

Atom Probe Tomography Characterization of Multilayer Films

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The performance-defining properties of multilayer films and devices are sensitive to the nature and quality of the inter-layer interfaces. In principle, atom probe tomography should enable the compositional variations across and along the interface and within the individual layers to be estimated [1,2]. However, the application of atom probe tomography to these materials has revealed some unexpected artifacts. Some of these artifacts are reported in this paper.

The multilayers comprising of 40 layer pairs of 2.2 nm Ti/3.3 nm Nb were sputter deposited under UHV conditions at ambient temperature onto a 5 Ω n-doped Si [100] wafer that was previously "Bosch Etched" to produce 5 x 5 μm posts. An \sim 0.5-nm-thick native oxide was present between the surface of the Si wafer and the first Nb layer. After deposition, individual posts were removed from the wafer and attached to stainless steel pins with conducting epoxy. A 3 x 3 x 2 μm Pt cap was deposited on the surface of each post to protect the multilayer during subsequent milling. The needle-shaped atom probe specimens were produced by annular milling [2] with 30 keV Ga ions in a FEI DB-235 Focus Ion Beam. Atom probe analysis conditions were a specimen temperature of 50K, a pulse fraction of 20% and a pulse repetition rate of 1.5 kHz. The atom probe analysis was initiated approximately 20 min. after loading the specimen into the pre-cooled cryostat. TEM cross section of the multilayer revealed a uniform lamellar microstructure with a columnar grain morphology of 5-15 nm grains.

The mass spectra from these multilayers differ from those from metallic specimens in that the sharp mass peaks have a pronounced high mass tail, Fig. 1. These high mass tails degrade the mass resolution and complicate the estimation of the concentrations due to peak overlap. These high mass tails are due to large energy deficits of the field evaporated ions. These large energy deficits are indicative of a significant reduction of amplitude of the high voltage pulse used to initiate field evaporation due to poor electrical conduction. The background noise level was significantly higher than in metallic specimens. The background noise also decreased with the elapsed time during the experiment and was higher in the Ti layers compared to the Nb layers, Fig. 2. The background hydrogen was also significantly higher in the Ti layers compared to the Nb layers. The background noise is due to a combination of the dark count of microchannel plates in the detector plus some other sources of noise that occur at a combined rate of \sim 0.0002 events per pulse (accounting for $<$ 5% of the noise) and to unsynchronized field evaporation of the specimen on the standing voltage [1]. As the timing system used is only active for 0.6% of the time, \sim 166 times more ions will be lost for each noise event detected due to random evaporation. The decrease in the number of lost ions with time is manifested in an apparent change in thickness of the films with distance especially during the initial stages of the experiment, Figs. 2 and 3.

The high background noise is consistent with uncontrolled field evaporation on the standing voltage of the specimen. The uncontrolled field evaporation was more prevalent in the Ti layers than in the Nb layers due to its lower evaporation field. The change in the amount of uncontrolled field evaporation with time is most likely due to a change in the temperature at the analysis surface of the multilayers due to poor thermal and electrical conduction [3].

- [1] M.K. Miller, *Atom Probe Tomography*, Kluwer Academic/ Plenum, New York, 2000.
 [2] D. J. Larson et al., *Microscopy and Microanalysis*, 7 (2001) 24.
 [3] Research at the Oak Ridge National Laboratory SHaRE Collaborative Research Center was sponsored by the Division of Materials Sciences and Engineering, U.S. Department of Energy, under contract DE-AC05-00OR22725 with UT-Battelle, LLC.

