ESRF	**Experiment title:** Nanocrystalline Ni-W Thin Films; Nanoscale Planar Faulting, Grain Growth, Segregation and Residual Stress	**Experiment number**: MA-1508
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Report:

Nanocrystalline thin films are in the focus of many investigations due to their outstanding properties. The presence of nanotwins was discovered to enhance the strength of pure metal films. Alloying of the films, furthermore, can stabilize microstructural features like grain boundaries but also twins, which enhances, in turn the thermal stability of the films. In that context we investigate magnetron-sputtered Ni-W films exhibiting a nanocrystalline and {111} fiber-textured microstructure, showing pronounced nanotwinning parallel to the surface. In this experiment, in particular, the evolution of the nanotwin structure upon annealing was studied non-destructively and partially in-situ on the basis of the associated weak diffuse scattering.

Experimental Procedure

The nanotwins aligned parallel to the film surface cause streaks of diffuse intensity in the reciprocal space, which can be evaluated using the software DIFFaX*plus [J. Appl. Crystallogr. 37 (2004) 166]* in order to determine the twin density. For each film and temperature, approximately 100 Θ-2Θ scans were performed by the use of a *Mythen* detector at different tilt angles χ. The construction of the intensity distribution along a diffuse-intensity streak in the reciprocal space as a function of the reciprocal space coordinate L_{hex} by the use of the measured Θ-2Θ scans is shown in Figure 1. The averaged intensity of the middle 5 % of each Θ-2Θ scan corresponds to one point of the intensity distribution along the streak in the reciprocal space. The temperature program was performed by the use of an Anton Paar DHS 900 heating chamber, heating from 305 K to 800 K in steps of 50 K and cooling down in 3 steps. Additionally, the intensity distributions along the streak in the reciprocal space of previously annealed films at 800 K for different aging times were investigated at ambient temperature.

The described in-situ investigation was unfortunately interrupted twice due to beam dumps, leading to deviations from the initially planned thermal treatments and thus difficultly interpretable diffraction data. Moreover, problems with the temperature control arose, which were not encountered earlier. Therefore, not all planned experiments could be performed and an additional beamtime would be appreciated.

Experimental Results

The evolution of the intensity distribution along the analyzed streak in the reciprocal space of the NiW12 film, i.e. containing 12 at.% W, as a function of temperature is shown in Figure 2a. At 700 K, the intensity maxima sharpen slightly, whereas no distinct change could be observed at lower temperatures. Upon further

heating, the sharpening proceeds, as also during the first cooling step from 800 K down to 650 K. The occurrence of intensity-maxima sharpening along the investigated streak in the reciprocal space corresponds to detwinning processes: The sharper the intensity maxima the smaller the twin density. The exact twin densities were extracted by the use of the program DIFFaX*plus*. As an example, a successful fit of the intensity distribution along the streak of a NiW12 film heated ex-situ 1 h at 800 K is shown in Figure 2b, indicating a twin probability of 17 %.

