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ENERGY-FILTERED PLASMON IMAGES OF $MgAl_2O_4$ IMPLANTED WITH Al^+ AND Mg^+ IONS

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Magnesium aluminate spinel ($MgAl_2O_4$) is a candidate material for specialized applications in proposed fusion reactors, and previously, has been irradiated with Al^+ or Mg^+ ions to assess the effects of high-dose irradiation. Electron energy-loss spectrometry (EELS) has been used to confirm the identity of metallic aluminum colloids located in the ion-implanted region of the spinel because electron diffraction experiments were inconclusive for phase identification.[1] In the present study, energy-filtered plasmon images of the ion-implanted region have been obtained to reveal this colloid distribution.

Following implantation with 2 MeV Al^+ ions to a fluence of 3.8×10^{21} ions/m^2 at 923 K, or with 2.4 MeV Mg^+ to a fluence of 2.8×10^{21} ions/m^2 at room temperature, spinel specimens were prepared in cross-section for analytical electron microscopy.[2] Energy-filtered images were obtained using a Philips CM30 microscope with an attached Gatan Imaging Filter. Acquired images were 512 × 512 pixels in size and gain normalized. Filtered images were recorded with 5-eV-wide windows and 1 s exposure times; windows were centered about 0, 10, 15, 20, and 25 eV losses. Images acquired using 15 eV loss electrons (I_B) contained contributions from both spinel matrix, I_s, and metallic colloids, I_m, and $I_B \sim I_s + I_m$. For some images, an estimate of I_s was subtracted from I_B to improve the contrast between the colloids and the matrix. This background subtraction of the spinel contribution was accomplished by estimating $I_s \sim I_A + (I_C - I_A)/\mathbf{n}$, where I_A and I_C were images with losses centered at 10 and 20 eV, respectively, and **n** is a suitable weighting factor (typically $2 \leq \mathbf{n} \leq 3$).[3]

Energy-filtered bright-field images were acquired from both Al^+ and Mg^+ ion-implanted spinel. These images appear similar to conventional bright-field images, and show little diffraction contrast between the spinel matrix and the colloids. A filtered bright-field image of the Mg^+ implanted spinel is shown in Fig. 1. Figures 2, 3, and 4 are energy-filtered images of the same region shown in Fig. 1. Figure 2 was acquired with loss electrons corresponding to the spinel volume plasmon (~25 eV), so the continuous spinel matrix appears bright. Because the zero-loss electrons have been attenuated by the filter, the hole in the specimen appears dark at the top of the figure. The dark features in the spinel image, which are complementary to bright features in Figs. 3 and 4, are metallic aluminum colloids. Figure 3 was acquired with 15-eV-loss electrons corresponding to the metallic aluminum volume plasmon. In this figure, the colloids in the implanted-ion region have the greatest intensity, although there is significant intensity from the spinel matrix. This is expected as the spinel volume plasmon can be excited over the loss range of ~10-40 eV. However, with the spinel background subtracted from Fig. 3, the contrast between the colloids and the spinel matrix is improved (Fig. 4). Similar features were observed in the spinel implanted with Al^+ ions. Presently, the background substraction routine assumes the weighting factor **n** is constant everywhere in the whole image. A possible further improvement in background subtraction could be made by allowing **n** to vary with thickness (position) in the image. A suitable routine for accomplishing this is being examined. Small colloids with diameters ~2 nm are clearly resolvable near the edge of the Mg^+ (Fig. 5) and the Al^+ implanted-ion regions (Fig. 6), respectively. Because of delocalization effects, the "chemical" resolution in a volume plasmon image has been suggested to be of the order of 10 nm.[4] However, an advantage of energy-filtering is clearly demonstrated here: very small features, similar to the matrix in chemistry but having different volume plasmon spectra, can be imaged in non-specific diffracting conditions.[5]

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2. N.D. Evans et al., in *Microstructure of Irradiated Materials, Mat. Res. Soc. Symp. Proc.* 373 (1995) in press.
3. N.D. Evans et al., *Proc. 29th Ann MAS Meeting* (1995), to be published.
4. R. F. Egerton, *Electron Energy-Loss Spectroscopy in the Electron Microscope*, New York: Plenum Press (1986)306.
5. Research sponsored by the Division of Materials Sciences and Office of Fusion Energy, U.S. Department of Energy, under contract DE-AC05-84OR21400 with Martin Marietta Energy Systems, Inc., and through the SHaRE Program under contract DE-AC05-76OR00033 with Oak Ridge Associated Universities.

