

In-situ EBSD Phase Transformation and Recrystallisation

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Abstract. With the advent of in-situ heating stages that can fit into SEM's and the combination with EBSD, it is now possible to directly observe phenomenon such as phase transformations and recrystallisation at high spatial resolution and to link these processes to microstructural parameters.

This presentation will report some results from preliminary in-situ EBSD heating experiments conducted in an SEM on the transformation of ausenite to ferrite in a plain carbon steel and recrystallisation in bronze alloy strip cast on a steel substrate. The microstructural changes observed during these experiments will be reported in terms of EBSD maps, grains size and crystallographic texture that evolves during a) a heating cycle from ferrite to austenite and cooling to ferrite and b) the recrystallisation microstructure for bronze and steel during isochronal heating.

1. Introduction

In-situ heating stages for SEM's with conventional wire wound heaters are too large to be tilted to 70 degrees or allowing shorter working distances. Consequently, for EBSD analysis most heating studies have been performed in the past [1,2], using dedicated Scanning Electron Microscopes (SEM) and heating stages. In addition, such stages may also lead to a lot of sample drift during analysis and also usually restrict the user to low heating rates.

A new heating stage developed by Gatan UK, shown in Figure 1, was used in this study. This stage has a very compact design allowing it to be used in almost any SEM. The compactness of this stage is due to its patented doped-Si wafer heater. It can be used in combination with EBSD analysis, where the stage can be tilted to 70 degrees while maintaining working distances as short as 12mm. It also has very stable high heating rates and sample drift is minimal. Furthermore the control thermocouple position allows very tight control and accurate sample temperature measurements. The heater also has a rapid heating rate, such that its maximum temperature of 950 °C can be reached in a few seconds.

The EBSD analysis was performed using the Oxford Instruments EBSD system. Heating above 400°C results in infra-red (IR) radiation which can disrupt EBSD analysis. However, this problem can be eliminated by using an IR filter in the EBSD detector, as implemented in the Oxford Instruments NordlysMax detector. Dynamic measurements such as in-situ heating are preferably done at high EBSD acquisition speeds in order to effectively capture rapid events that occur during heating.





Figure 1) In-situ heater at 70° stage tilt during EBSD analysis sample heated to 750°C.

2. Results and Discussion

The new heater was used to study the recrystallisation behaviour of a deformed bronze strip cast on a ferritic steel substrate system and the phase transformation of austenite to ferrite in a low carbon steel sample.

2.1. In-situ heating study of recrystallisation

It is usually very difficult to establish the nucleation sites of recrystallisation from the results of conventional ex-situ post-mortem studies nor is it easy to show the effect of interface such as steel and bronze on the recrystallisation.

The results from a successful in-situ heating with EBSD analysis are shown here from a steel-bronze system. A small sample size of about 2mm x 2mm x 2mm was used to have a very rapid response with good temperature control from the heater. The chamber was purged with nitrogen to reduce effects of surface oxidation.

Figure 2a shows the sample heated to 520 °C where there is evidence of nucleation sites for recrystallisation in bronze. It is apparent that as expected the nucleation starts at grain boundaries in the bronze and not at the steel interface [3].

Figure 2b shows that at 680 °C the steel goes through a sudden recrystallisation, whereas the bronze is partially only recrystallised. The bronze starts to recrystallise earlier than the steel substrate, however, the recrystallisation rate is much slower in bronze as compared to the steel.

2.2. In-situ heating study of phase transformation

Figure 3 shows the phase transformation from austenite to ferrite in a series of EBSD IPF and phase coloured maps, for a cooling rate of 1 °C per minute between 945 – 895 °C and 895 – 880 °C. Upon reaching 895 °C the temperature was held constant for a period of 6 minutes before cooling was resumed. EBSD data was acquired at 20kV and 870Hz at 1.5µm step size, where each map took 6 minutes. The average hit rate or indexing percentage for the maps was above 80%.

The relatively fine austenite grains of about 20µm are seen to transform to large ferrite grains of about 200µm. (The scale bar in the maps is 200µm)

The high speeds used for the EBSD analysis allowed this investigation to capture the details of the rapid dynamic events during the phase transformation in order to study phase boundary and texture analysis, which will be reported elsewhere.

