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Atom-Probe Analyses of Carbide-Containing Steels—Comparison of Laser- and Voltage-Pulsed Results

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Laser-pulsed and voltage-pulsed atom-probe tomography (APT) has been applied to the analysis of a carburized nano-structured steel [1]. The material analyzed is the case material of an alloy developed by QuesTek Innovations, LLC (Evanston, IL). It is a vacuum melted alloy of Fe, Co, Ni, Cr, Mo, V, and W, with trace impurities. The microstructure is lath martensitic matrix, with a fine dispersion of strengthening carbide precipitates (primarily M_2C, where M is Mo, Cr, Fe, V, or W). In the analyzed region of the carburized case material, the carbon content is ~1.6 at. %. The properties of alloy, including an outer case hardness of >65 on the Rockwell C scale and outstanding bending fatigue resistance, make it an excellent candidate for gear and rolling-contact applications [2].

Fig. 1 shows a comparison of mass spectra from laser-pulsed (a) and voltage-pulsed (b) APT analyses of the developmental alloy [3]. The differences fall into two main categories. First, the laser-pulsed APT mass spectrum has significantly better mass resolution. The FWHM (full-width half-maximum) mass resolutions, measured for the $^{56}Fe^{2+}$ peak at a mass-to-charge state ratio of 28 amu, are ~670 and ~350 for the laser- and voltage-pulsed analyses, respectively. The FWTM (full-width tenth-maximum) resolutions are ~275 and ~100, respectively (measured for the same peak). The improved mass resolution of the laser-pulsed analysis allows for better separation of the closely-spaced Fe, Cr, Ni, and Co 2+ peaks in the mass spectrum. In addition, the seven different isotopes of molybdenum are better resolved in the laser-pulsed mass spectrum.

Second, the laser-pulsed APT mass spectrum exhibits peaks that are less-predominant, or absent, in the voltage-pulsed APT mass spectrum. In particular, laser-pulsed APT leads to a much larger proportion of $(MoC)^{2+}$ ions in the mass spectrum, which are associated with the field-evaporation of the carbide precipitates. Laser-pulsed APT also results in a presence of a strong and distinct C_3^{1+} peak at 36 amu, a peak which is much less distinct in the voltage-pulsed APT analysis. Multi-carbon atom clusters of this sort are also associated with the field-evaporation of carbide precipitates. Another difference of the laser-pulsed APT mass spectrum is the presence of Ni^{1+} ions, essentially absent in the voltage-pulsed APT mass spectrum (not included in Fig. 1). With more Ni^{1+} ions, much less Ni^{2+} is observed in the laser-pulsed spectrum. Likewise, much less Mo evaporates as 3+ charged under laser-pulsed conditions. As a result of these two trends, the overlap of Ni^{2+} with Mo^{3+} peaks, which is a problem when determining quantitative compositions, is much less of a problem in laser-pulsed APT analyses of steels. The distinct differences in the charge states and the peaks present in the laser- and voltage-pulsed APT analyses are related to differences in the physical mechanisms of voltage-pulsed and laser-pulsed field-evaporation, and have yet to be fully explained and explored scientifically.

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[1] The provision of the steel samples by QuesTek Innovations, LLC, Evanston, IL, and Ben Tiemens, Northwestern University, Evanston, IL, is gratefully acknowledged.
[2] C. J. Kuehmann and G. B. Olson, Adv. Mater. Process., 153 (5) (1998) 40.
[3] Samples analyzed in the LEAP 3000X laser-pulsed APT at Imago Scientific Instruments and the LEAP 3000 voltage-pulsed APT at NUCAPT.