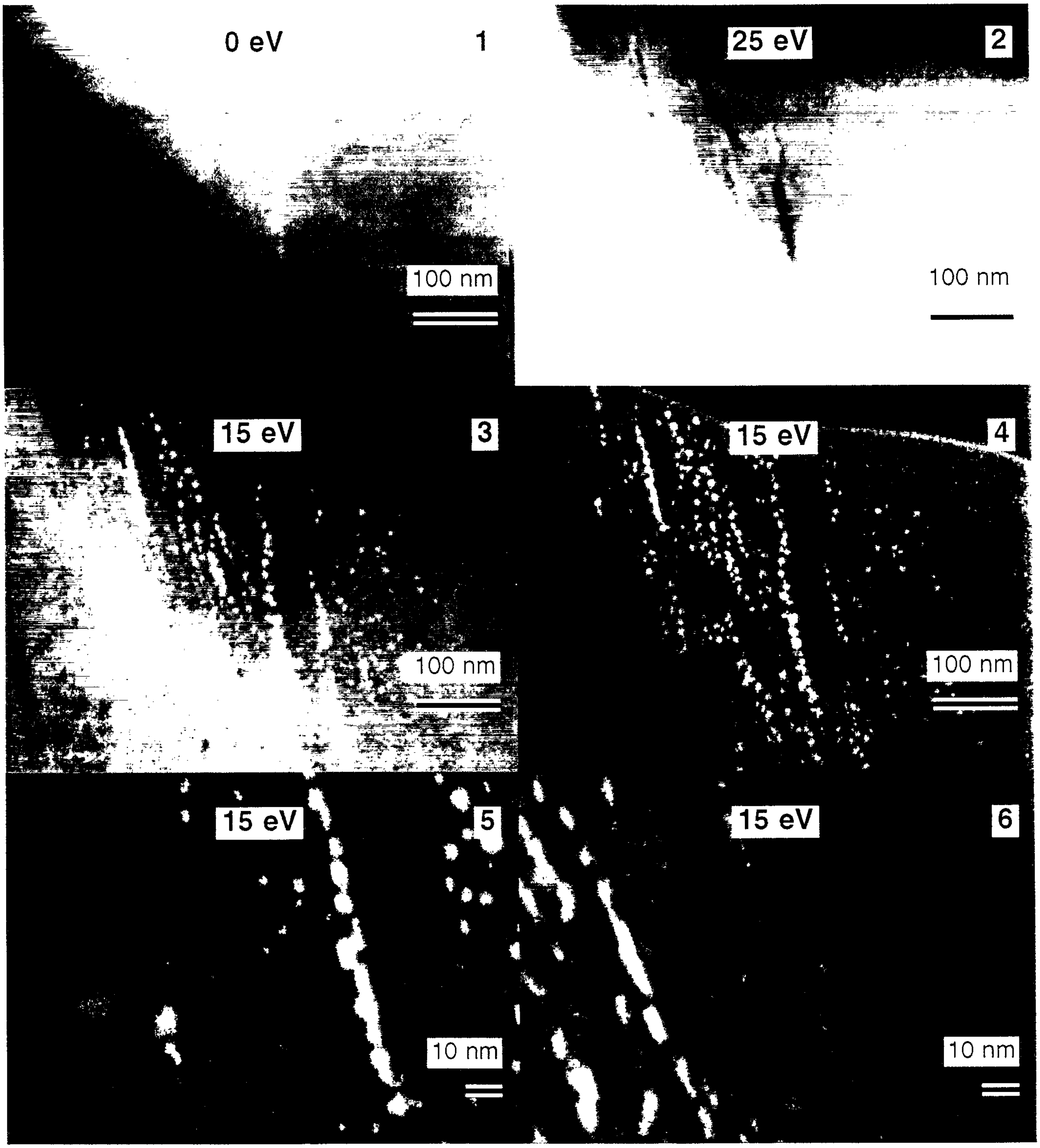

FIG. 1.--Energy-filtered bright-field image of implanted Mg^+ region of $MgAl_2O_4$ spinel.
FIG. 2.--25 eV loss plasmon image, implanted Mg^+ region.
FIG. 3.--15 eV loss plasmon image, implanted Mg^+ region.
FIG. 4.--Background subtracted 15 eV loss plasmon image, implanted Mg^+ region.
FIG. 5.--nanometer-sized metallic aluminum colloids in Mg^+ implanted spinel.
FIG. 6.--nanometer-sized metallic aluminum colloids in Al^+ implanted spinel.

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