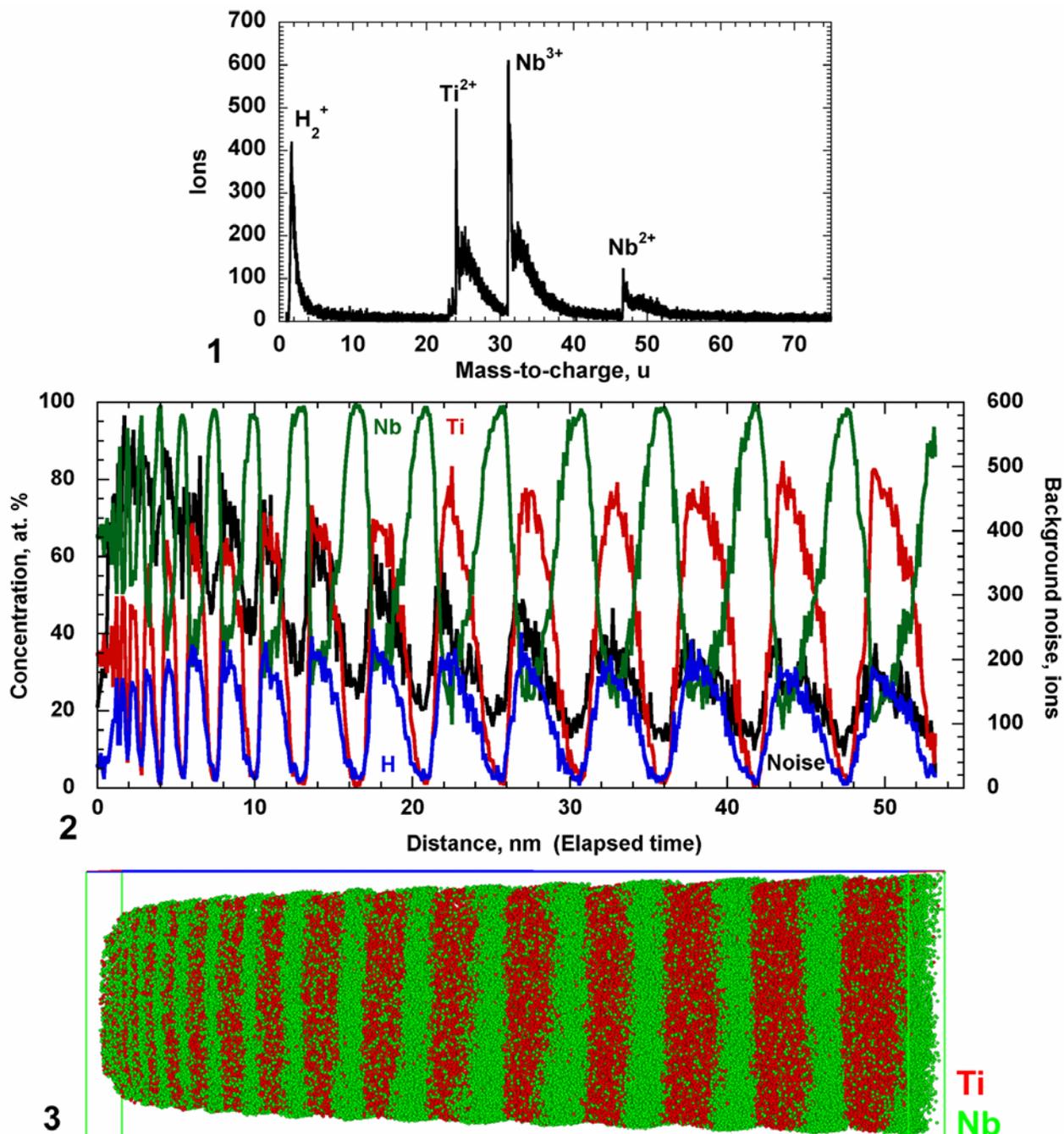


FIG. 1. Mass spectrum from a Nb-Ti multilayer. Significant high-mass tails are evident.
 FIG. 2. Composition profile from the surface of the specimen showing the Nb and Ti layers and the number of noise events decreasing with distance or elapsed time.
 FIG. 3. Atom map showing the Nb and Ti layers. The apparent change in the thickness of the layers is an artifact due to uncontrolled field evaporation on the standing voltage.