

A BACKGROUND SUBTRACTION ROUTINE FOR ENHANCING ENERGY-FILTERED PLASMON IMAGES OF MgAl_2O_4 IMPLANTED WITH Al^+ AND Mg^+ IONS

N. D. Evans⁺, E. A. Kenik^{*}, J. Bentley^{*}, and S. J. Zinkle^{*}

⁺Oak Ridge Institute for Science and Education, P.O. Box 117, Oak Ridge, TN 37831-0117

^{*}Metals and Ceramics Division, Oak Ridge National Laboratory, P.O. Box 2008, Oak Ridge, TN 37831-6376

Magnesium aluminate spinel (MgAl_2O_4) is a candidate material for specialized applications in proposed fusion reactors. In a previous study, the spinel was irradiated with Al^+ or Mg^+ ions to assess the effects of high-dose irradiation; electron energy-loss spectrometry and energy-filtered plasmon images of Al^+ and Mg^+ ion-irradiated spinel revealed that metallic aluminum colloids are present in the ion-implanted regions.¹ In this previous study, energy-filtered images acquired using 15-eV-loss electrons, I_B , contained significant contributions from the spinel matrix, I_S , as well as the metallic colloids, I_M . The component I_S was removed from I_B by making a rudimentary approximation of I_S . In the present study, the subtraction of the spinel plasmon component in images acquired using 15-eV-loss electrons is treated in a more detailed manner.

Following implantation with 2 MeV Al^+ ions to a fluence of 3.8×10^{21} ions/m² at 923 K, or with 2.4 MeV Mg^+ to a fluence of 2.8×10^{21} ions/m² at room temperature, spinel specimens were prepared in cross-section for analytical electron microscopy (AEM).¹ Energy-filtered imaging was performed at 300 kV with a Gatan Imaging Filter (GIF) attached to a Philips CM30 AEM. Filtered images of 512×512 pixels (2 \times binning) were recorded with 5-eV-wide windows, 1-s exposure times, and gain normalization; windows were centered about losses of 0, 10, 15, 20, and 25 eV. A zero-loss bright-field image of spinel following Al^+ ion-implantation is shown in Fig. 1. The same region imaged with 15-eV-loss electrons (corresponding to the volume plasmon of metallic aluminum) shows metallic aluminum colloids as small bright features in the spinel matrix, Fig. 2.

Figure 3 shows electron energy-loss spectra from the spinel matrix and from a region having both colloid and matrix contributions; the volume plasmons at ~15 and ~24 eV correspond to metallic aluminum and spinel, respectively. The energy-filtered image in Fig. 2 was acquired using loss-electrons corresponding to window B; the image intensity (I_B) has contributions from both metallic aluminum (I_M) and spinel (I_S), where $I_B \sim I_M + I_S$. Background subtraction of the spinel contribution was accomplished using three images of intensity I_A , I_B , and I_C , acquired with windows A, B, and C as defined in Fig. 3. In the absence of any metallic aluminum, $I_S \sim I_A + (I_C - I_A)/n$, where the pixel-by-pixel weighting factor n is a function of specimen thickness. The factor n would be 2 were the background changing linearly. A map of n for the image in Fig. 2 was acquired from $n = (I_C - I_A)/(I_B - I_A)$ and is shown in Fig. 4. Dark regions in the image (low values of n) correspond to the presence of colloids. From this map, a typical value of n in regions containing only spinel, n_0 , was taken as 2.5. A map of I_S was calculated using n_0 and the subtraction of I_S from I_B produced I_M , the background subtracted 15-eV-loss image shown in Fig. 5. Background subtracted images from the Mg^+ implanted spinel have also been obtained.

Integrated-intensity line profiles were made for the boxes drawn in some figures; intensities were integrated in the direction normal to the major axis of each box. The profile in Fig. 6a is from the n map. Where only spinel is present, values of n are approximately 2.5, accounting for the above assigned value of n_0 . The integration appears to be noisy due to the colloid morphology (they cluster in the direction of integration). Integration through many colloids lowers the local value of n . Furthermore, the broad minimum in the profile corresponds to the volume fraction of colloids present in the spinel going through a maximum. The line profiles in Figs. 6b and 6c are from the boxes within Figs. 2 and 5, respectively. With background subtraction, features can be easily resolved in Fig. 6c, particularly at the edges of the implanted-ion region, that are barely detectable in the profile from same image prior to background subtraction, Fig. 6b. Lastly, the background fitting routine should be improved by using locally appropriate values for n to reflect its dependence on specimen thickness.²

1. N.D. Evans et al., in *Microstructure of Irradiated Materials*, Mat. Res. Soc. Symp. Proc. 373 (1995) in press.
2. 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.

