ID-12-O-3099 In-situ environmental TEM studies using MEMS based devices


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Investigating dynamic changes in specimen while applying a stimulus inside a transmission electron microscope (TEM) has always been an exciting field of study. With the advancements in TEMs and microelectromechanical systems (MEMS), in-situ TEM has progressed extensively over the last decade. Here, we show the application of MEMS based devices developed in-house to carry out in situ heat-treatment and environmental TEM studies. For environmental TEM studies, a controlled environment is achieved inside the TEM by one of the following approaches: the open type, using a differentially pumped vacuum system where the reactive gases are spread around the specimen area of the TEM; and the closed type, using a windowed environmental cell. Our studies are based on the closed environmental cell called the nanoreactor, Fig.1. The nanoreactor consists of two silicon chips facing each other with thin electron-transparent silicon nitride membranes. One half of the nanoreactor (bottom half) is embedded with a Pt coil for resistive heating. These MEMS based devices in combination with specially developed TEM holders, Fig.3, make it possible to carry out a wide range of experiments, a few of which are mentioned here.

Using one half of the nanoreactor, we have demonstrated the three-dimensional compositional and structural evolution during heat treatment at 100-240°C in a FIB specimen of a commercial aluminium alloy AA2024, revealing in unparalleled detail where and how precipitates nucleate, grow or dissolve. On closing the nanoreactor with the other half, we have shown that it is also possible to carry out gas-liquid-material interactions. One such example is the room temperature corrosion studies of AA2024-T3 exposed to oxygen bubbled through aqueous hydrochloric acid of pH = 3 at a pressure of ~ 1.5 bar. In the present study, we investigate the corrosion behaviour of AA2024 after growing the needle-like S-phase type precipitates by in situ heat-treatment. These sort of experiments are critical to understand the performance of engineering materials in service conditions. Furthermore, will present some of our preliminary results on metal-liquid electrolyte interfaces to extend the environmental-cell based approach for introducing liquids inside the TEM.

References:

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Fig. 1: (a) Conceptual sketch of a nanoreactor. (b) Photographs of the top and bottom chip, with ~ 400 nm Si₃N₄ window in 800 X 800 µm² area; inlet and outlet for reactive gases in the bottom chip.

Fig. 2: Optical micrographs of the 400 nm Si₃N₄ window showing (a) top and (b) bottom chips respectively with 10 µm circular holes over spanned with ~ 20 nm Si₃N₄ membranes for electron transparency. The bottom chip is embedded with a Pt spiral for heating. The red encircled region in (c) showing aligned assembly.

Fig. 3: Holders for in-situ TEM studies: (a) Gas holder with Pt tubing; (b) A single-high-tilt heating holder in which the bottom chip of the nanoreactor can be used for in situ heating experiments. This holder has a capability to tilt through ±70°, which makes it possible to carry out tomography experiments too.