Nanoparticle characterization by automated acquisition and analysis of images and EDS data in the TEM

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Getting meaningful statistics on size, shape, composition and location of nanoparticles is typically a tedious task given the small amount of particles that can be measured manually in a practical time frame. This also means that operator bias can become a serious issue.

In this study a workflow is presented where the acquisition of the STEM images and the EDS data on a user-selected region is automated. A large area can be mapped by making a mosaic of individual frames with each frame being analyzed while the next frame is being recorded. In this way it becomes possible to get statistical data on roughly 500 particles per hour. The actual throughput depends mostly on the number of particles per unit area.

This workflow has been demonstrated on different precipitation strengthening alloys such as high strength steels; high performance aluminum alloys and nickel-based superalloys. The technique works well to measure the nanometer-sized precipitates in samples prepared by the replica method though some of the spatial context inevitably gets lost. Samples prepared with a PlasmaFIB or by electropolishing where an electron transparent region is created across multiple grains allow for a statistical analysis of precipitates including their location relative to the grain boundaries.

Also for catalyst applications, the statistical analysis of metal nanoparticles plays a crucial role in developing new catalysts and predicting the performance. Three different catalyst samples are selected: a pristine sample and two catalyst samples with different lifetime. The morphological characteristics and spatial distribution are compared to get better insight into the degradation process during use.

Following new REACH regulations in Europe, for instance on the popular food additive E171 (titanium dioxide), a correct measurement of the particle morphology and composition is needed when new materials get certified for use in the food and feed chain. A direct measurement method like TEM ensures that also mixes of different nanoparticles such as used in confectionery are correctly characterized.

References

Verleysen et. al.; "Physicochemical Characterization of the Pristine E171 Food Additive by Standardized and Validated Methods", Nanomaterials 2020, 10, 592

