

Compositional Analysis of as-cast and Crystallized Pd₄₃Cu₂₇Ni₁₀P₂₀ Bulk Metallic Glass

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Noble metal-based bulk metallic glasses (BMG) composed of Pd-Cu-Ni-P are viewed as potential materials for use in thermoplastic nanofabrication/transfer mold lithography [1] and electrocatalytic applications [2]. The stability, formability and physical properties of these glasses are related to the kinetic pathways taken towards their decomposition and crystallization, which can be probed by analyzing on the nanometer scale the local composition and structure. Atom probe tomography (APT) is capable of atomic resolution elemental mapping and with the use of laser pulsing has proven to be capable of efficiently detecting nanometer scale compositional fluctuations indicative of phase separation in BMGs [3].

In this study, the composition and microstructures of a Pd₄₃Cu₂₇Ni₁₀P₂₀ bulk metallic glass were characterized with a laser pulsed local electrode atom probe operating at a laser wavelength of 532nm and 12ps pulse duration. Studies were carried out on samples in the as-cast and fully crystallized state. Specimens for APT were prepared from these samples using standard in-situ FIB lift-out techniques. Specimen geometry and APT instrumental parameters were optimized to obtain agreement of APT analysis of the as-quenched specimens with the nominal specimen stoichiometry as well as maximize the yield and length of successful APT analyses of the crystallized specimens so as to study nanometer scale compositional variations across micron scale contiguous regions of the sample, as shown in Fig. 1. The nanometer scale distribution and partitioning of Pd, Cu, Ni and P within the annealed sample was identified from APT data with the presence of several distinct phases and atomic segregation to interfaces between these phases, as shown in Fig. 2. The atom probe investigations have been coupled with preliminary transmission electron microscopy (TEM) studies on the crystallized Pd₄₃Cu₂₇Ni₁₀P₂₀ bulk metallic glass and will be presented.

References:

[1] G. Kumar *et al.*, Nature **457** (2009) p. 868.

[2] R. Sekol *et. al.*, International Journal of Hydrogen Energy, **38** (2013) p. 11248.

[3] L. Yang *et al.*, Adv. Mater. **21** (2009), p. 305.

[4] The authors acknowledge use of the electron microscope and atom probe facilities of the University of North Texas Center for Research and Technology.

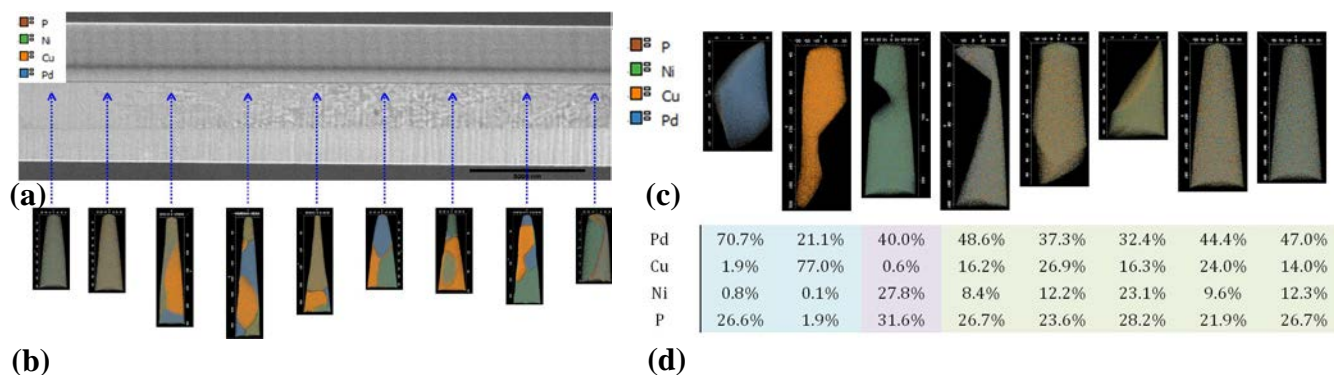


Figure 1. (a) SEM micrograph of a sample wedge extracted from the 723K annealed Pd₄₃Cu₂₇Ni₁₀P₂₀ BMG, scale bar = 5μm, and (b) APT reconstructions and the approximate location of the APT reconstructions with regards to the sample wedge as indicated by the blue vertical arrows, not shown to the same scale. (c) Representative isolated compositionally distinct regions from the analyzed Atom Probe reconstructions of the 723K annealed Pd₄₃Cu₂₇Ni₁₀P₂₀ BMG and (d) the atomic concentration of these regions.

