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Monte Carlo modeling of cavity imaging in pure iron using back-scatter electron scanning microscopy

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ABSTRACT

Backscattered electrons (BSE) in a scanning electron microscope (SEM) can produce images of subsurface cavity distributions as a nondestructive characterization technique. Monte Carlo simulations were performed to understand the mechanism of void imaging and to identify key parameters in optimizing void resolution. The modeling explores an iron target of different thicknesses, electron beams of different energies, beam sizes, and scan pitch, evaluated for voids of different sizes and depths below the surface. The results show that the void image contrast is primarily caused by discontinuity of energy spectra of backscattered electrons, due to increased outward path lengths for those electrons which penetrate voids and are backscattered at deeper depths. Size resolution of voids at specific depths, and maximum detection depth of specific voids sizes are derived as a function of electron beam energy. The results are important for image optimization and data extraction.

1. Introduction

Microstructural changes and mechanical property degradation of reactor structural alloys under neutron damage in reactors present large challenges for reactor performance and safety [1–3]. Since its first observation in 1967 [4], neutron-induced void swelling in stainless steels especially has been a subject of intensive research, as swelling leads to significant dimensional instabilities via swelling and irradiation creep, with concurrent changes in mechanical and physical properties and also in new modes of embrittlement [2–8]. Swelling and bubble formation (both hereafter referred to as cavities) induced by charged particle irradiation is also being increasingly studied as a surrogate for neutron irradiation [9–18]. Void swelling is caused by incomplete interstitial-vacancy recombination and biased defect-sink interactions during displacive irradiation by neutrons or charged particles, and its general dependence on damage dose defined in displacements per atom (dpa), dpa rate, temperature, and stress are relatively well known [1–3].

Characterization of cavities is primarily accomplished using transmission electron microscopy (TEM) which requires electron-transparent specimens of 50 nm–200 nm thickness, prepared using either electrochemical polishing, ion milling, or focused-ion-beam (FIB) lift out techniques. For neutron-irradiated materials, TEM specimen preparation involves creation of radioactive waste, posing safety, disposal and cost concerns, especially for smaller research entities such as universities.

An alternative non-destructive examination approach to cavity characterization can be realized using backscatter electrons (BSE) in scanning electron microscopy (SEM). This technique has the advantage that a very large area of a specimen can be characterized, significantly increasing the measured volume compared to techniques involving transmission microscopy. The SEM-BSE technique has been used previously to detect and quantify surface-connected cavities in nuclear fuel, shale, ceramic coating on dental implant, integrated circuits, but also to measure near-surface cavities covered with metallic films [19–24]. Most recently, the technique has been used to detect and quantify subsurface cavities in neutron-irradiated steels by Pastukhov and coworkers [25]. The potential for such quantification is shown in Fig. 1, where a complex Russian alloy is exhibiting very heterogeneous void swelling imaged with BSE that would be difficult to characterize by transmission microscopy alone.

