Influence of embedded Au-nanoclusters on the Atom Probe Tomography of Bulk MgO

Satyanarayana V. N. T. Kuchibhatla*, P. Adusumilli*, ***, T. J. Prosa **, R. M. Ulfig**, V. Shutthanandan*, B. W. Arey*, S. Thevuthasan*

* EMSL, Pacific Northwest National Laboratory, Richland, WA 99354
** Cameca Instruments Inc, Madison WI 53711
*** Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208

The advent of laser-pulsing in atom probe tomography (APT) has significantly expanded the scope of this high spatial resolution, three-dimensional analysis technique to study insulating materials such as oxides. Recent first-of-their-kind studies on bulk insulating magnesium oxide single crystal substrates, with and without embedded gold nanoparticles, have revealed some very interesting information. This presentation will include some of these results describing the influence of laser energy on the field evaporation characteristics of pure and Au clusters imbedded MgO specimens during laser-assisted APT. The Au clusters imbedded specimens were prepared by implanting 2.0 MeV $2x10^{17}$ Au^{+}/cm^{2} in bulk MgO crystals at various temperatures and subsequently annealing the specimens at $1000^{o}C$ for 10 hours in air. Samples for APT were prepared using a dual-beam focused ion beam microscope using the lift-out technique. Significant variations have been observed in the mass resolution and the TOF-mass spectrum as a function of the presence of nanoclusters in the oxide matrix. While the average dimensions of the gold nanoparticles in the matrix agree with the TEM and STEM measurements, APT data indicates that the Au-clusters may not be pure as reported by researchers in the past. Furthermore, using atom-probe reconstructions, (see Figures 1 and 2 below), the presentation will shed light on the different evaporation characteristics observed at high and low laser energies. Additionally, the influence of laser energy and Au nanoparticle presence on the bulk composition will also be discussed here.

While it is too early to make conclusive propositions on the Au-MgO system, these results are highly encouraging to pursue the analysis of bulk insulators and dielectric materials using laser assisted atom probe tomography. It is being hypothesized that the selective absorption of the laser by the Au nanoparticles might result in localized heating and there by result in magnesium oxide clustering in the vicinity of Au clusters. MgO clustering effects appear to depend on the laser energy and the results clearly demonstrate that complex MgO clustering can be avoided by utilizing low laser energies as shown in Fig. 1.

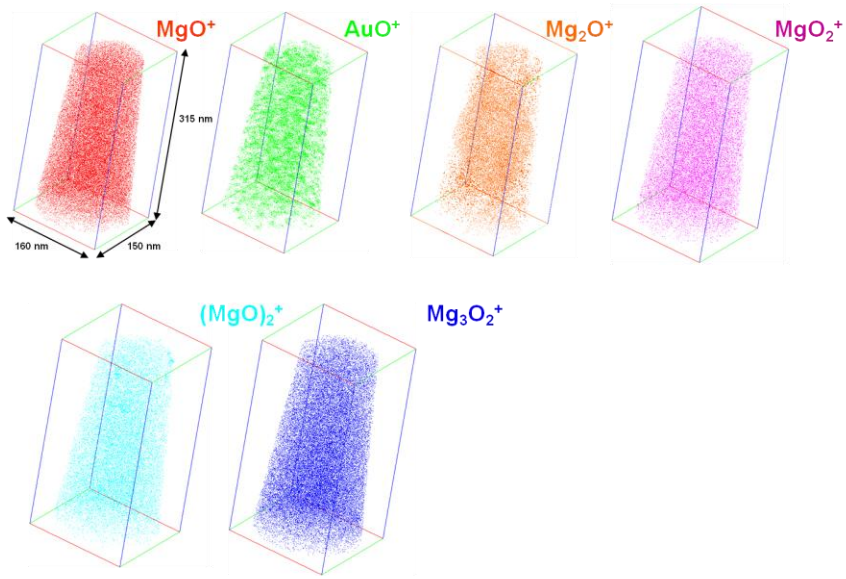

FIG. 1. Atom probe tomographic reconstructions of various complex species at 0.3nJ laser energy from an Au-nanoparticle-embedded MgO sample.

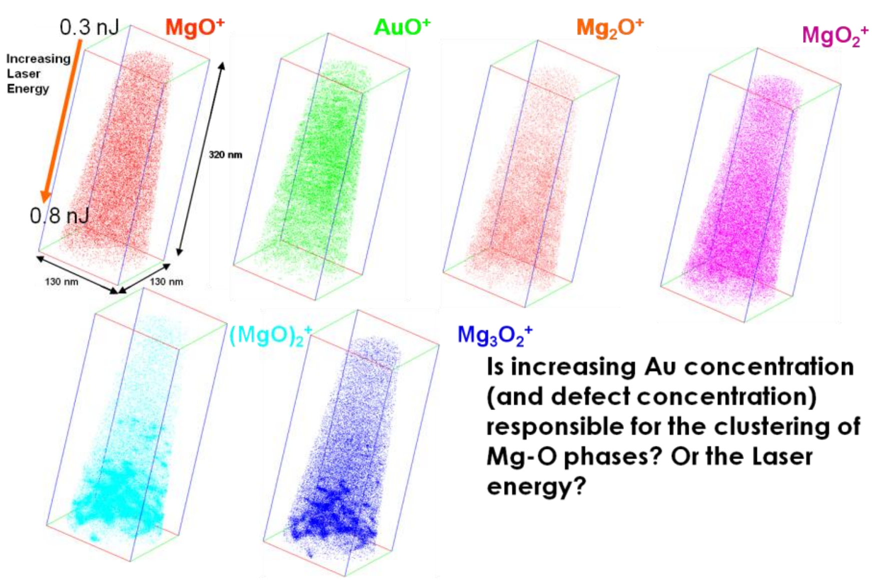

FIG. 2. Atom probe tomographic reconstructions of various complex species at varying laser energy from 0.3nJ to 0.8nJ from Au-nanoparticle-embedded MgO sample. The influence of increasing laser energy on the clustering of complex species is evident.