Metallic nanocrystalline materials have been emerging with rapid pace in the past decade, thanks to many of their superior properties. With characteristic length scales on the order of a few nanometers to a few tens of nanometers, they have numerous applications. Processing of materials under non-equilibrium conditions (NEP) can be used to produce nanostructured materials with novel microstructures and metastable phases which can significantly improve materials properties. Nanocrystalline materials produced by two NEP techniques were studied for this dissertation. One of them is 304-type austenitic stainless steel (SS304 or Fe-18Cr-8Ni)-aluminum coatings produced by plasma enhanced magnetron sputter-deposition (PEMS) and the second is nanocrystalline Ti, Zr and Hf powders processed by mechanical milling (MM). The objective of the study was to understand the crystallographic and microstructural aspects of these materials.

In the former topic, four SS304+Al coatings with nominal compositions of 0, 3, 7 and 10 wt.%.Al were deposited on a SS304 substrate by PEMS using SS304 and Al targets. The as-deposited coatings were characterized by advanced metallographic techniques, x-ray diffraction (XRD), scanning electron microscopy (SEM), transmission electron microscopy (TEM) and three-dimensional atom probe microscopy (3DAP). The microstructure of all the coatings consisted of columnar grains with the columnar grain width (referred to as grain size) increasing with increasing Al content. The coatings had grains with rounded average grain sizes of 100, 290, 320 and 980 nm, respectively, for 0, 3, 7 and 10 wt.%Al. The observed grain structures and increase in grain size were related to substrate temperature. In the phase identification part, 0 wt.% Al coating consisted of ferrite and sigma phases. In the 3, 7 and 10% Al coatings, ferrite and the B2 phase (FeAl, NiAl or (Fe,Ni)Al) were observed but no sigma phase was found. Also, the quantity of the B2 phase present increased with the increase in Al content. Note that while ferrite and an intermetallic B2 are equilibrium phases for these compositions, sigma is a non-equilibrium phase. In 10 wt.%Al coating, we were able to demonstrate experimentally using 3DAP studies that NiAl phase formation is preferred over the FeAl phase at nano scale. In summary, the coatings were studied right from macroscopic scale down to angstrom scale and understood in terms of grain size and phase formation.

In the second topic of mechanical milling of metallic powders, an unknown nanocrystalline phase with face centered cubic (fcc) structure was found in Cu-Hf powders during glass forming ability studies by MM. This phase was identified as an allotrope of Hf originating from the hexagonal close packed Hf metal phase. The decrease in crystallite size down to nanometer levels, an increase in atomic volume, lattice strain, and contamination were the factors responsible for the transformation. The study was extended to Ti and Zr metals, and a similar phase transformation was found. A thermodynamic model known as the isothermal equation of state was used to study the role of nanocrystalline grains in stabilizing the metastable fcc phase.

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The public is welcome to attend.