Exploring the Dynamic Behavior of Compositionally Complex Alloys through Real-Time Observation with *In Situ* TEM/STEM

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Introduction

The development of multicomponent high entropy alloys (HEAs) has opened the door to sculpt advanced microstructures in the near-infinite compositional space, resulting in materials with unprecedented mechanical and functional properties. However, fully harnessing the potential of these intricate alloys necessitates a profound comprehension of the underlying phase transformation mechanisms throughout their fabrication and utilization. Nowadays, *in situ* transmission electron microscopy (TEM)/scanning TEM (STEM) techniques stand as formidable tools to unravel the complexities of such alloys. By facilitating real-time observation of microstructural evolution at the atomic scale, these techniques provide unparalleled insights into how these materials react under diverse temperatures or strains. Such capabilities are indispensable for refining alloy design methodologies and elevating material performance to unprecedented levels.

Objectives

This presentation focuses on advanced *in situ* probing techniques within the TEM/STEM, designed to observe plastic deformation and temperature-dependent phase changes in HEAs. I will introduce a novel mechanism of transformation-induced plasticity discovered through *in situ* straining in an FeMnCoCr alloy. Furthermore, I will show the results of *in situ* heating experiments conducted in the TEM/STEM, illustrating how they track temperature-induced phase transitions and the emergence of secondary phases in a carbon-doped FeMnCoCrNi alloy.

Materials & methods

The HEAs are fabricated by vacuum induction melting and investment casting using at least 99.5 wt.% pure metals. Following fabrication, thermo-mechanical treatment is applied to homogenize the alloys and attain the desired initial microstructure. *In situ* tensile straining experiments are performed using a specially designed straining inset within a model 625 Gatan straining holder. For *in situ* heating, samples are positioned in the TEM utilizing a DENSsolutions Lightning heating/biasing holder, enabling observation of microstructural changes through both TEM and STEM modes in Titan Themis microscopes (Thermo Fisher Scientific).

Results

The dual-phase $Fe_{50}Mn_{30}Co_{10}Cr_{10}$ (at.%) alloy primarily undergoes displacive phase transformation from a face-centered cubic (FCC) to a hexagonal close-packed (HCP) structure during deformation. Our *in situ* tensile straining experiments unveil a bi-directional transformation: FCC transitions to HCP and reverts back to FCC under strain. This dynamic phase transformation not only refines the microstructure but also enhances the alloy's work-hardening capabilities significantly.

While this nanostructure shows exceptional room temperature properties, it faces challenges such as rapid coarsening at elevated temperatures, leading to a decline in mechanical properties. To overcome this, we explore interstitial doping with C to stabilize the microstructure. Our *in situ* TEM/STEM observations provide valuable insights into the underlying phase transformation mechanisms. At intermediate temperatures (~300 ºC), we observe the emergence of nano-twinned FCC structures originating from initial HCP grains. Upon further heating to 700 ºC, elongated nanoscale carbides start forming along the FCC twin boundaries, effectively stabilizing the nano-twinned regions up to 900 ºC. Observations at extreme temperatures (~900ºC) reveal the dissolution of carbide phases and subsequent de-twinning, offering crucial insights into microstructural evolution in these compositionally complex alloys.

Conclusions

In the present work, we showed that *in situ* straining and heating in the TEM/STEM provides unprecedented insights into the phase transformation mechanisms in compositionally complex alloy systems. *In situ* probing is pivotal in observing the underlying transformation dynamics and is able to reveal previously overlooked phase transformation mechanisms.