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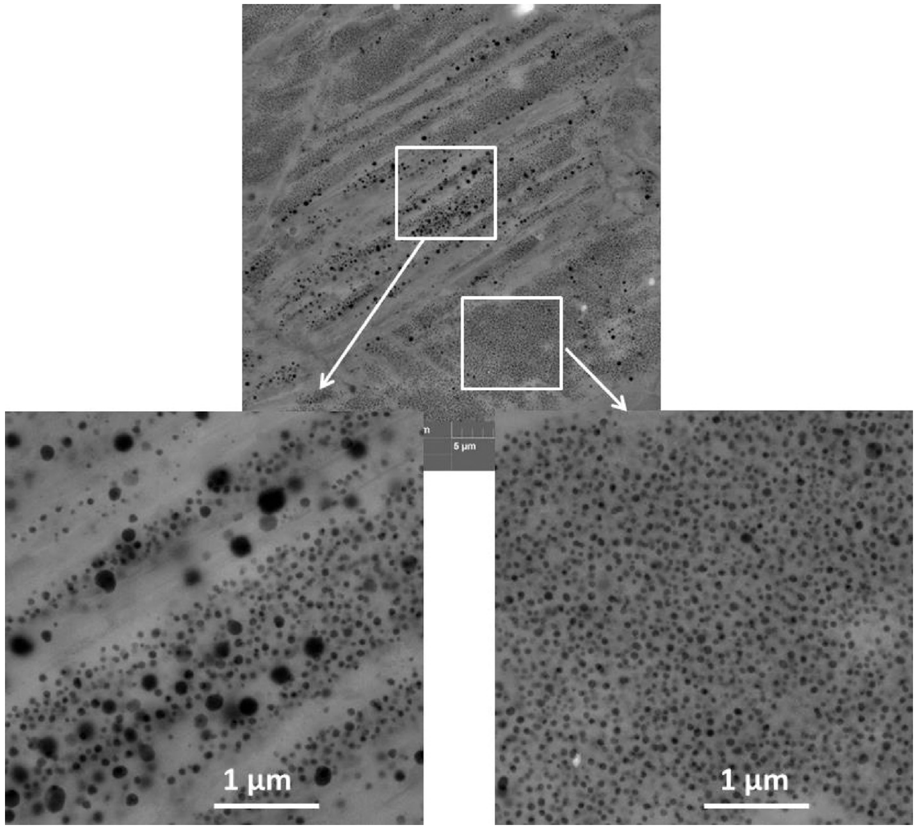

Fig. 1. A SEM-BSE image obtained by using 30 keV electron analysis beam and from Russian EK-164 fuel cladding irradiated to 82 dpa at 515 °C. These micrographs were supplied by Vladimir Pashtukov of the Institute of Nuclear Materials [25].

While the study that produced the micrographs discussed above has demonstrated the feasibility of the SEM-BSE technique and has begun to explore experimentally the combination of materials, specimen preparation procedures, detector type and configuration, and beam characteristics that influence cavity imaging, additional studies are required to better define the physical fundamentals of image generation, and to guide future experimental efforts to better define choices of parameters that determine resolution, sensitivity, and limitations of the SEM-BSE technique.

In this study, Monte Carlo modeling is used to understand the mechanism of image formation, and to identify key factors that influence cavity imaging. The derived knowledge can be applied to better optimize the conditions for best imaging.

2. Modeling procedure

This study used the CASINO Monte Carlo simulation code [26–30] to obtain backscattered-electron (BSE) images of cavities under different configurations and beam conditions The code has been widely used to simulate electron trajectories in solids, often needed for SEM imaging or electron lithography [30]. The code is based on the binary collision approach with electron-atom scattering described by tabulated Mott cross sections. The electron energy loss is given by Ref. [31].

$$\frac{dE}{ds} = -7.85 \times 10^4 \frac{\rho Z}{EM} \ln\left(\frac{1.166E}{J^*}\right) \quad (\mathrm{KeV/cm}) \tag{1}$$

where J^* is the modified ionization potential, given by

$$J^* = \frac{J}{1 + k\frac{J}{E}}, \text{ where } J = \begin{cases} 11.5Z (Z < 13) \\ 9.76Z + 58.5Z^{-0.19} (Z \geq 13) \end{cases} \tag{2}$$

Z is the element number of the target atom. Eqs. (1) and (2) are known to be valid to describe electron-solid interactions to energies as low as 100 eV.

The code uses tabulated values from ELSEPA (Dirac partial-wave calculation of elastic scattering of electrons and positrons by atoms, positive ions and molecules) cross-section to calculate elastic collisions, which involves the calculation of the relativistic partial-wave for scattering [32]. Modeling-obtained backscattering efficiency—the ratio of backscattered electron number to incident electron number—agrees with experimental results [30].

The present study simulated BSE images arising from pure iron, which was selected as a model substrate to represent stainless steels currently being investigated as fuel cladding materials for fast reactors. The modeling considered different electron-target configurations in order to evaluate the resolution and limitation of cavity imaging by the BSE technique. The electron beam energies range from 0.5 keV to 40 keV. The beam spot sizes range from 1 nm to 10 nm. Cavities are introduced at different depths, with their sizes ranging from 5 nm to 60 nm. Furthermore, a comparison is made among bulk Fe and Fe membranes of 100–200 nm thickness, since the latter represent typical electron-transparent TEM specimens.