Unconventional mechanical and magnetic properties are often reported for materials with a microstructure dimension of less than 100 nm. To develop new nanostructured materials with improved properties, it is important to understand the structure-property relationships. In this talk, we will give an overview of our recent studies on the structure-property relationships of various metallic nanostructures that were processed by the crystallization of amorphous precursors or mechanical milling. The three-dimensional atom probe (3DAP), which can map alloying elements in a three dimensional real space with a near atomic resolution, was mainly employed for the characterization of metallic nanostructures. We will demonstrate how the 3DAP can be used to obtain critical information on the mechanism of nanocrystalline microstructure evolution by reviewing our recent studies on two phase metallic glasses and nanocrystalline steel.

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