

In Situ SEM at Elevated Temperature for Materials Science

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Understanding the relationships between microstructure and properties under various conditions is essential for further material development. SEM is suitable for studying not only microstructure but also changes in these materials under different and variable conditions. In the case of elevated temperatures, phenomena associated with diffusion, phase transformations, grain boundaries and precipitation are particularly significant in materials science [1]. Several limitations of in situ experiments in SEM are related to the vacuum environment, which acts as an electrical and thermal insulator. The thermal insulating property of vacuum reduces the risk of damage to microscope components during heating experiments but also complicates sample heating and cooling and temperature measurement. Vacuum prevents both sample oxidation and equipment oxidation during experiments at high temperatures. The development of specific detectors has been necessary to carry out in situ studies under extreme conditions, such as high pressure and temperature [2]. The aim of these experiments was to verify the possibilities of observing phenomena characteristic of materials science using in situ SEM at elevated temperatures. In addition to the phenomena themselves, the aim was to verify the visualization of structure by thermal etching [3]. Pilot experiments were conducted, including melting of the AlMgSi1 alloy, dissolution of secondary phases and visualization of primary austenite grains of TRIP steel, dissolution of intermetallic phases and melting of the AlSi9Cu3(Fe) alloy, phase transformation of the Ti6Al4V alloy, precipitation of phases during artificial aging of CuBe2 and AlMgSi0.5 alloys, and thermal etching of ZrO₂ ceramic for grain visualization.

The samples underwent a complete conventional metallographic procedure, including cutting on the precision metallographic cutter, followed by mounting in resin, and successive grinding and polishing steps. Grinding was performed using SiC papers with grit sizes of 220, 500, 1200, 2000, and 4000, while polishing utilized diamond pastes with abrasive sizes of 3 μm, 1 μm, and 0.25 μm, concluding with chemical-mechanical polishing using the colloidal suspension with SiO₂ particles. Before heating, the samples were chemically or color etched to reveal the real structure. Some samples underwent imaging in the SEM using the LOM Zeiss Axio Observer 7 before heating for correlative imaging. In situ heating experiments took place in the SEM Thermofisher Scientific Quattro S, utilizing the Newtec Scientific FurnaSEM 1300 stage for heating up to 1200 °C. The sample was restrained to a 12 mm diameter by a platinum pad, ensuring uniform heating. To enhance thermal conductivity and dissipate charge, samples were affixed to the platinum pad using a carbon suspension. Subsequently, a thermal shield with a 4 mm diameter aperture was installed above the sample to mitigate thermal stress on the microscope chamber components. In addition to the ETD detector, samples were also imaged during heating using the BSE detector Crytur Karmen, equipped for in situ experiments at elevated temperatures. The scintillation crystal of the Karmen detector is coated with a thin layer of aluminum, 100 nm thick, designed to prevent penetration of external light signals into the photomultiplier, such as light emitted by the heated sample [4]. During the melting of the AlMgSi1 alloy, an Al₂O₃ pad was inserted between the sample and the platinum pad. Upon reaching a temperature of 700 °C, microdendrites were observed at the edges of the sample, indicating localized melting. However, this temperature is approximately 150 °C higher than the theoretical melting temperature of the alloy. In the case of TRIP steel, reaching a heating temperature of 900 °C made the boundaries of primary austenite grains visible. With prolonged exposure at this temperature, secondary phases

began to dissolve, gradually increasing the contrast of the grains. Further experiments were carried out using a carbon suspension, and it was verified with an optical pyrometer that the heating temperature and the sample surface temperature could differ by approximately 50 °C. For the AlSi9Cu3(Fe) alloy, at a heating temperature of 550 °C, the dissolution of CuAl₂ was observed. During exposure at a heating temperature of 900 °C for the Ti6Al4V alloy, it was possible to observe the transformation of the globular α phase with an HCP lattice to the β phase with a BCC lattice. While a transformation to the α phase was expected during cooling, there was no observed change in morphology. Artificial aging of the CuBe2 alloy occurred at a temperature of 315 °C (Fig. 1). Precipitation of the γ phase likely began before reaching the selected temperature. During exposure, the precipitated particles slightly increased in size. Apart from precipitation, the contrast at the boundaries of some grains began to increase, resulting in their expansion. In the case of artificial aging of the AlMgSi0.5 alloy at a heating temperature of 180 °C, no structural changes were observed. The feasibility of in situ thermal etching was confirmed on ZrO₂ ceramic. The sample was imaged at room temperature and then continuously during exposure to heat. During heating, unstable behavior was observed due to sample charging, the evaporation of water from the carbon suspension used to adhere it to the platinum pad, and thermal expansion. Upon reaching a heating temperature of 750 °C, the contrast of grains in both detectors began to increase slightly. There was no sample charging. Already at a heating temperature of 900 °C, the contrast of grains substantially increased, remaining largely unchanged throughout the exposure period for both detectors.

The in situ SEM experiments at elevated temperatures were successfully conducted, demonstrating the potential applications of this method in materials science. During the experiments, partial insights were gained, and practical procedures were developed, leading to greater validity of the results. The window for pyrometer measurements allowed for further refinement of surface temperature measurements. Direct temperature measurement on the sample may lead to further precision. The carbon suspension for high temperatures improved heat conduction and charge dissipation from the sample. Under specific conditions, samples can also be melted. Thanks to special metallographic preparation, it was possible to observe the real structure already at room temperature; however, further experiments are needed to verify the introduction of artifacts from the preparation process.

Graphic:

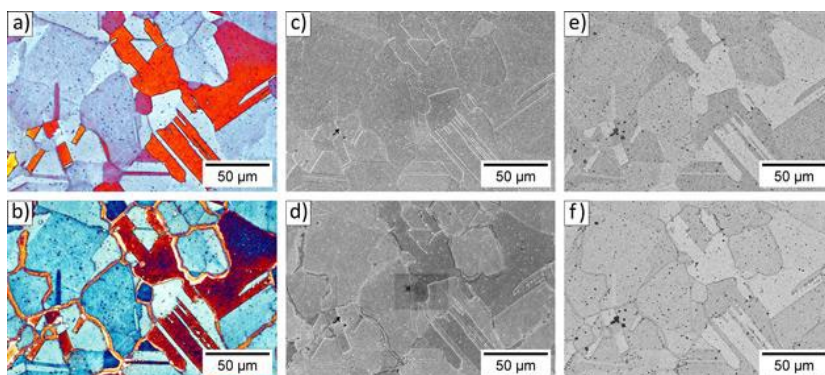


Fig. 1 CuBe2 alloy artificial aging at 315 °C; a) LOM before aging; b) LOM after aging; LOM 500× (cropped); c) SEM SE ETD before aging; d) SEM SE ETD, 315 °C, 2.7 h; e) SEM BSE Karmen before aging; f) SEM BSE Karmen, 315 °C, 2.7 h; SEM 1000×, 0,09 nA, 10 kV, WD 18 mm; color etched Klemm I.

Keywords:

in-situ SEM, correlative-microscopy, heat-treatment, thermal-etching

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