Materials Science & Engineering Doctoral Defense

Characterization of Composition Amplitudes and Wavelengths of Spinodal Decomposition Induced Modulated Structures in Small Unit Cell Metal Alloys Using Aberration Corrected STEM

School for Engineering of Matter, Transport and Energy

Ronit Sawant

Advisor: Prof. Ray Carpenter

Abstract

Modulated nanostructures are short-range periodic composition fluctuations observed in metals, semiconductors, and ceramic alloys and have an important effect on the mechanical, electrical, and magnetic properties of the materials. Their presence is often attributed to spinodal decomposition. In the past, such modulations have been analyzed using X-ray diffraction, electron diffraction, and diffraction contrast imaging techniques. The investigations gave useful information about the spatial distribution of the modulations but nothing about the composition fluctuation of the modulated structure. In this study, we measure the composition amplitude of the fluctuations directly using STEM Imaging and atomic resolution spectroscopy in Au-Pt and Cu-Ti alloys. With the development of field emission sources, aberration correctors, and ADF imaging for STEM microscopes, measuring the amplitude of the modulation directly and examining the diffused interface is possible.

Au-Pt alloys, featuring a nearly symmetrical solid-state miscibility gap, and a Cu-Ti alloy with an asymmetrical metastable miscibility gap were chosen for investigation. Three Au-Pt alloys of different compositions were analyzed at a specific temperature for varying aging times. The study successfully employed atomic resolution energy dispersive X-ray spectroscopy (EDS) for Au-Pt alloys and atomic resolution electron energy loss spectroscopy (EELS) for Cu-Ti alloys to measure composition variation and diffused interfaces across modulations.

In Au-Pt alloys, it was shown that the wavelength, as well as the composition amplitude of the modulations, increases as the alloy is aged for a longer time. Non-uniform distribution of wavelength and composition amplitude of modulated structures was observed across the samples. Results shows competitive growth mechanisms occurring in nanostructures with a range of wavelengths/amplitudes. Composition of Au-rich and Pt-rich regions deviates significantly from equilibrium at the selected temperature, more aging is necessary to observe coherency loss. Similarly, in Cu-Ti alloys, variations in wavelengths and composition amplitude of modulations were observed, along with clustering and ordering reactions. Ordering was specifically noted in Ti-rich regions of the alloy aged for an extended period. Diffused interfaces were observed in both alloy systems rather than chemically sharp interfaces, and the contrast in STEM images in both systems was predominantly influenced by strain effects.

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Arizona State University