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3D reconstruction size effect on the quantification of solid oxide fuel cell nickel–yttria-stabilized-zirconia anode microstructural information using scanning electron microscopy-focused ion beam technique

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Abstract Self-made conventional nickel–yttria-stabilized zirconia composite anodes after reduction and 500 h operation were analyzed by three-dimensional microstructure reconstruction based on focused ion beam-scanning electron microscopy technique. Interfacial area, three-phases-boundary density and phase volume fraction were measured based on the three-dimensional microstructure reconstruction to quantitatively study the statistical characterization of solid oxide fuel cell nickel–yttria-stabilized-zirconia anode microstructure before and after operation. It is found that for anode operated with long time, it is necessary to increase the corresponding three-dimensional reconstruction size to suppress the influence of microstructure variation caused by Ni agglomeration in order to obtain more accurate microstructural quantification information.

Keywords FIB-SEM · 3D reconstruction · TPB density · Tetragonal mesh

1 Introduction

Solid oxide fuel cell (SOFC) has been treated as a high energy efficiency power generation system with fuel flexibility, which attracted more and more attentions in the last few decades. The usual operation temperature for SOFC anode made of nickel–yttria-stabilized zirconia (Ni–YSZ) [1], ranged from 973 to 1273 K, results in the challenge of long time stability and durability of SOFC electrodes. The SOFC anode degradation mechanism has been intensively investigated, which is mainly caused by Ni coarsening, especially during the initial operation period [2–6]. Recently, three-dimensional (3D) reconstructions of SOFC electrode has been achieved by applying focused ion beam-scanning electron microscopy (FIB-SEM) technique [6–11]. This technique provides quantitative information of composite cermet microstructure in 3D, which can be correlated to the electrode performance via specific surface areas and active three-phase boundary density [1]. Compared to quantitative characterization of composite cermet based on 2D images [5], FIB-SEM 3D reconstruction technique leads to more accurate microstructure analysis, and facilitates 3D electrochemical numerical simulation.

Several researchers have investigated the influence of 3D reconstruction size on the statistic quantitative information of composite cermet microstructure [12–15]. The maximum size of 3D reconstruction based on FIB-SEM technique was limited by SEM image drifting and shadow effects which is quite difficult to be solved in the current stage. The typical maximum length of FIB-SEM reconstruction is always under 30 μm, which might be insufficient to be used for extracting the representative quantitative information from a specific microstructure. In this paper, the effect of FIB-SEM 3D reconstruction size is quantitatively studied based on two 3D reconstructions with the largest sizes which we can supply with the current experimental conditions. Quantitative characterizations of microstructures of Ni–YSZ composite anodes after reduction and 500 h operation were compared to study the minimum size of 3D reconstruction which can be representative for the quantification of microstructural information.

SPECIAL TOPIC: Modeling of Solid Oxide Fuel Cells

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2 Experimental

2.1 Anode fabrication and operation

Electrolyte-supported cells were used in this study. The anode cermet was prepared by ball-mixing NiO and YSZ powders (AGC Seimi Chem. Corp., Japan) for 48 h where the volume ratio of Ni–YSZ was 43 %:57 % after Ni reduction. The mixture was then mixed with terpineol solvent and the ethylcellulose binder in agate mortar to obtain anode slurry. The slurry was then screen-printed onto commercial dense 8 mol% YSZ pellet (diameter 20 mm, thickness 0.5 mm, Fine Ceramics Corp., Japan) with a diameter of 10 mm and then sintered at 1400 °C for 3 h. The cathode material was prepared by mixing $(La_{0.8}Sr_{0.2})_{0.97}MnO_3$ (LSM) powder with YSZ powder in a mass ratio of 50 %:50 %. Similar method as the anode was used to obtain cathode slurry. The slurry was screen-printed onto the counter side of the commercial YSZ pellet with a diameter of 10 mm as cathode. The cathode was then sintered at 1200 °C for 3 h. The anode was then operated in 3 % H_2O humidified hydrogen with a current density of 200 mA cm^{-2} for 500 h to compare with the initial anode microstructure after reduction. The details of anode operation have been introduced in Ref. [6].