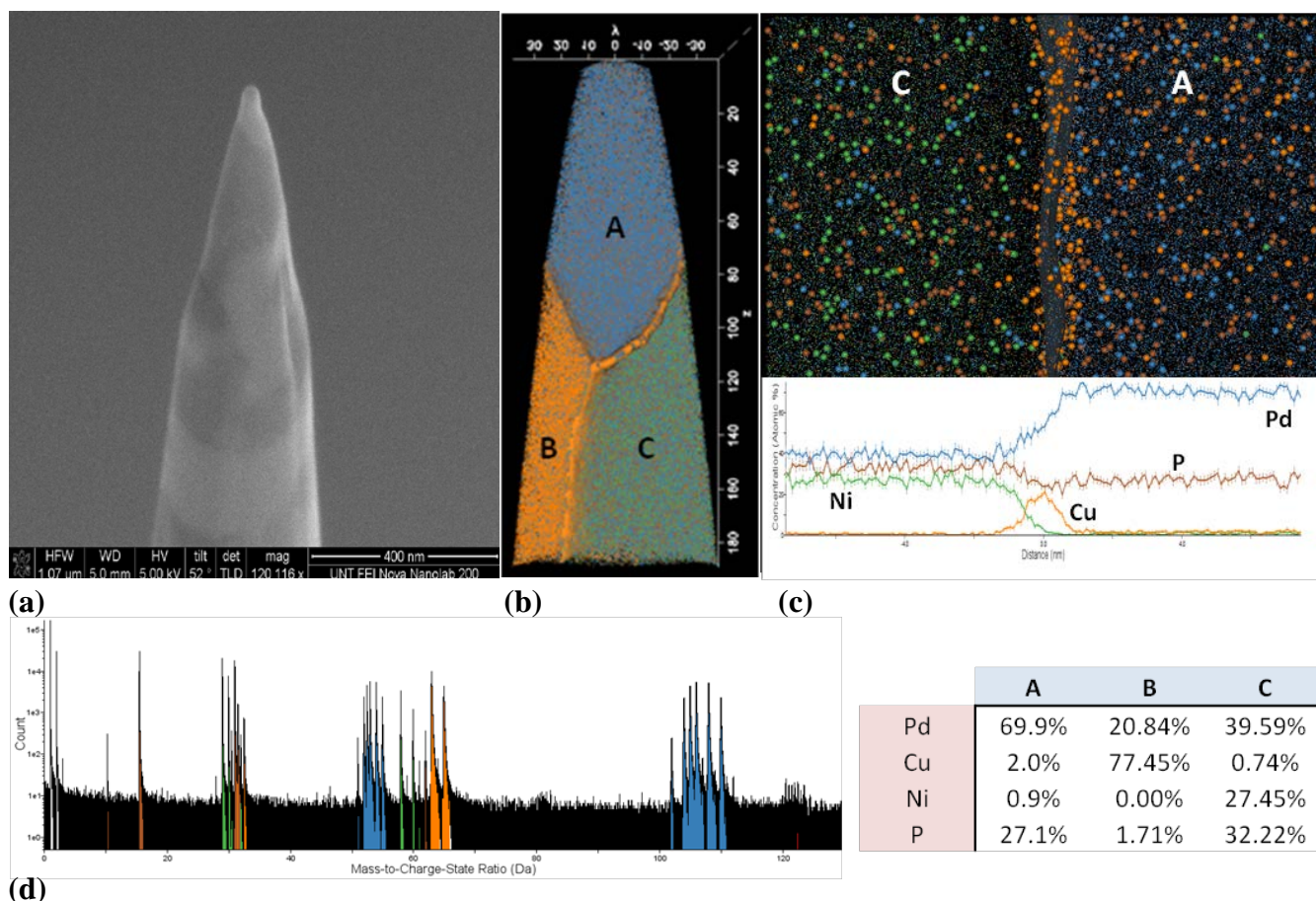


Figure 2. (a) SEM micrograph of a FIB prepared Atom Probe sample and a (b) APT reconstruction of the sample. (c) A 15×15×15nm³ region centered about the A-C interface and (below) the composition profile across the interface. (d) Mass-to-charge spectrum for this specimen and (e) atomic percent composition of the regions indicated in (b).