From In-Situ Surface Observation to Post-Test Sub-surface Microstructure and Chemical Analyses: Oxidation of Coated SOFC Steel

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Coatings on Solid Oxide Fuel Cell (SOFC) interconnects are complex and interesting systems. Due to the multiple requirements to fulfill their functions, state-of-the-art SOFC interconnects are made of high-chromium-content multi-components steel and are coated to improve their stability at high temperature. This leads to complex interactions between the different chemical elements during their lifetime.

In this study, a combination of electron microscopy techniques is used to analyze the interconnect steel oxidation in its initial stage (the first hours to the first days), where scale is built up and first elemental diffusions and interactions may be clearly seen. The studied system so far concerns Ce-Co coated SSHT steel (Sandvik Sanergy High Temperature steel). First the surface of a coated steel piece is observed in an environmental scanning electron microscope (E-SEM) at high temperature (up to 900°C) under a certain pressure of pure oxygen (up to 500Pa) allowing to directly observe its morphological evolution during first oxidation. Ripening of the cobalt oxide grains can be observed in real time, showing regular square shapes due to the cubic spinel structure of Co_3O_4 (Fig.1 (a)). From about 20 hours of oxidation, new grains nucleate and grow, tearing apart the cobalt oxide grains (Fig.1 (b)-(c)). For post data treatment, images were segmented using a Weka machine-learning plug-in from ImageJ (Fig.1 (d)). The size of the grains on the surface was quantified as a function of time (*). After the oxidation experiment in the E-SEM, a focused ion beam (FIB) was used to prepare a TEM lamella from the exact same zone as that observed during the in situ exposure (white rectangle in Fig.1 (a)-(c) and black rectangle in Fig. 1(d)). This targeted post-test TEM cross section observation (Fig. 1 (e)-(f)) shows that the first oxidized grains (<20 h) contain cobalt only while later oxidized grains (>20 h) also contain manganese. This suggests that the diffusion of manganese from the steel is responsible of the nucleation of new grains after 20 hours of in-situ oxidation.

This complete analysis method allows the correlation of the sub-surface microstructure and chemistry with the surface structures seen during the in situ reaction process. This can be applied to other parts of the SOFC to help improving our understanding of the degradation and evolution of the various components.

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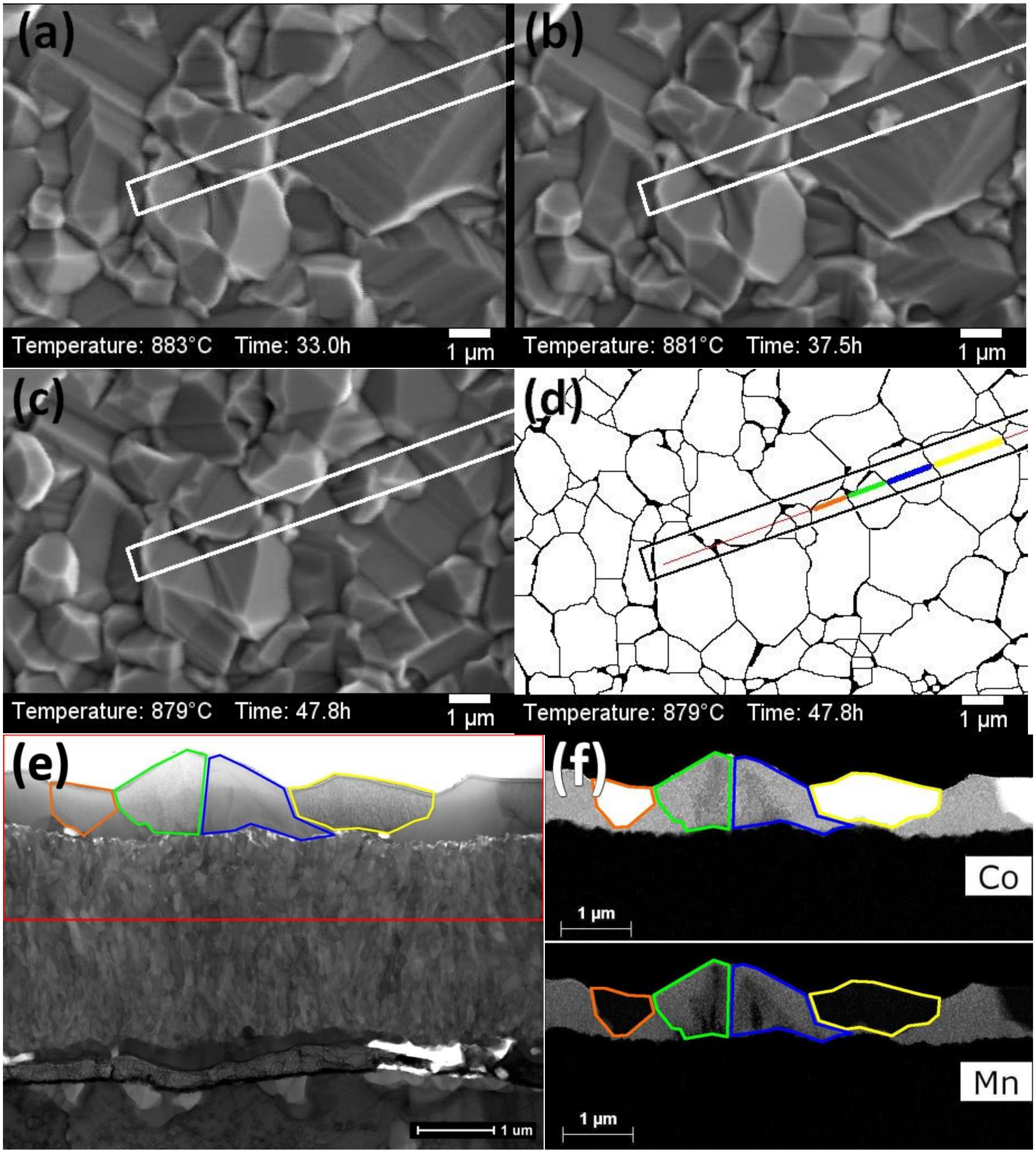

Fig.1: (a-c) Observation with ESEM (images are extracted from: https://goo.gl/VBXYDh, the complete video of the 48 hours oxidation: https://goo.gl/rMRXyB), (d) segmentation, (e) TEM analysis and (f) EDX analysis of the Ce-Co coating oxidation with color identification for 4 grains. The green and blue colored grains grew out of the structure starting at ≈35h (see (b)) and contain Mn and Co (f), unlike the red and yellow colored grains, that are part of the initial scale and only contain Co (f).