A TECHNIQUE FOR REAL-TIME, *IN SITU* SEM OBSERVATION OF GRAIN GROWTH AT ELEVATED TEMPERATURES

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INTRODUCTION & ABSTRACT

The marketing hype for the Electroscan/Phillips/FEI Environmental Scanning Electron Microscope (ESEM) promised microscopists a new era of imaging hot specimens with few constraints. In practice, imaging hot specimens was very limited from the perspective of grain growth studies. This poster introduces the techniques and tools developed to make grain growth studies in the ESEM a reality and how these could be applied to any hot-stage SEM. The accompanying paper describes the technique more fully.

THE PROBLEM

SEM Grain growth studies need crystal orientation-sensitive contrast.

This requires backscattered (or "forward scattered") electrons.

Only these electrons interact with the crystal structure and carry information from it.

The ESEM is only capable of hot imaging in ESEM mode and fitted with the hot-stage specific detector.

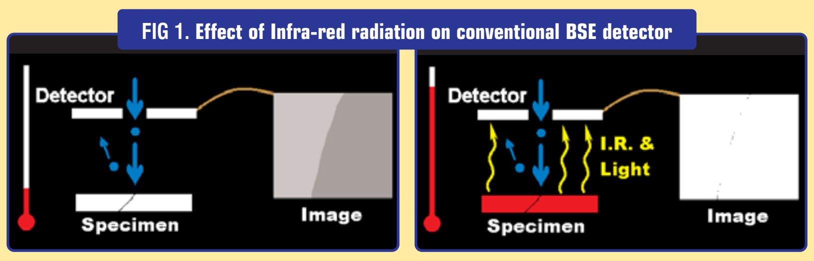
This detector is designed to minimise the contribution from "undesirable" backscattered electrons. It is therefore unable to give satisfactory grain orientation contrast.

Backscattered electron (BSE) detection is by a conventional SEM solid-state backscatter detector.

This solid-state detector has the same limitations as all solid-state detectors – sensitivity to light/infra red and vulnerability to heat damage (Fig 1).

It is incapable of imaging very hot specimens – the electron signal is flooded out by photons from the specimen.

FIG 1. Effect of Infra-red radiation on conventional BSE detector



THE SOLUTION

- Must detect backscattered electrons but not heat or photon radiation
- Must be sensitive enough to discriminate the relatively weak orientation contrast

The converter plate was originally developed by Moll *et al* [1] and Reimer *et al* [2]. It was soon largely obsolete due to cheap solid-state detectors. A plate with a high secondary electron yield coating is placed where it will be struck by backscattered electrons from the specimen.

The electrons striking the plate will generate additional secondary electrons (SE3 in Fig. 2).

These SE3 can be detected by an SE detector or ESEM detector – but they carry the BSE contrast information.

This conversion operates irrespective of photon radiation and temperature – the plate and coating can be made from heat-resistant materials.

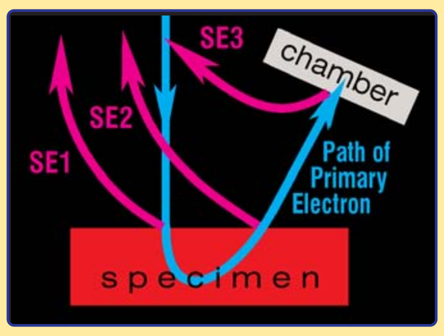


FIG 2. Origins of type 1, 2 & 3 Secondary Electrons

GRAIN ORIENTATION CONTRAST

Some electrons entering a crystal will backscatter, typically 10-30%.

This proportion, η, is the backscattering coefficient – determined primarily by electron energy and atomic number of the material (hence atomic number contrast).

The value of η is modified by factors such as:

- angle between incoming beam and local surface (giving topographic contrast)
- angle between incoming beam and certain critical zone axes in the specimen crystal (giving orientation contrast).

The maximum possible change in η across all orientations of a target crystal lattice is $\Delta\eta$ – typical values for most materials lie in the range 1-2% (5% at best) [3].

This change is small compared to the changes caused by atomic number or topography effects – the specimen must be flat & homogenous.

Weak contrast requires high-efficiency detection and high beam current for fast scan times.