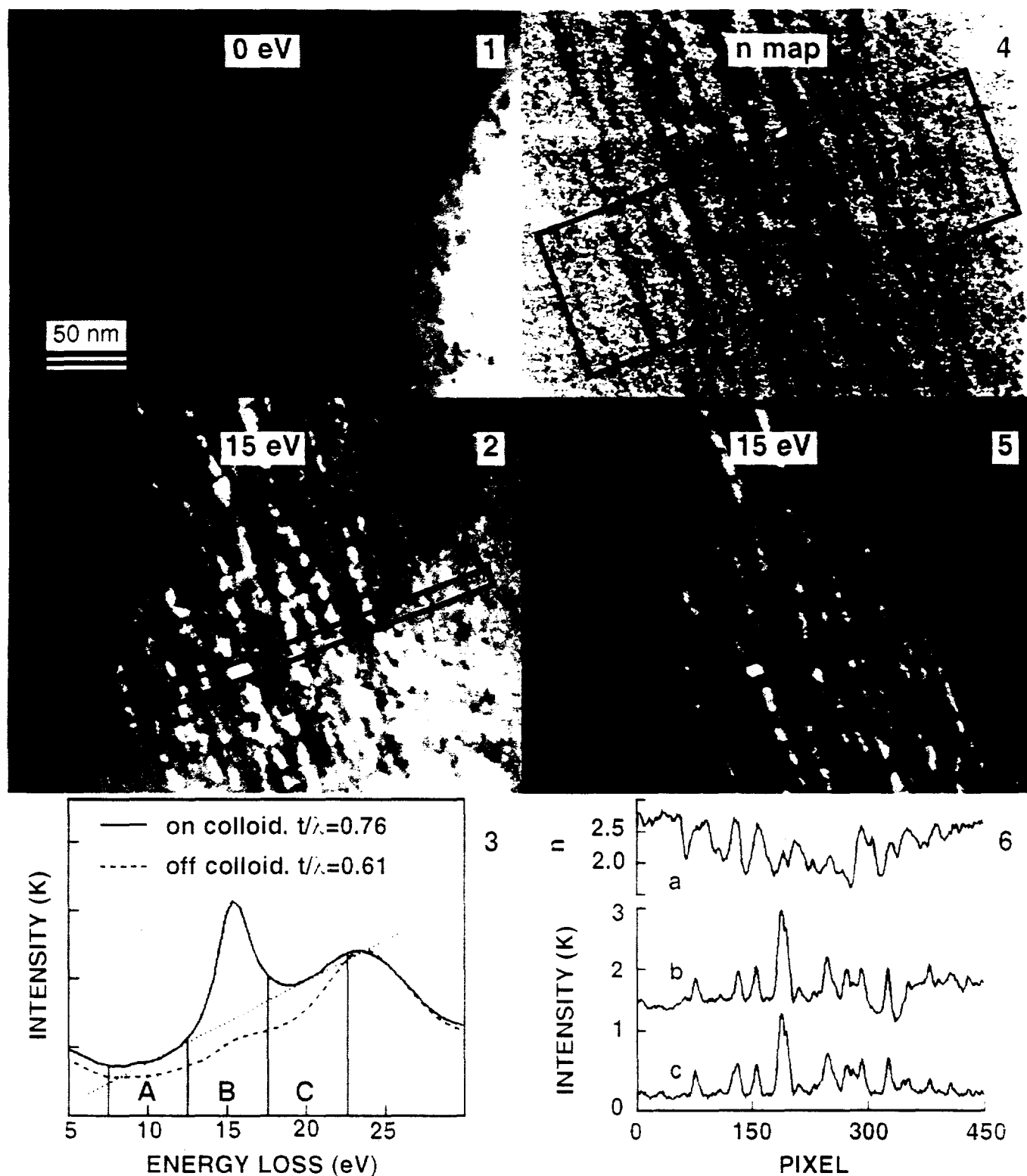


FIG. 1.-- Zero-loss image of Al^+ ion-implanted region of MgAl_2O_4 spinel.
 FIG. 2.-- Energy-filtered plasmon image, without background subtraction.
 FIG. 3.-- Low-loss EELS spectra showing windows defined for energy-filtered plasmon imaging.
 FIG. 4.-- n map.
 FIG. 5.-- Energy-filtered plasmon image, with background subtraction.
 FIG. 6.-- Intensity profiles in boxes defined in (a) FIG. 4, (b) FIG. 2, and (c) FIG. 5.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available to DOE and DOE contractors from the Office of Scientific and Technical Information, 175 Oak Ridge Turnpike, Oak Ridge, TN 37831; prices available at (615) 576-8401.

Available to the public from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161; phone orders accepted at (703) 487-4650.