and Power Equipment, Xi'an Jiaotong University, Xi'an 710049, People's Republic of China

**Xiahui Yao** – Department of Nuclear Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, United States

Complete contact information is available at: <https://pubs.acs.org/10.1021/acseenergylett.1c02630>

### Author Contributions

<sup>||</sup>W.F. and Z.R. contributed equally to this work.

### Notes

The authors declare no competing financial interest.

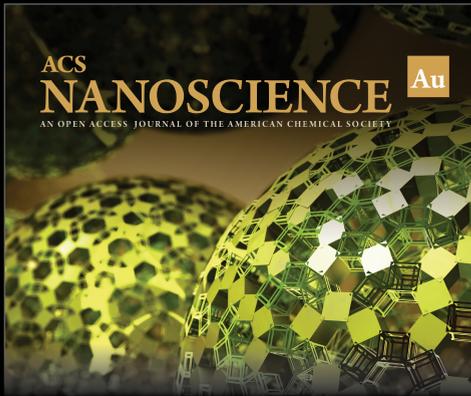
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