2.2 FIB-SEM observation

The anode samples before and after operation were observed by FIB-SEM (Carl Zeiss, NVision 40). The samples were first infiltrated by low viscosity epoxy resin under low pressure atmosphere (ca. 15 Pa). The samples were then polished by using Ar-ion beam cross-section polisher (JEOL Ltd., SM-09010) for FIB-SEM observation, so that the pores of the porous electrode could be distinguished during FIB-SEM observation as shown in Fig. 1. The white, gray and black regions correspond to Ni, YSZ and pore, respectively. The details of dual-beam FIB-SEM observation technique have been introduced in Refs. [6, 11]. The 3D microstructure of the Ni–YSZ anodes before and after 500 h operation were virtually reconstructed based on 2D image series, as shown in Figs. 2

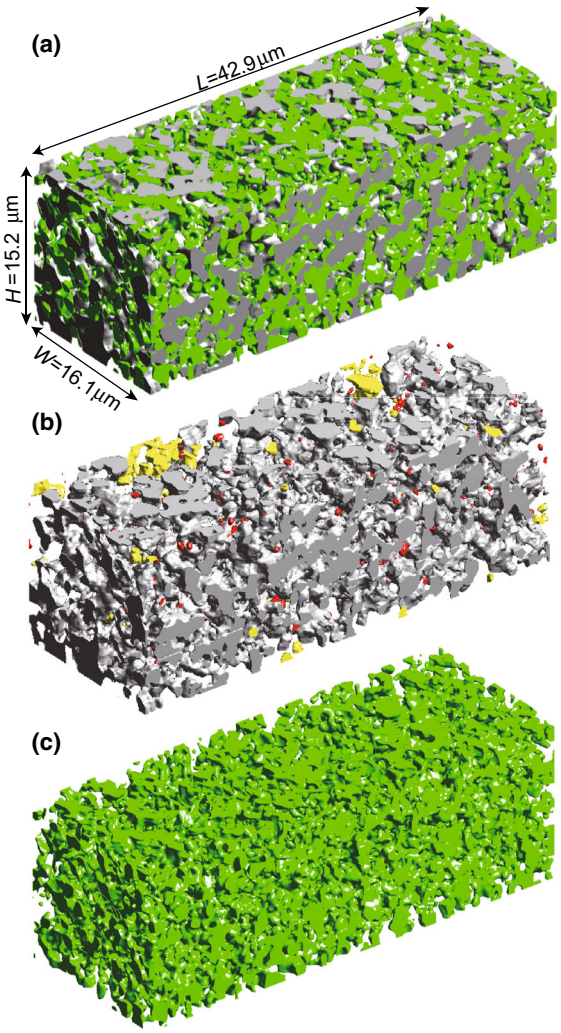

Fig. 2 (Color online) 3D reconstruction images of anode **a** Ni–YSZ, **b** Ni phase (red isolated particle, yellow unknown-status particle) and **c** YSZ phase after reduction. Gray Ni and green YSZ

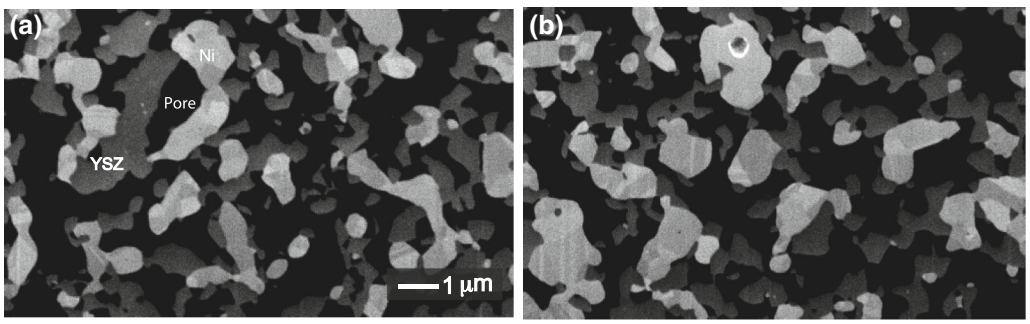

Fig. 1 2D SEM images of anodes (**a**) after reduction and (**b**) after 500 h operation. White Ni, gray YSZ and black pore