Solid oxide fuel cell (SOFC) is considered as a highly efficient device to convert chemical fuels directly into electrical power through the electrochemical oxidation of fuels in the anodes, typically NiO-YSZ (yttrium-stabilized zirconia). The synthesis procedure will influence the microstructure and further the performance. Sintering is one of the most important procedures. Although quite a lot of work has been done on the shrinkage of NiO or YSZ as well as the microstructure of cells sintered at high temperature (>1200 °C), little is known on the microstructure evolution of electrodes during the sintering process. Herein, we use high-resolution scanning electron microscopy (HRSEM) to observe the microstructure of NiO-YSZ cells after sintering to different temperatures.

Four NiO-YSZ half-cells were prepared by using ethyl cellulose as a binder or pore former through the screen-printing technique (C1). C1 was sintered in air to 700 °C (C2), 1100 °C (C3), 1250 °C (C4) and 1400 °C (C5) respectively without holding, as well as 1400 °C with holding for 3 h (C6). The heating/cooling rates were 2 °C/min. The surfaces and interfaces morphology was observed in SEM (Zeiss ULTRA55) with four detectors: lateral secondary electron (SE), InLens SE, energy selective backscatter (EsB) and angle selective backscatter (AsB).

In Fig. 1(C1 & C2), the particles with sharp edges in SE images correspond to the gray colour in the ESB images and they are NiO. NiO particles have relatively larger sizes than YSZ particles, which are apparently round shape. ESB signal gives planar image and it is difficult to see the boundary between two particles of one phase if they are closely connected. The connection of particles becomes closer in C2 than that in C1 since ethyl cellulose is already burned away at 700 °C and the space is easily occupied by fine YSZ or NiO. The morphology of C3 is obviously changed. Different particles start to sinter together and form short skeletons. The porosity increases significantly compared with C2. In Fig. 1(C4), the ESB and ASB images are compared. The ASB signal contains not only the contrast information as ESB, but also the morphological information. So it is favourable to separate connected particles of one phase in ASB image. Sintering becomes significant and porous skeleton is formed with pore size ranging from ~100 nm to ~2 μm. For C5 and C6, only the ASB images are shown. The sintering continues and the skeleton becomes coarser and stronger from C4 to C6. The porosity is decreasing due to the coarsening and shrinkage of particles. Also, it is noticeable more and more YSZ particles migrate onto the top surface to form larger networks.

The particle size distributions as well as the activation energy were evaluated based on these images.

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Fig. 1: SEM images of the surfaces of C1 ~ C6. Images were taken by different detectors at the same position. YSZ: bright; NiO: gray.