## "Nanoscale investigations by atom probe tomography and electron microscopy"

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Keywords: nanoscale chemistry, segregation, precipitation, atom probe tomography, analytical TEM

Modern material design and technology must be based on a profound knowledge of the correlation between material properties and their microstructure. In this context, especially challenging is the understanding of early stages of phase separation or segregation phenomena when changes in the material properties are observed but are not easy to track back to the changes in the microstructure. Generally, the most successful strategy in solving this type of problems is to use complementary methods for microstructural studies in order to obtain the most complete information. At the Department of Applied Physics at Chalmers University of Technology different electron microscopy techniques and atom probe tomography (APT) are used to study material chemistry, structure and their development down to the atomic level. Investigations incorporate analyses of interfaces and precipitation processes in steels, nickel base alloys, hard or oxidation resistant coatings and thin films. Some examples from these works will be presented with the focus on early stage of phase separation in maraging steels and studies of Ti<sub>2</sub>AlC (so called MAX-phase) coatings produced using the high velocity oxy-fuel (HVOF) technique.

Investigation of precipitation and its development in maraging steels has long been an experimental challenge since the steels consist of densely distributed, nanometer-sized, intermetallic particles embedded in a martensitic matrix. Studies of very small precipitates by TEM are always difficult, but in maraging steels additional complications arise from the highly magnetic martensite making acquisition of diffraction patterns experimentally demanding and time consuming. Furthermore, the chemistry of clusters and small precipitates or early stages of segregation can be hard, if not impossible, to obtain using different TEM techniques. Application of APT, on the other hand, offers the possibility to study the chemistry variation on nearly atomic level with equal sensitivity for all the elements. In the past the major drawback of the APT was its very small volume accessible for analysis. The situation has substantially improved during the recent years due to the development of wide angle APT that allows for investigations of volumes that are about 100 times larger than previously. Fig1 shows the distribution of different elements in a maraging steel that was heat treated only for 5 min at 475°C. After this short aging no signs of segregation or precipitation of elements were observed by TEM while precipitation of Cu, Ti, Ni and Al and segregation of Mo, Ti and P could be clearly revealed by APT.

 $Ti_2AlC$  coatings, made by HVOF spraying from  $Ti_2AlC$  powder, have a complex structure, both in terms of microstructure and composition. During the spraying process, parts of the  $Ti_2AlC$  powder transform so that the resulting microstructure is composed of large  $Ti_2AlC$  and TiC grains and very small TiC grains embedded in titanium aluminides with

different composition dependent on the local surrounding. Even in this case application of different electron microscopy techniques together with APT is essential for understanding the processes governing the final appearance of the coating. This is decisive for optimization of the coating procedure.



Fig. 1 Distribution of Mo, Cu P and Ti in a maraging steel that was aged for 5 min at  $475^{\circ}$ C (box size  $60 \times 60 \times 80 \text{ nm}^3$ ). Cu precipitation with subsequent depletion at lath boundaries is very clear. The observed enrichment of Mo at the lath boundaries exceeds the matrix level by only 0.5%.