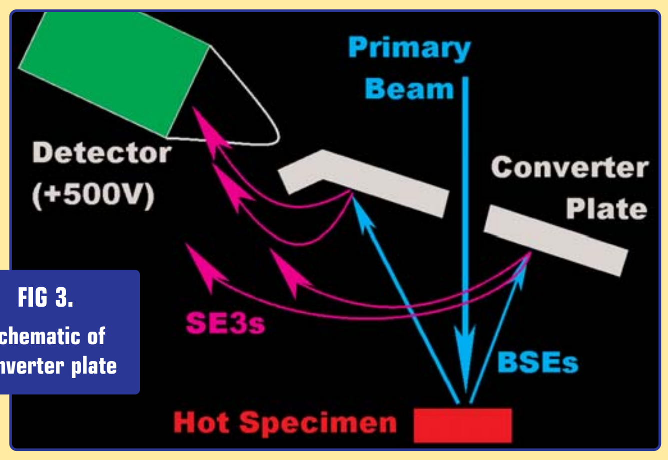


FIG 3. Schematic of converter plate

OPTIMISATION

The plate can be placed between the specimen and the SE detector to help shield the detector from photon radiation (Fig. 3). It can also function as a heat shield for other parts of the microscope.

Maximum detection efficiency requires maximum detector angle – the ideal is a hemispherical "plate", covering all angles at which an electron could exit the specimen [4]. In practice, holes must be provided for the beam and for electrons to pass through to the SE detector.

The coating material of the plate must give a high SE yield. Magnesia and carbon are recommended by the literature.

The specimen continues to emit its usual strong SE1+SE2 signal. It is desirable, but not essential, to suppress these signals by biasing or using a biased grid or electrode.

RESULTS

The converter plate has given good images from hot specimens of steel (Figs. 4, 5 & 6), aluminium (Fig. 7), gold (Fig. 8) and electronic material. At a resolution of approximately 700 by 500 pixels (NTSC video format), frame rates can be as low as 1 per second for gold and steel specimens; longer for low atomic number specimens. Video sequences have been recorded which clearly show the kinetics and dynamic behaviour of many of the phenomena of recrystallisation, grain growth [5] and phase transformation.

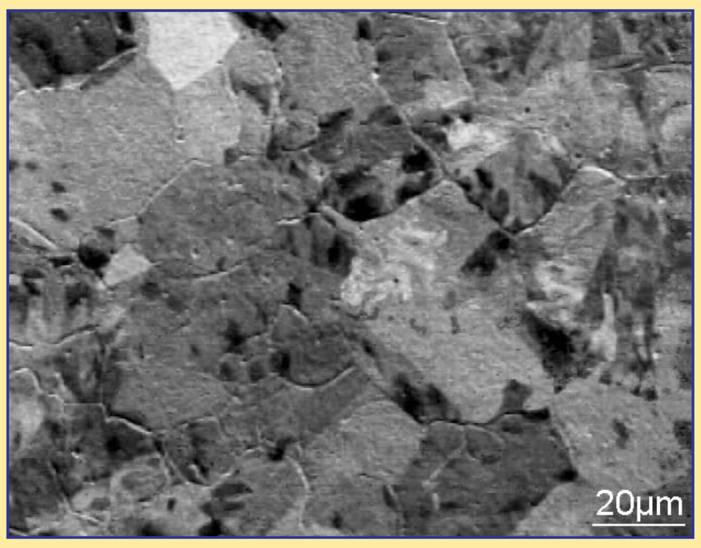


FIG 6. Several pearlite transformation fronts sweeping through an austenite structure, also showing surface carbide precipitation.

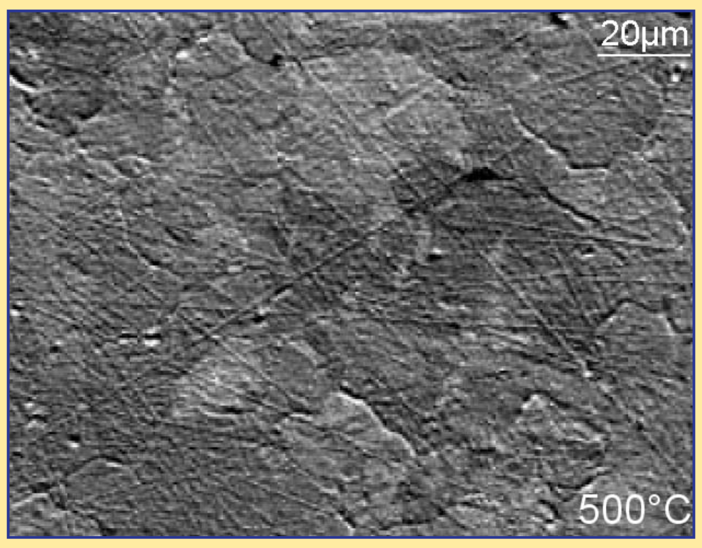


FIG 8. Grains growing in silver/gold alloy at 500°C

CONCLUSIONS

The converter plate has removed the detector as the limiting factor in hot stage microscopy for grain growth studies. The limiting factor will now be hot stage capability and the need to protect other microscope components from heat and contamination.

