

In-situ heating (scanning) transmission electron microscopy for exploring the thermal stability of a nanoscale complex solid solution thin film

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Merging thin film deposition by direct current sputtering (DCS) with in-situ heating (scanning) transmission electron microscopy ((S)TEM) allows us to study the thermal stability of complex solid solution nanomaterials which are promising candidates for energy applications (e.g. oxygen reduction reaction).

Despite many studies in bulk alloys and thin films with thicknesses >100 nm, there is a knowledge gap towards thin films with an average thickness of ~10 nm. Thus, in this investigation, we study the growth process and the thermal stability of 10 nm thick CrMnFeCoNi thin film and compare our results with bulk alloys or bulk-like films.

DCS process onto a heating chip leads to islands forming on the top of a continuous layer (Stranski-Krastanov growth mechanism). Immediately after DCS, two different phases are detected: CoNi-rich nanoscale islands and a continuous CrMnFe-rich layer.

In-situ annealing of the film up to 700 °C induce an Ostwald ripening effect of the islands, which is enhanced in the areas irradiated by the electron beam during the experiment. Moreover, the chemical composition of the continuous layer and the islands changed during the annealing process. After heating, the islands are still CoNi-rich but lower amount of Cr and Fe are detected and Mn was completely absent on them. Moreover, on the continuous layer the Co and Ni were removed, and the amount of Cr lowered. Furthermore, during the heating process oxygen (O) atoms presents on the film surface start to diffuse causing an O enrichment of the layer and in the interface between layer and islands preventing that Mn leaves the continuous film.

In the present work, our atomic scale in-situ studies on CrMnFeCoNi thin film will show how the different diffusion constants of the various atomic species may affect the thin film stability, morphology and chemical composition, illustrating the power of in-situ STEM in order to understand the growth mechanisms of such an interesting material.

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