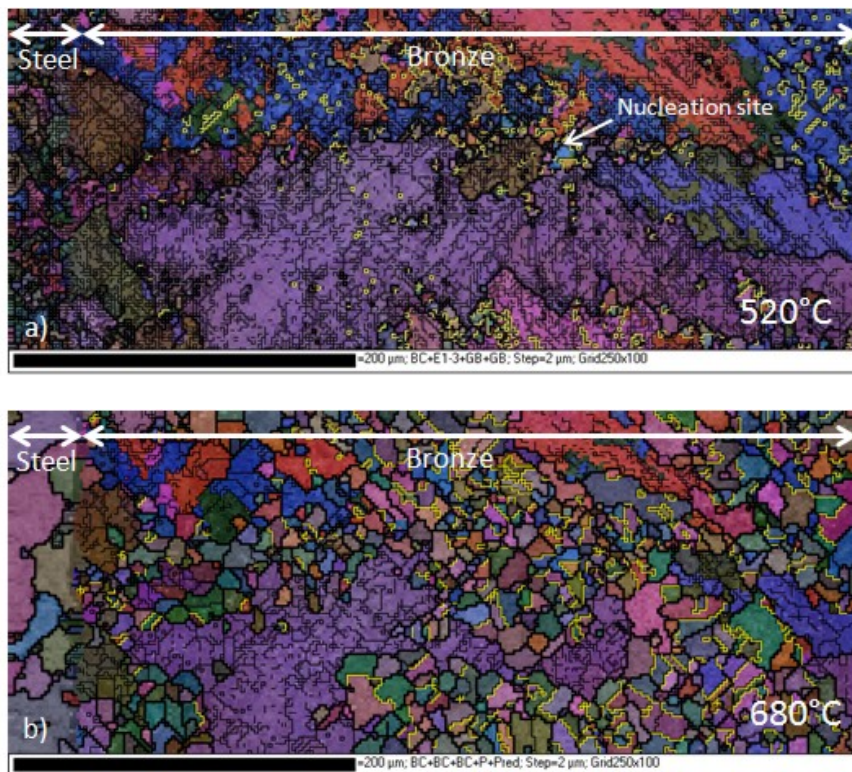


Figure 2) In-situ heating EBSD maps from bronze on a steel substrate in Euler colours where thick dark lines show high angle boundaries, thin dark lines show low angle boundaries and light lines show twin boundaries at a) 520 °C and b) 680 °C.

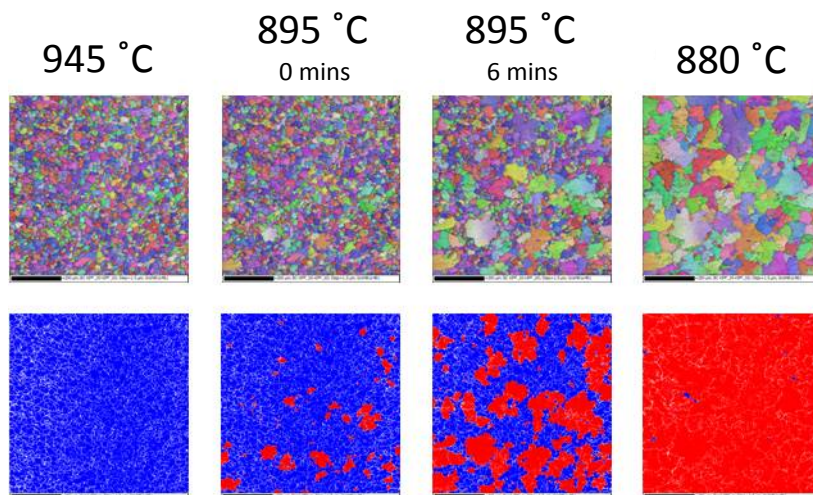


Figure 3) In-situ cooling EBSD maps are shown from 945 °C to 880 °C (for a cooling rate of 1 °C per minute between 945 – 895 °C and 895 – 880 °C). Upon reaching 895 °C the temperature is held constant for a period of 6 minutes, before cooling is resumed. The top row shows the IPF colour maps and the bottom row phase maps where austenite is blue (dark) and ferrite is red (light).

References

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