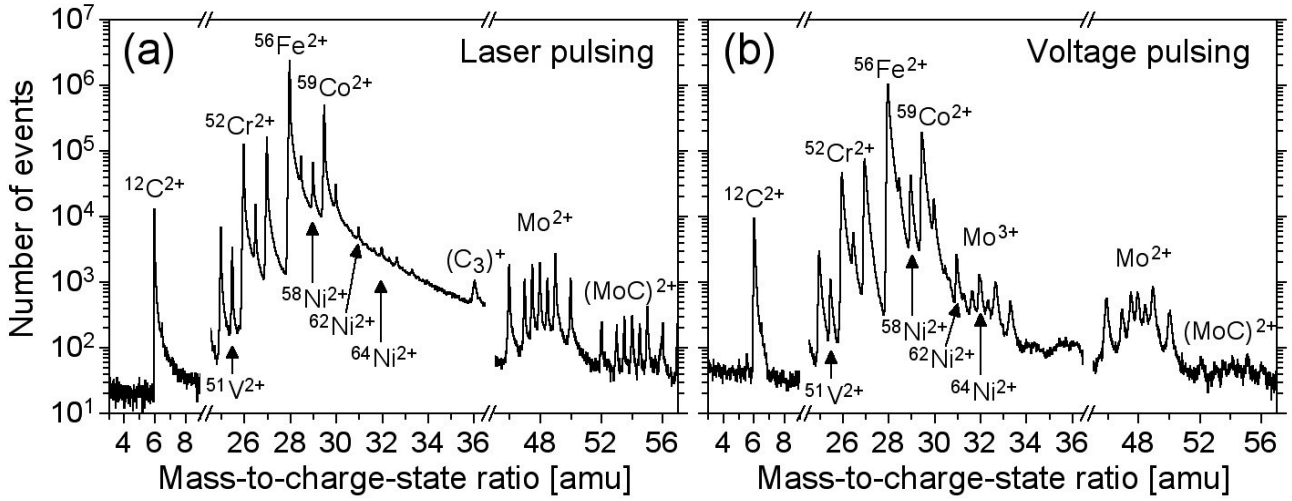

Fig. 1 Comparison of laser-pulsed (a) and voltage-pulsed (b) mass spectra from the atom-probe tomography analyses of a carburized, nano-structured steel. Note the log scale of the ordinate, and that the abscissa is truncated twice.

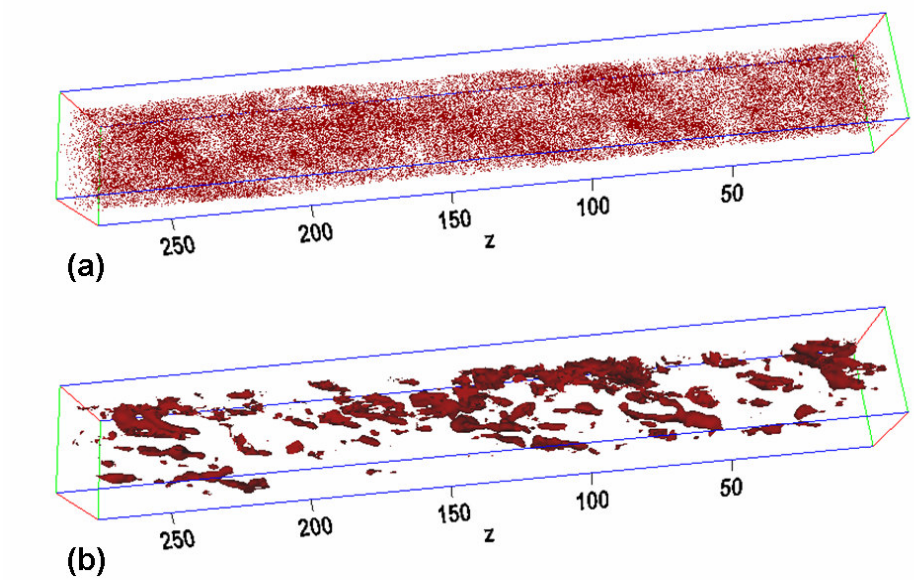

Fig. 2 Three dimensional atom-by-atom reconstruction of a laser-pulsed APT analysis of a carburized, nano-structured steel. For the sake of clarity, only carbon atom events associated with the mass spectrum peaks at 6 and 12 amu are shown in the atom map (a). The analysis volume is 37 nm x 37 nm x 276 nm and contains ~13 M ions. The overall carbon content is approximately 1.6 at. %. A very fine dispersion of carbide precipitates is apparent, as highlighted by the 5 at. % threshold carbon isoconcentration surface shown in Fig. 2b.

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