ESRF	**Experiment title:** In-situ twin density measurements upon thermal treatments of highly planar-faulted, nanocrystalline Ni(W) thin films	**Experiment number**: MA-1790
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Report:

The beamtime was a continuation of the experiment MA-1508, performed in June 2012.
The investigated Ni(W) films are of interest due to their peculiar microstructure: The magnetron-sputtered {111} fiber-textured films exhibit columnar grains with a high density of planar faults aligned perpendicular to the growth direction. These planar-fault structures generate diffuse scattering along special directions in the reciprocal space (see Figure 1) and the intensity distribution along these streaks reveals the planar-fault density. Since highly planar-faulted, so-called nanotwinned, films exhibit improved mechanical properties, their density is of practical interest, in particular their evolution during thermal treatments. The thermal stability of nanotwins in Ni(W) films with different W contents was investigated by in-situ XRD measurements.

Experimental Procedure

A Rh-coated Si mirror, the Si(111) double crystal monochromator and a Rh-coated toroidal mirror were used in the primary beam path, setting the photon energy to 16 keV, and a linear detector Mythen (Dectris Ltd., Switzerland) in the secondary beam path. The diffuse scattering along a special direction in the reciprocal space was measured in the PSIC mode using the hkl scan method as visualized in Figure 1. The coloring indicates different locations on the streak, measured at different 2Θ-Ψ pairs. The step size on the L_{hex} scale was 0.01 or 0.02. Three Ni(W) films with different W contents were investigated: 9, 17 und 25 at.% W. The in-situ heating experiments were performed by the use of an Anton Paar (Graz, Austria) DHS 900 chamber utilizing a flowing reductive gas containing 2 vol.% H_2. After evacuating and flushing the chamber three times, the samples were heated in steps of 50 or 100 K up to 800 K and after an annealing time of 1 h they were cooled down again to room temperature. At each temperature step, the temperature was held constant for the measurement time of about 20 min. Besides an additional annealing time of 1 h at 750 K for the film containing 25 at.% W due to irregularities in the synchrotron radiation stability, the three different films experienced the same temperature program.

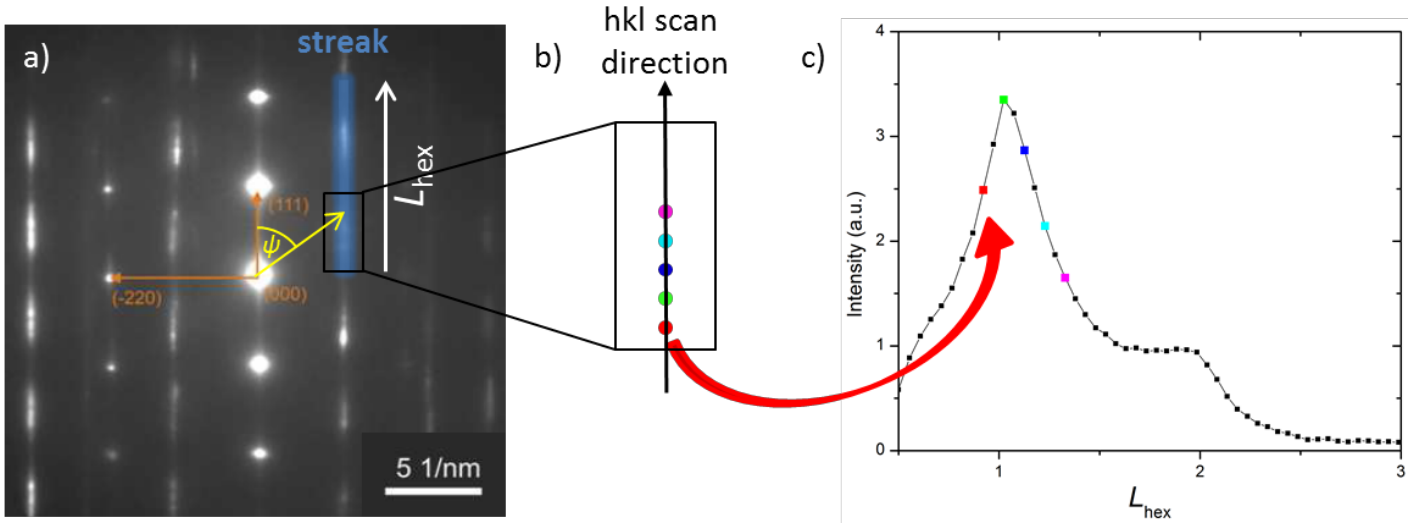

Figure 1: Illustration of the XRD measurement of the intensity distribution along the streak in the reciprocal space; a) electron diffraction pattern of a Ni(W) film showing the streaks in the reciprocal space; b) direction of the hkl scan, each color corresponds to a different 2Θ-Ψ pair; c) resulting intensity distribution on the L_{hex} scale.

Experimental Results

The measured intensity distributions were analyzed by the software DIFFaX*plus* using two different sophisticated transition matrices to describe the extraordinary stacking sequences. The analysis was only performed if a change of the intensity distribution occurred compared to the measured intensity distribution of the temperature step before. The resulting degree of hexagonality is plotted in Figure 2 as a function of temperature. Obviously, the film containing only 9 at.% W shows distinct detwinning, whereas the film containing 25 at.% W does not change its stacking sequence considerably. The film containing 17 at.% W tends to detwinning but at temperatures about 200 K higher than observed for the film containing 9 at.% W.

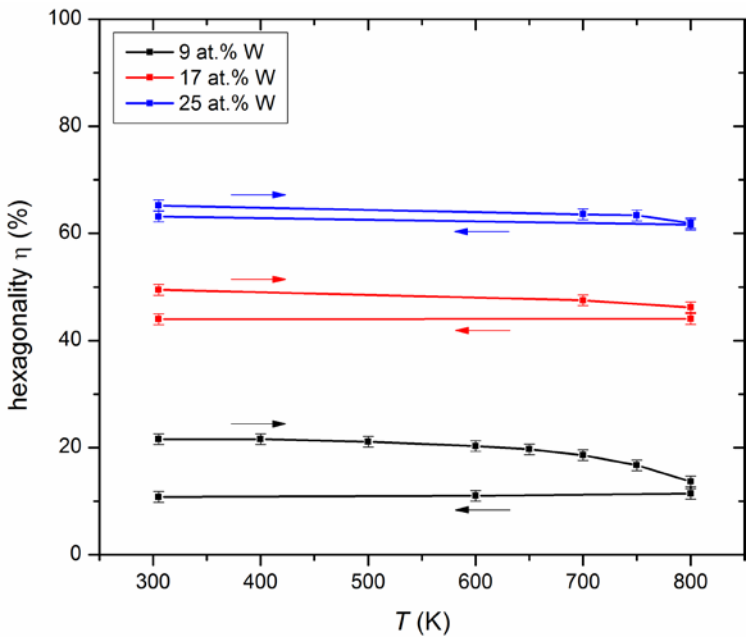

Figure 2: In-situ measurement results: Degree of hexagonality as a function of temperature for the three investigated Ni(W) films.

Implication of the Results

The here-described analysis method, measuring intensity distributions along a streak and interpreting those with DIFFaXplus, is a very convenient method to determine planar-fault densities even in-situ. The used method and further results will be thoroughly discussed in a paper being submitted soon.