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TITLE ➡ **EELS OF COLLOIDS IN Mg^+ IMPLANTED $MgAl_2O_4$ SPINEL**

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Because magnesium aluminate spinel ($MgAl_2O_4$) shows a strong resistance to void swelling during neutron irradiation at elevated temperatures, it is a candidate material for specialized applications in proposed fusion reactors. During implantation at 25°C with 2 MeV Mg^+ ions to ~2.8×10^{21} Mg^+/m^2, dislocation loops are formed at midrange depths (~0.5 - 1.0 μm) on {110} and {111}.[1] The microstructure in the implanted ion region (~1.5 - 2.0 μm) is shown in cross-section in Fig. 1. Within this implanted ion region, small features (4 - 10 nm diam.) were observed in dark field (DF) images using a spinel 222 reflection (Fig. 2). No evidence was found in electron diffraction patterns to suggest these features are (hexagonal) metallic Mg. However, in an earlier study, similar features in Al^+ implanted spinel were identified by parallel electron energy loss spectrometry (PEELS) as metallic Al colloids.[2] Phase identification of metallic Al within this spinel by electron diffraction is complicated because the lattice parameter of spinel (0.8083 nm) is almost exactly twice that of aluminum (0.4049 nm) and the phases are oriented cube-on-cube. However, spinel 222 reflections are weak whereas aluminum 111 reflections are intense. The diffracting conditions for Fig. 2 suggest the colloids are either metallic Al or another phase that is coherent with the surrounding spinel.

Volume plasmons from metallic magnesium, metallic aluminum, and spinel are significantly different (Fig. 3). Reference low-loss spectra were obtained from undamaged spinel (well beyond end-of-range), metallic aluminum, and metallic magnesium; relative thicknesses were measured from X-ray spectra acquired simultaneously. Spectra acquired from Mg^+ implanted spinel were separated into components from metal and spinel by linear, least-squares multiple-regression analysis using the reference spectra over an interval from 10 to 40 eV at ~0.3 eV/channel. The standard error of estimate and 95% confidence intervals about the fitting parameters were also determined. A spectrum from the implanted ion region, and a spectrum constructed using the best-fit values (indicating 2.1 ± 0.3 vol.% metallic Al in spinel) from the least-squares multiple-regression analysis are shown in Fig. 3. Agreement between the constructed best-fit spectrum and the spectrum from the implanted region is acceptable; the standard error of the estimate is less than 200 counts. None of the low-loss spectra acquired from the implanted ion region of the spinel exhibited a component characteristic of hexagonal metallic Mg. Spectra were acquired with the specimen at -130°C with a Gatan 666 PEELS and a Philips EM400T/FEG analytical electron microscope operated at 100 kV in the image mode (beam convergence α=2.7 mrad, collection semi-angle β=22 mrad). To avoid possible spurious results, spectra were obtained at high magnification with area selection by a 2 mm spectrometer entrance aperture. No objective aperture was used because of specimen charging and the need to perform simultaneous X-ray microanalysis when acquiring new reference spectra. Specimen thicknesses varied from 20 to 70% of the inelastic scattering mean free path length; all spectra were deconvoluted by the Fourier-log method prior to regression analysis.[3]

Two spectra from the regions indicated (1) and (2) in Fig. 2, but obtained under different (weaker) diffracting conditions, are shown in Fig. 4. Regression analysis with reference spectra from spinel and metallic aluminum indicated the presence of 3.6 ± 0.3 and 0.5 ± 0.1 volume% metallic aluminum in (1) and (2), respectively. This analysis was applied to similar spectra acquired across the implantation range and a profile for metallic aluminum was developed (Fig. 5). These spectra were acquired at high spatial resolution (<40 nm); spectra showing the largest amount of metallic Al correspond to analyzed volumes containing one or more colloids whereas those showing little or no metallic Al were acquired between colloids. The small negative values for metallic Al shown in Fig. 5 are artifacts associated with fitting spectra acquired from regions different in thickness (and in surface plasmon intensities) than that of the reference spectra.[4]

The regression analysis of low-loss spectra and the diffraction data are consistent with the colloids present in the implanted ion region being metallic aluminum. This implies additional Mg is present in the spinel lattice, though substitution of Mg for Al would create a large charge imbalance. Conversely, we cannot rule out the possibility that the colloids are metallic Mg in a metastable cubic structure. Such unusual allotropes have been observed in other instances of ion implantation. This uncertainty is being addressed by an examination of the core-loss near-edge structures from regions typical of (1) and (2) in Fig. 2, and making suitable comparisons to reference spectra.[5]

1. S. J. Zinkle, J. Am. Ceram. Soc., 72(1989)1343.
2. N. D. Evans et al., Proc. Ann. EMSA Meeting 49(1991)728.
3. R. F. Egerton, Electron Energy-Loss Spectroscopy in the Electron Microscope, New York: Plenum Press (1986) 229.
4. N. D. Evans and Z. L. Wang, Proc. Ann. EMSA Meeting 50(1992)1256.