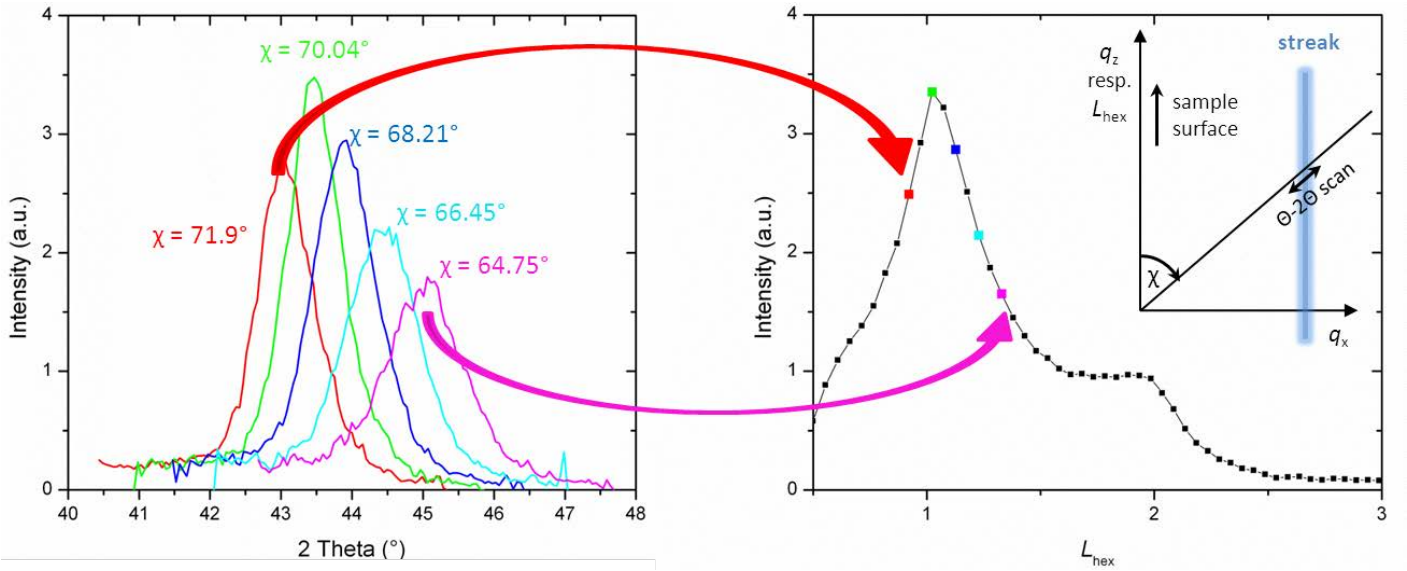

Figure 1: Example for a construction of the intensity distribution along the investigated streak in reciprocal space as function of the coordinate L_{hex} *by the use of Θ-2Θ scans. The inset shows schematically the investigated streak and the performed Θ-2Θ scans at different tilt angles χ.*

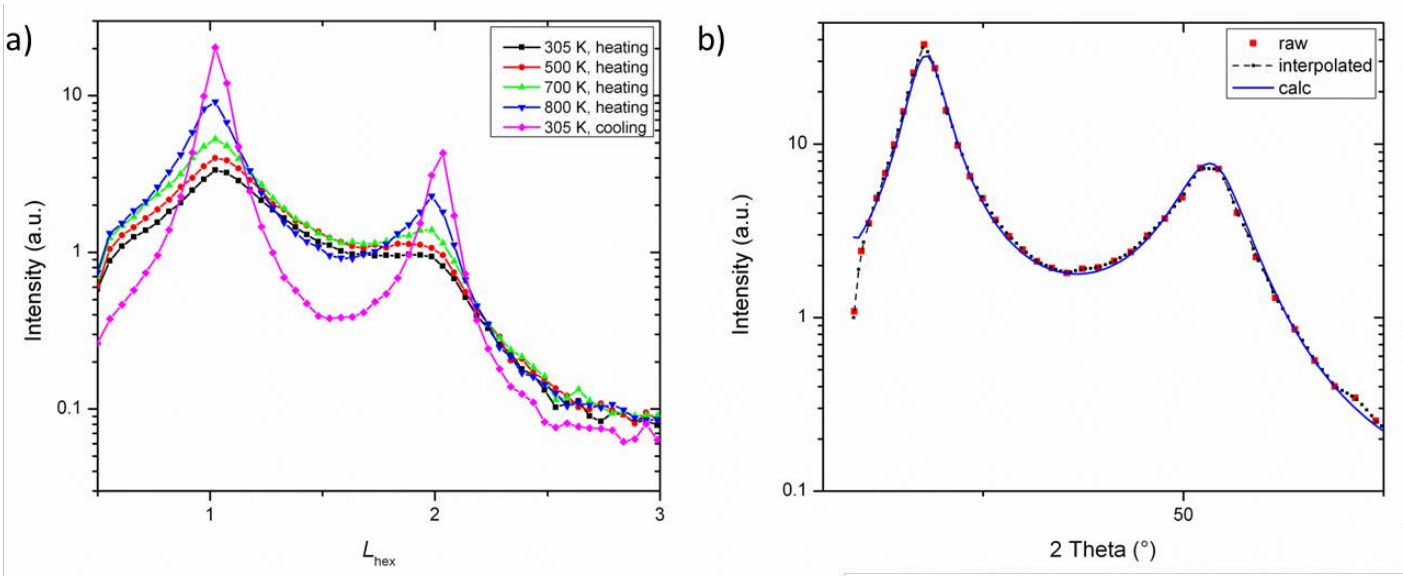

*Figure 2: (a) Diffracted intensity of a NiW12 film along the investigated streak in the reciprocal space at different temperatures during heating and cooling. Obviously, the peak maxima sharpen at temperatures above 700 K, suggesting detwinning processes. For the sake of clarity, not all temperatur steps are plotted. (b) Intensity distributions of a NiW12 film along the investigated streak, which was heated 1 h at 800 K, and the corresponding fit by DIFFaX*plus *indicating a twin probability of 17 %.*

Implication of the Results

We could demonstrate that it is possible to measure twin probabilities in-situ during heating and cooling. Moreover, reliable and meaningful measurements of ex-situ annealed samples could be performed, i.e. the measurement method is robust. The measurements indicate that the thermal stability of nanotwins is strongly influenced by the W content. Further and more detailed evaluation and interpretation are in progress.