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Pu L_{III} and Fe K Edge X-ray Absorption Spectroscopy of Bulk Pu₆Fe Synthesized via Arc Melting

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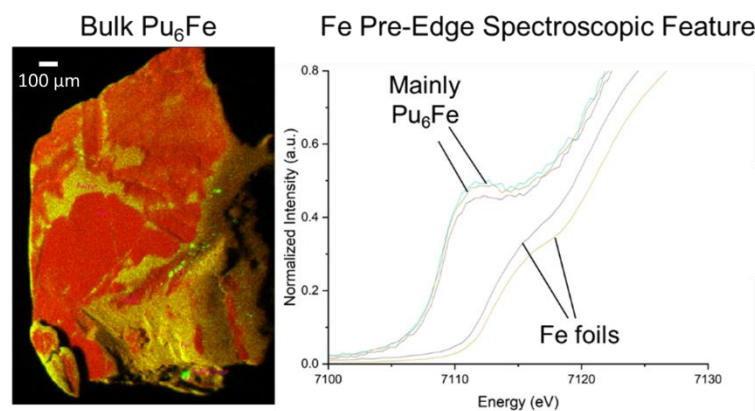


Figure 1. Elemental map (Pu – red, Fe – green) of an arc melted piece of Pu₆Fe without surface preparation. Maps were collected in fluorescence mode at beamline 2-3, SSRL, X-ray spot size ~5 μm.

Plutonium has the most complicated phase behavior of any single element, exhibiting no less than 6 crystalline phases between room temperature and melting. This inherent complexity of Pu is only exacerbated when it is alloyed, either intentionally or incidentally as with impurity elements. Among others, systems containing Pu and Fe were studied early in the history of Pu materials science, where the Pu₆Fe eutectic alloy was used in the first metallic plutonium fuel elements, which powered the Los Alamos Molten

Plutonium Reactor in the late 1940s. The presence of the Pu₆Fe has also been identified in Pu-Ga alloys containing Fe impurities via TEM by scientists at Lawrence Livermore National Laboratory (2002). Synthesis of Pu₆Fe has been executed recently at Los Alamos National Laboratory by fusion of powders, and then for this work via arc melting of metallic Fe and Pu components in stoichiometric proportions. Although the phase space of the Pu-Fe system has been generally explored, atomic scale structural characterization, including manifestation of impurity phases as a result of processing conditions, has not been fully realized for this compound. X-ray absorption spectroscopy has proven viable for characterizing a wide range of local structural effects in general, as well as in Pu alloys specifically, hence our application of this technique to Pu₆Fe. Herein we present the first fluorescence mode X-ray absorption spectroscopy of this material, including Pu L_{III} edge and Fe K edge fluorescence data for two separately synthesized and processed pieces of Pu₆Fe. We seek to establish X-ray absorption signatures for this material and to make a start at associating these signatures with processing conditions, thereby providing a basis for future study of this system.

Two pieces of Pu₆Fe, each synthesized separately, were analyzed using fluorescence mode X-ray absorption spectroscopy at beamline 2-3 within the Stanford Synchrotron Radiation Light source (SSRL) at SLAC National Accelerator Laboratory. This analysis resolved the Pu₆Fe phase itself, as well as various other phases such as alpha plutonium, PuFe₂, and various degrees of oxidation exhibited in both the Pu and Fe spectroscopic data (see elemental map in Figure 1). While oxides were observed each set of data, plutonium oxide was present more often and with higher scattering intensities. This could suggest that the plutonium within the Pu₆Fe or PuFe₂ phases may oxidize more readily than the iron. Somewhat unexpectedly, the Fe pre-edge features in the from both pieces provide an interesting signature for Pu₆Fe and its associate phases (Figure 1). Point spectroscopy associated with Pu₆Fe, PuFe₂, or other phases shows

a clear difference, in the form of an intense single shoulder feature, in the pre-edge compared to metallic Fe or Fe₂O₃. Pre-edge features are influenced by a variety of factors, from simple symmetry considerations to electronic density of states and bonding interactions. While deconvoluting or modeling those features are out of the scope of this work, we nevertheless call attention to transition metal pre-edge features, within the X-ray absorption spectra, as a possible probe for bonding and/or hybridization in actinide intermetallics.