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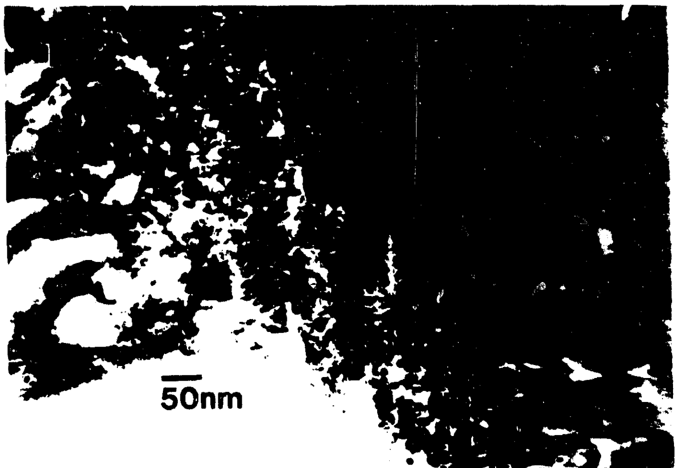

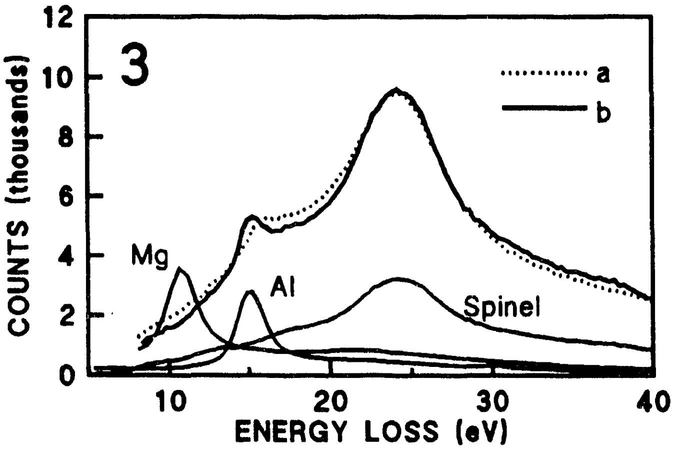

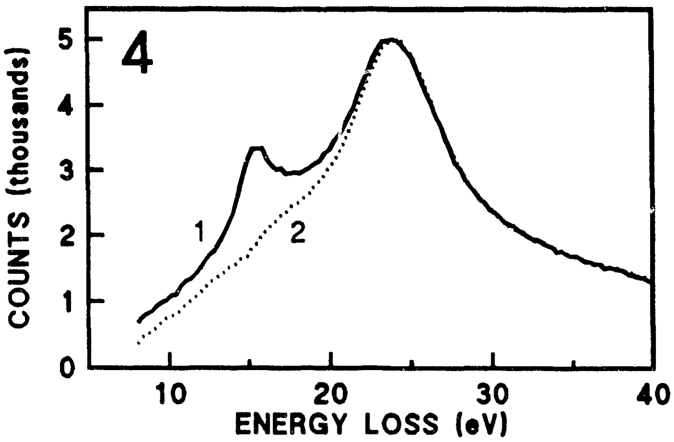

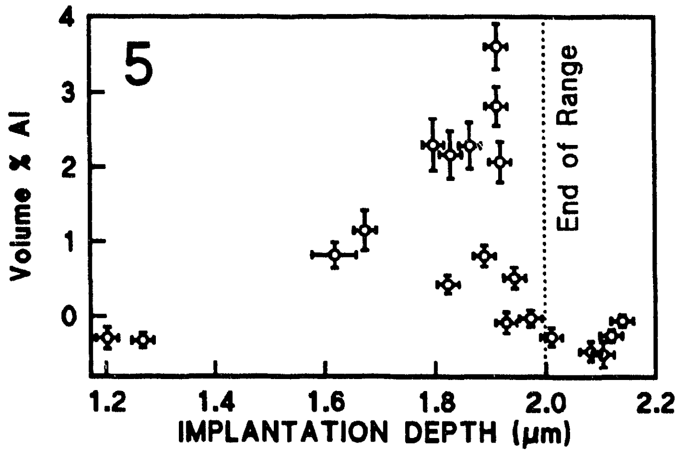

FIG. 1.--Implanted ion region of Mg^+ implanted $MgAl_2O_4$ spinel.

FIG. 2.--Spinel 222 dark-field image reveals small features in implanted ion region.

FIG. 3.--Fourier-log deconvoluted low-loss spectra from metallic Mg, metallic Al, undamaged spinel, material in implanted ion region (a), and best-fit prediction from multiple regression analysis (b).

FIG. 4.--Spectra from regions (1) and (2) indicated in Fig. 2.

FIG. 5.--Metallic Al profile across implanted ion region.

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