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# Characterization of nanometals

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This talk deals with experimental characterization of nanometals. The internal structure of nanometals is highly diverse, consisting of different types of internal interfaces, spatially arranged in different morphologies. Therefore detailed characterization often using different experimental techniques are required to quantify the key microstructural parameters and to understand the microstructural evolution during for example annealing. Also new experimental techniques may be required to address specific scientific questions. These aspects are dealt with in this talk by 3 examples:

## Steel wires with extreme strength

The strength of a metal can be increased many times by cold deformation and for steel it can be raised more than 10 times and reach 5 GPa in a steel wire – the strongest metallic material in the world. The high strength has its cause in the nanometer scale lamellar structure within the wire, see Fig 1. By means of HREM the fundamental strengthening mechanisms have been identified as strain hardening, grain boundary hardening and solid solution strengthening, and they have been found to be linear additive [1]. These microstructural observations underpin the strength-structure relationship found experimentally.

## Three-dimensional orientation mapping in the transmission electron microscope

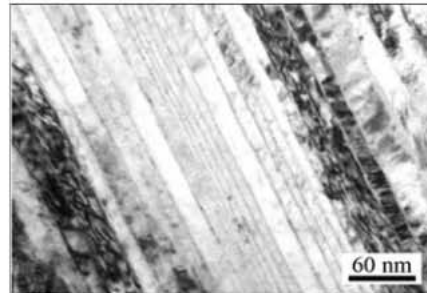
Most materials structures are 3D structures, and over the past decade lots of efforts have been on developing techniques for 3D mapping, examples are the 3D x-ray diffraction (3DXRD) microscope [2] and the 3D polychromatic x-ray micro diffraction technique [3]. Both techniques have spatial resolution of hundred(s) of nanometers. To describe nanometals this is not sufficient. Therefore, a new so-called 3DOMiTEM technique that enables 3D orientation mapping in the TEM with a spatial resolution down to 1 nm has been developed [4]. The data collection is based on conical-scanning dark-field imaging. The complete 3D crystal orientation map of all grains is obtained by recording images at many sample tilt angles. The 3D reconstruction required a development of new algorithms originally developed for 3DXRD. A 3D map from an Al film sample is shown in Fig 2. The volume that can be mapped is typically 100-300 nm in thickness times the illuminated area (e.g. tens of square of micrometers). The potentials of the techniques have been demonstrated and further work is underway to consolidate the technique.

## Local boundary migration

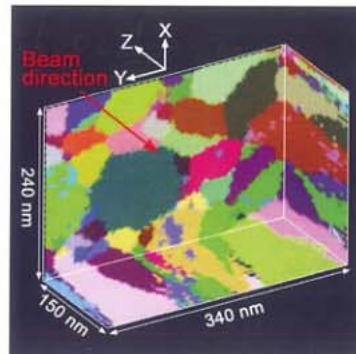
With the aim of identifying mechanisms and key parameters which guide the development of nanostructures with improved thermal stability, grain boundary migration during recrystallization has been characterized by EBSP and ECC SEM in cold rolled pure aluminium. In-situ as well as ex-situ experiments have been performed [5]. It is found that many local protrusions/retrusions on the migrating boundary develop by faster/slower movement of small boundary segments for short periods of time. Contradictory to common assumptions, it is observed that protrusions/retrusions can provide a local driving force which is comparable in magnitude to the driving force from stored energy in the deformed matrix. The formation and evolution of protrusions/retrusions can be directly related to the deformed microstructure but the SEM/TEM investigations need to be supplemented by direct non-destructive 3D studies to quantify such relationships. Measurements of this kind is underway using the 3D polychromatic x-ray micro diffraction technique.

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**Figure 1.** Microstructure of steel wire showing the lamellar structure.



**Figure 2.** Grain map obtained by 3DOMiTEM from a 150 nm thick aluminium film specimen. The colours represent different crystal orientations.