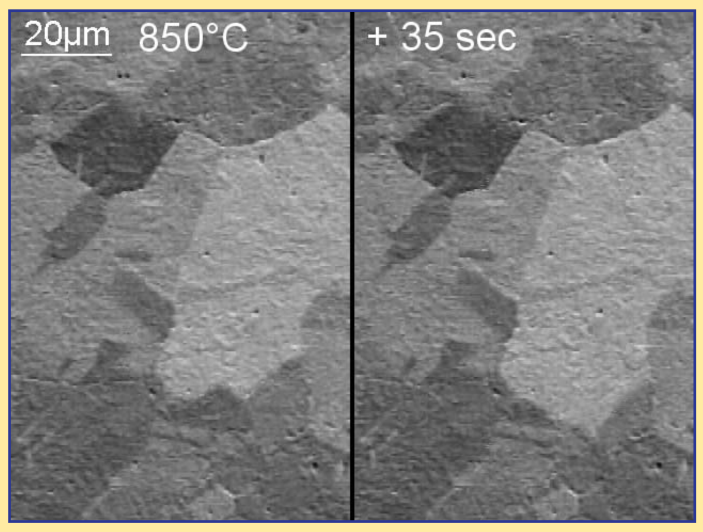


FIG 4. Grains growing in 0.61% carbon steel at 850°C

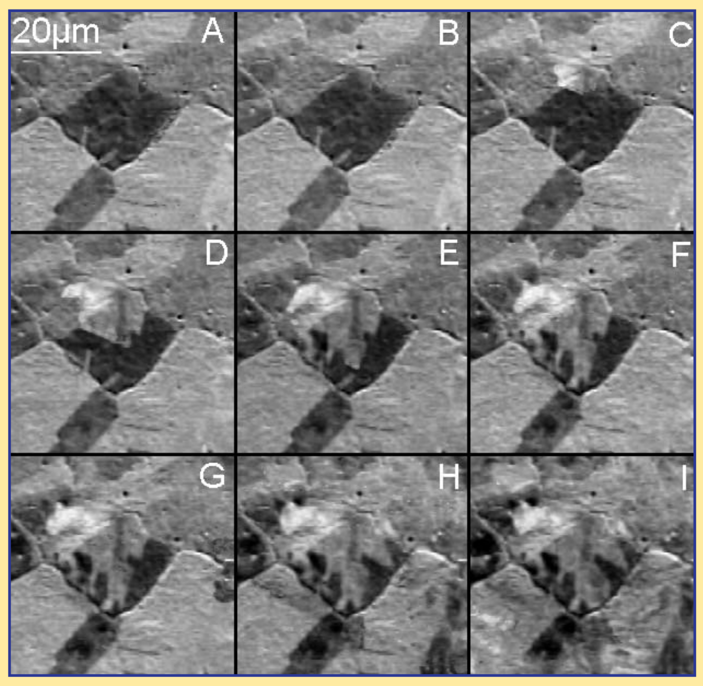


FIG 5. Austenite transforming to pearlite, images A-I at 20-second intervals, 660°C

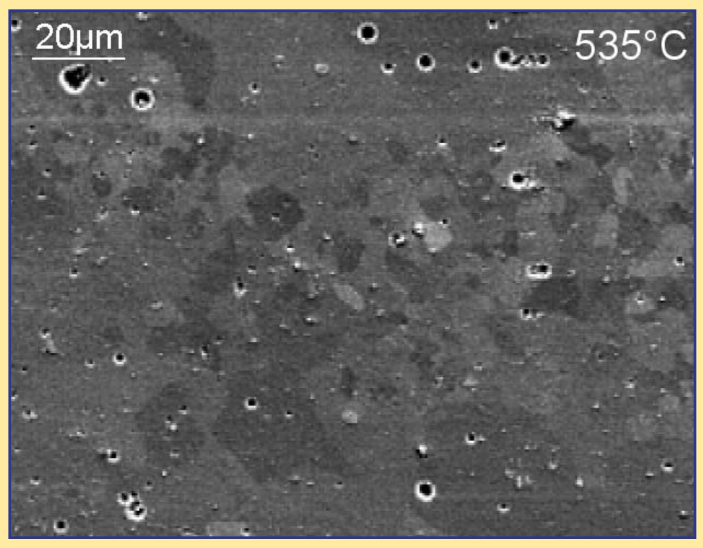


FIG 7. Grains growing in 1050 aluminium at 535°C

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