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Analysis of Multiple-ion Events in Atom Probe Tomography Studies of MoNbTi and HfNbTaTiZr Refractory High Entropy Alloys

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High entropy alloys (HEAs) are a class of alloys containing several principal elements, typically five or more in nearly equiatomic proportions. Development of this new class of alloys has resulted in a significant expansion of the composition spaces and attainable properties in novel alloy systems. Recently there has also been a surge of interest in refractory HEAs for structural applications in extreme environments. Despite the potential advantages of HEAs, fundamental questions remain regarding the mechanical properties of these materials [1]. In order to better understand the physical mechanisms controlling mechanical behavior in these alloys, a better understanding of dislocation motion and of diffusion is needed. The high spatial resolution and chemical sensitivity of atom-probe tomography (APT) can provide unique insights into phenomena such as atomic clustering, short range order, and interstitial content which, in turn, can govern the mechanical behavior of these alloys. These analyses require optimal spatial and chemical data making APT particularly well suited for the study of the fundamental properties of HEAs.

In this work we will present the effects of experimental parameters, such as APT laser pulse energy, laser pulse frequency, and specimen base temperature on the analysis yield, data quality, and tendency for generation of complex ions in the equiatomic refractory HEAs MoNbTi and HfNbTaTiZr. APT data was collected using a Cameca LEAP 4000X Si in laser pulsing mode on tips prepared using standard liftout methods in a ThermoFisher Nova 600 FIB-SEM [2]. Fig.1a-b shows APT mass spectra from a MoNbTi sample collected at 80 K and a laser pulse energy of 40 pJ. The mass spectra of the single hits is shown in blue, while the multiple-ion events, or multi-hits, when two or more ions arrive after a single laser pulse, are shown in peach. Fig.1a shows the region of the mass spectra for Ti^{2+} ions. It is clear that most of the Ti^{2+} ions are detected as single hits, with only 5% detected as multi-hit events. Fig.1b shows Mo^{3+}, Nb^{3+}, NbN^{3+}, and NbO^{3+} peaks. Unlike Ti, Mo and Nb multi-hit events account for the same order of magnitude of collected ions as single-hit events, as demonstrated by the overlapping curves in Fig.1b. Here, 39% of Mo atoms and 34% of Nb atoms are detected in multi-hit events. Given the large fraction of atoms collected in multi-hit events, understanding the field evaporation of these species is important. Correlation histograms [3] of the region of multi-hits involving $Mo^{1+} + H^{1+}$ and $Nb^{1+} + H^{1+}$ are shown in Fig.1c. These tracks indicate molecular ion dissociation, which are identified in the theoretical ion dissociation tracks overlayed in Fig.1d. There are ion dissociation tracks for Mo^{1+} and Nb^{1+} with H^{1+}, and also Nb^{1+} with molecular Hydrogen, H_2. The consequences of these multi-hit events on quantitative APT measurements in these refractory alloys will be discussed.

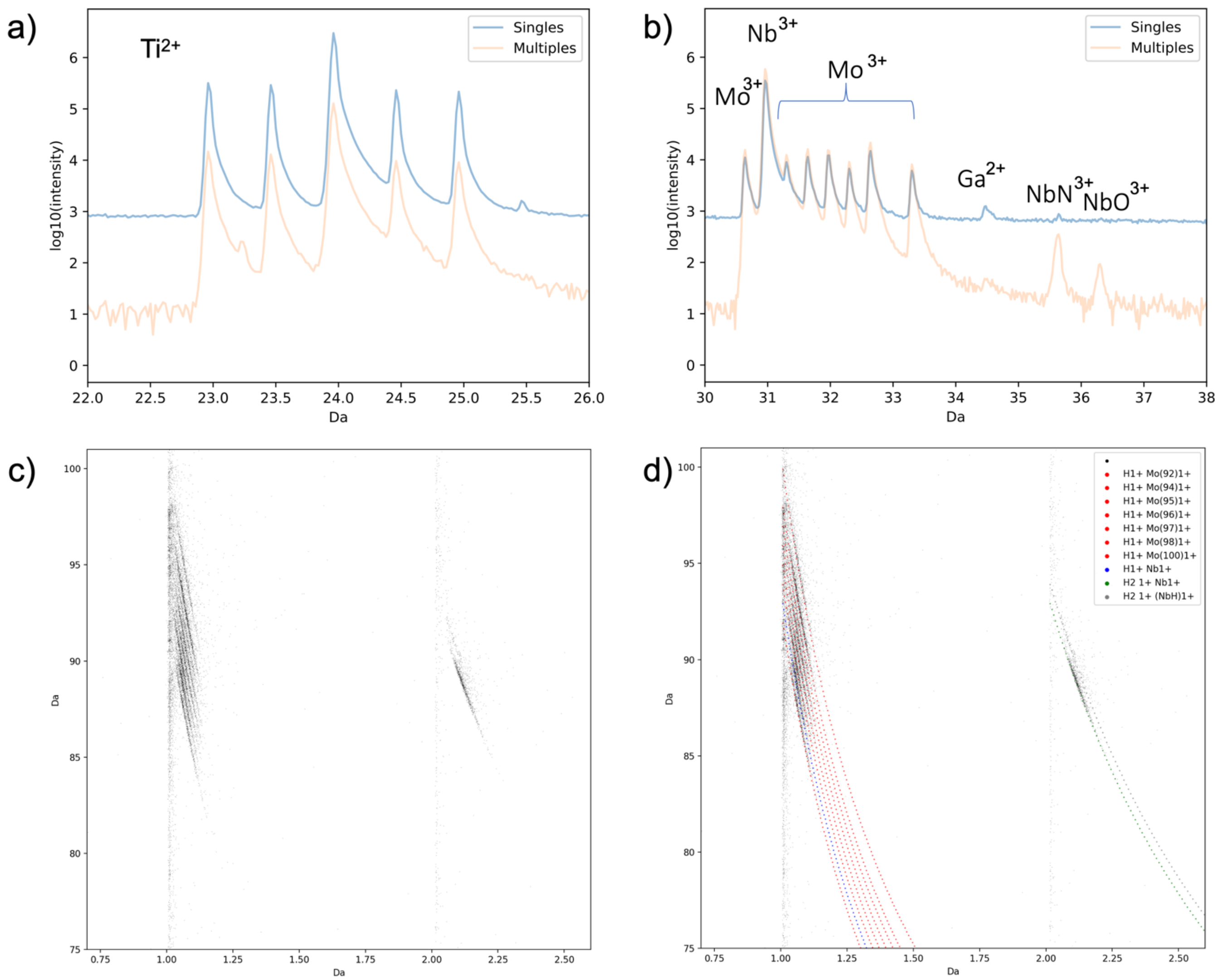

Figure 1. a) Shows the Ti^{2+} region of a mass spectrum from a MoNbTi refractory HEA. b) Shows the region of a mass spectrum from a MoNbTi refractory HEA with Mo^{3+}, Nb^{3+}, NbN^{3+}, and NbO^{3+}. c) Shows a correlation histogram highlighting multi-hits involving H, and Mo and Nb, and d) shows the same region with theoretical ion dissociation tracks overlayed.

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