

実験報告書様式(一般利用課題・成果公開利用)

(※本報告書は英語で記述してください。ただし、産業利用課題として採択されている方は日本語で記述していただいても結構です。)

 MLF Experimental Report	提出日 Date of Report
課題番号 Project No. 2013B0184 実験課題名 Title of experiment Reduction of nickel oxide and induced strains during high temperature operation of anodes and anode supports for Solid Oxide Fuel Cells. 実験責任者名 Name of principal investigator Markus Strobl 所属 Affiliation European Spallation Source ESS-AB	装置責任者 Name of responsible person Kenichi Oikawa 装置名 Name of Instrument/(BL No.) BL 10 実施日 Date of Experiment 2014/03/02 09:00 – 2014/03/05 21:00

試料、実験方法、利用の結果得られた主なデータ、考察、結論等を、記述して下さい。(適宜、図表添付のこと)
 Please report your samples, experimental method and results, discussion and conclusions. Please add figures and tables for better explanation.

1. 試料 Name of sample(s) and chemical formula, or compositions including physical form. Support and anodes for Solid Oxide Fuel Cells Ni+YSZ (Zr ₂₀ +Y ₂₀) Solid state layers
--

2. 実験方法及び結果 (実験がうまくいかなかった場合、その理由を記述してください。) Experimental method and results. If you failed to conduct experiment as planned, please describe reasons. We study progress of reduction reaction in anodes and anode supports for solid oxide fuel cells (SOFC) and its relation to applied external stress to the material. In this phase of our project we expected to observe a dependence of the reduction rate on external stress. We have performed neutron energy resolved imaging with spatial resolution better than 200µm (using MCP detector brought by Anton Tremsin) of samples in different stages of the considered process. We have observed the distribution of Ni, respectively NiO within anode supports (porous Ni-YSZ cermet) after different reduction times and under different conditions i.e. different temperatures and external stresses. Reduction process of samples was conducted in advance at Technical University of Denmark (DTU) without stress and under applied external stress. This reaction was carried out under different conditions i.e. different temperatures, different atmospheres (different partial hydrogen pressures) and different values of external stresses and different reduction times. The geometry of our setup caused bending of the layers and in result we've obtained compressive stress on one side of the sample and tensile stress on the other side. Reduction rate in these two situations was expected to be different, therefore we have measured samples also after reduction conducted without applied load in order to
--

2. 実験方法及び結果(つづき) Experimental method and results (continued)

compare the results and observe the influence of the external stress on the reaction progress.

In fig. 1 we present examples of results from J-Parc, where we have measured anode supports (prepared in advance) after different times of reduction, both with and without external stress. The inset in fig. 1 shows Bragg edge patterns for three different parts of one sample, which was partially reduced under external stress. Three plots show the patterns for the compressed side, center area and tensioned side of the sample. Differences for the three regions are apparent and it is clear that on the compressed side, the amount of Ni phase is higher than on the tensioned side.

Most of the bent (reduced under external stress) samples (about 70-80 %) exhibit the same effect of difference in NiO and Ni phases in anode supports for SOFC between compressed and tensioned side. Moreover, in the reference samples (not stressed) no difference was observed in reduction rate between different sides.

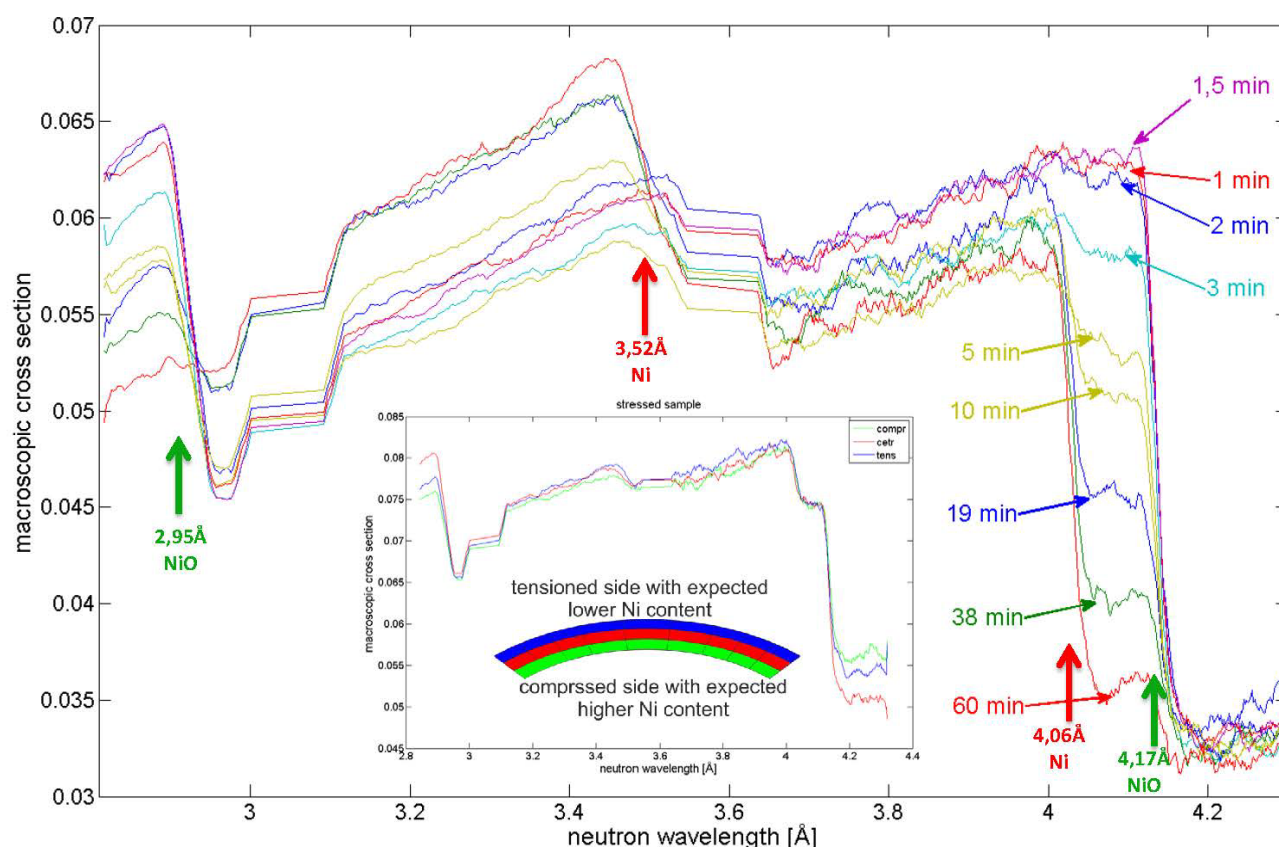


Fig.1 Bragg edge patterns for samples after different times of reduction. Inset shows plots for compressed (green), center (red) and tensioned side (blue) of a bent sample (reduced under external stress). All the samples had the same dimensions: $20 \times 6 \times 1 \text{ mm}^3$.

Measurements were performed with different exposure times, and even few minutes acquisition time was enough to obtain sufficient signal to distinguish Ni and NiO phases. This proves the feasibility of time resolved experiment, which will be the next step of this research project.

Information obtained in this experiment is of great interest for SOFC researchers. It was a first and very successful feasibility test towards in-situ measurements, and moreover it was first and unique experiment of this type.