The scope of this work is the characterization of nanoparticles prepared by aggregation from the gas phase. The main focus is on their microstructure and real structure investigated by x-ray scattering methods, although combined with complementary methods such as electron microscopies and optical measurement. The microstructural characteristics, i.e. nanoparticle morphology and crystal and inner structure, were found to be tightly connected with the deposition parameters. For chosen systems, we also analysed the nanoparticle thermal or temporal stability and evolution.

Multiple gas aggregation deposition systems were used for the preparation of nanoparticles consisting of different materials. In detail, we studied single-metal nanoparticles – gold, silver and copper. For these, the behaviour with increasing temperature changes with the type of material and also its amount on the substrate. However, in general, the annealing of nanoparticles reduces the amount of structural defects in the crystal structure and causes the coalescence of nanoparticles. In the case of the copper nanoparticles, the coalescence is limited due to the nanoparticle oxidation in the air atmosphere. Similarly, in another system composed of niobium nanoparticles, we observed and described the gradual oxidation during annealing. On the contrary, for Nb nanoparticles, complete amorphization occurs, followed by the formation of a slightly substoichiometric niobium pentoxide phase, which structure was fully refined for the first time.

The gas aggregation cluster sources showed to be advantageous for the creation of core@shell nanoparticles, which inner structure can be tuned simply by varying the deposition parameters. Two different series were studied, i.e. Ag@ppHMDSO and Ag@TiO_x nanoparticles. Despite the thorough analysis of the microstructure, we further found that the thermal stability of silver nanoparticles is greatly enhanced by the other material coating.

Another investigated systems were nanofluids composed of vacuum-compatible liquid – polyethylene glycol and different single-metal nanoparticles prepared by direct deposition. By small and wide angle x-ray scattering techniques, nanoparticle sedimentation, separation and size evolution were observed during months of storage.