doi:10.1017/S1431927611003023
Microsc. Microanal. 17 (Suppl 2), 2011

Heating Experiments in the Scanning Electron Microscope

C.C. Appel, C. Bøgvad

Haldor Topsøe A/S, Microscopy Department, Research and Development Division,
DK-2800 Lyngby, Denmark

Heating stages developed for the Environmental Scanning Electron Microscopes (ESEMs) make it possible to investigate changes in surface structure in situ at temperatures up to 1500˚C. The ability of the ESEM to work with up to 20 torr pressure in the specimen chamber makes it possible to study solid gas reactions in various gas compositions. The gas may fill the whole specimen chamber or the reaction gas may only be introduced locally close to the specimen surface. This gives flexibility in choice of experimental parameters. In situ observation during heat treatment in ESEM is not a method for obtaining quantitative information at conditions comparable to actual conditions because there are several restrictions on this technique: Image acquisition is determined by the scan rate, actual temperature is difficult to determine, often pressure has to be kept considerably lower than during the actual heating process, and the effect of the electron beam and the positively charged ions present at the surface of the investigated sample must be considered. Despite limitations of the technique, ESEM is an excellent method to observe local structure changes on surfaces during heating.

ESEM investigations of samples held at elevated temperatures have been used to study oxidation of metals and alloys, corrosion at high temperature, and interdiffusion between solid phases. Interdiffusion is not possible to study directly in situ in cross sections of the reacting materials because surface diffusion will be dominating in the exposed surface of the cross section. For investigation of interdiffusion between Ni and Sn an approximately 5 μm thick layer of Sn was electrolytically applied on Ni. The surface of the Sn coating was studied during heating in ESEM at temperatures between 200 and 850°C. A modified heating stage [1] with a hollow crucible and reaction gas lead through the bottom of the heating stage was used. To avoid oxidation during heating a mixture of 93% Ar and 7% H_2 was used as reaction gas. The microscope chamber was filled with N_2 to improve the image quality. The original Sn-surface was structureless, therefore a dark easy recognizable feature was placed in the center of the area, selected for investigation. One example of a heating sequence (Fig. 1) shows a surface where holes are formed in the Sn-surface around 200°C. This is close to the melting temperature of Sn which is 231.15°C. The sample was held at 195°C for 35 minutes and then further heated to 250°C where it stayed for another 45 minutes. During the last part of the heat treatment more contrast appeared from the surface and it is clearly seen that a topographical relief was created indicating that the surface is solid and not liquid as Sn is expected to be at 250°C.

The Ni-Sn two component phase diagram [2] shows that three stable Ni-Sn-phases may be formed at atmospheric pressure, Ni_3Sn_4, Ni_3Sn_2, and Ni_3Sn. Ni_3Sn_4 is only stable up to 794.5˚C. Diffusion studies in the two component system Ni-Sn have earlier shown that Ni diffuses rapidly through the Sn-liquid and Ni_3Sn_4 crystallizes from the liquid phase [3]. In the in situ experiment the holes formed at 195°C persisted through the remaining part of the heating program. This shows that Ni diffusion in Sn is so fast that formation of liquid Sn is prevented. Energy Dispersive X-ray Spectrometry (EDS) across a section of the sample after the heating experiment shows that Ni has diffused through the whole Sn-layer. The Ni/Sn-ratio in the layer corresponds to that of Ni_3Sn_4.

The observations show that in situ SEM heating is an excellent tool to study the phases and structures formed while the heating experiment is carried out. However, the actual temperatures, diffusion coefficients, and concentration variations have to be determined by other characterization techniques.

References

[1] C.C. Appel et al., *ICEM-15* (2002) 227.
[2] ASM Handbook, Volume 3 “Alloy Phase Diagrams”. ASM International, Materials Pak, Ohio, USA (1999).
[3] S. Bader et al., *Acta Metall. Mater.* 43 (1995) 329.
[4] Sven Ullmann is gratefully acknowledged for his assistance during heating experiments

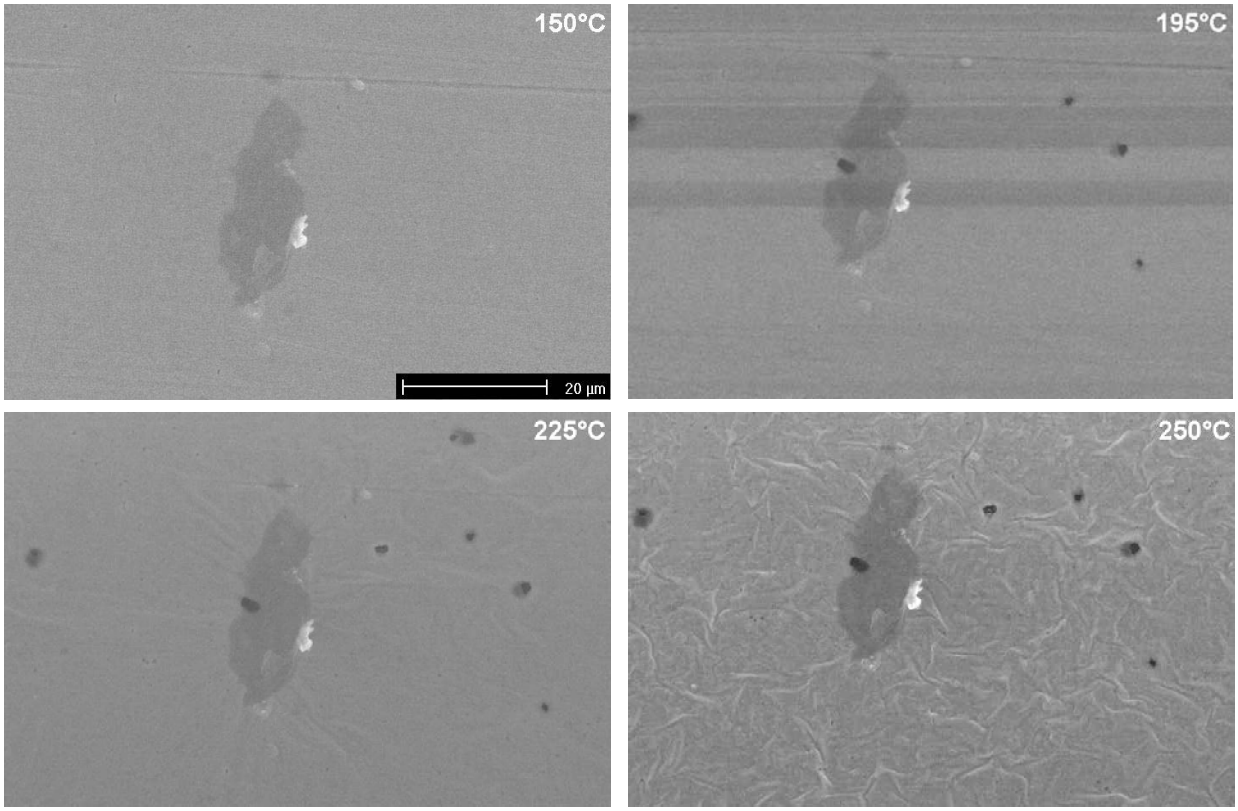

FIG. 1. Selected images of an area on a Sn surface on a Ni substrate during in situ heating. The scale bar on the upper left image is valid for all the images. The noise on the image at 195°C is due to appearance of holes in the surface.

https://doi.org/10.1017/S1431927611003023 Published online by Cambridge University Press