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Three-dimensional imaging and metrology of yttria dispersoids by electron tomography

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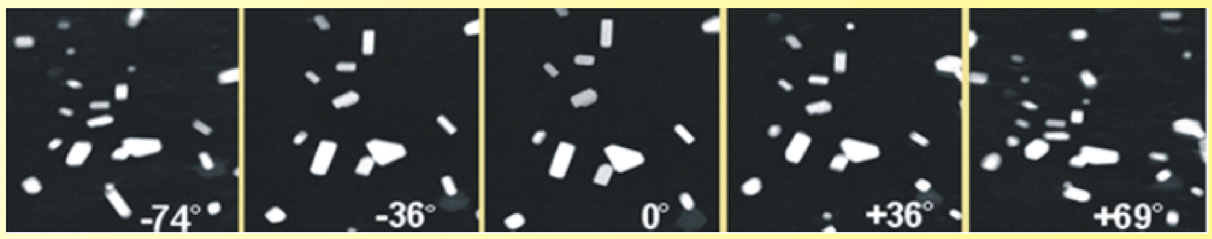
ABSTRACT

Microstructure of the Oxide Dispersion Strengthened (ODS) alloys for high temperature application was studied mainly by scanning- and transmission electron microscopy (SEM, TEM). The precise qualification of the shape, size and chemical composition of dispersoids (mostly Y-Al oxide particles), which are the strengthening particles of these materials, is of great importance for determination their influence on material properties. Electron tomography allows for generation of 3D model (image) of the investigated object(s) from the multiple 2D projection images, obtained over a range of viewing directions. Promising results were achieved using electron tomography with HAADF-STEM imaging. Therefore another tomography technique, FIB tomography, was used for detailed characterisation and metrology of Y-Al dispersoids in the Incoloy MA956 alloy.

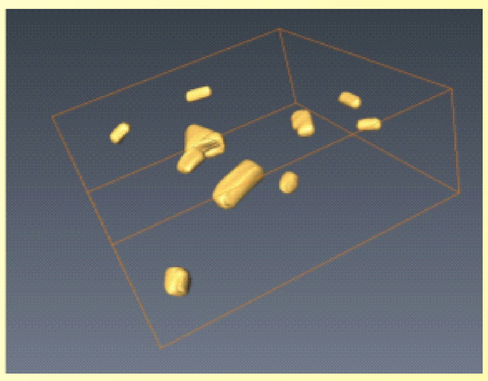
MATERIAL & EXPERIMENTAL PROCEDURE

HAADF-STEM Tomography

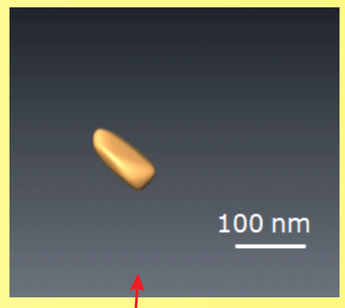
Electron tomography studies of the Y-Al dispersoids in ferritic ODS alloy, Incoloy MA956, after isothermal annealing at 1350 C up to 1000 hours was performed. The samples of dispersoids were prepared as extracted double-replicas. HAADF-STEM technique was used to imaging of Y-Al oxide particles. Tilt series in the range -74deg to +74 deg with 2 deg step were acquired using FEI Tecnai G^2 microscope with Xplore 3D software. Digital images were recorded with Gatan 794 CCD camera. Tilt series images were aligned to a common tilt axis by cross-correlation and the 3D surface was reconstructed with SIRT technique.

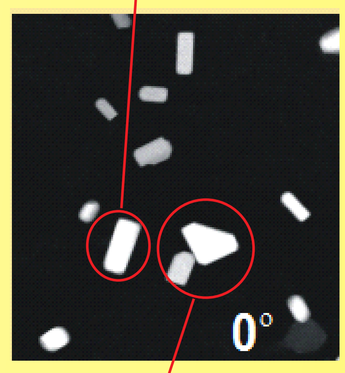


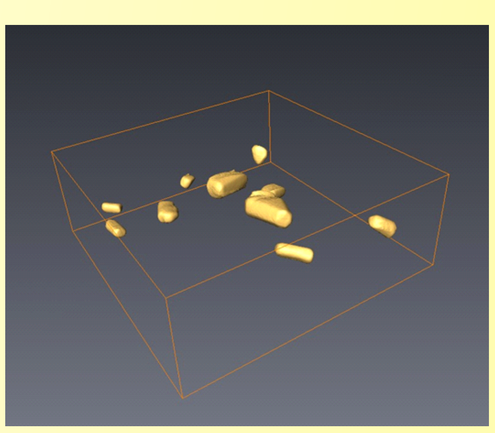
HAADF-STEM tilt series of Y-Al oxide nanoparticles images



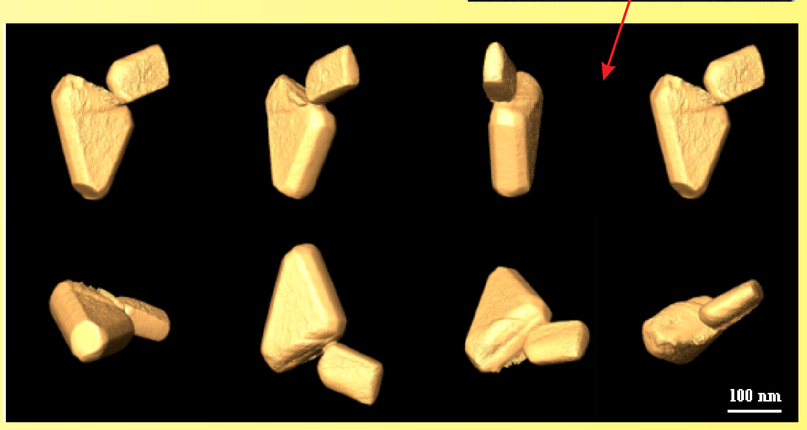
Reconstruction & 3D visualisation



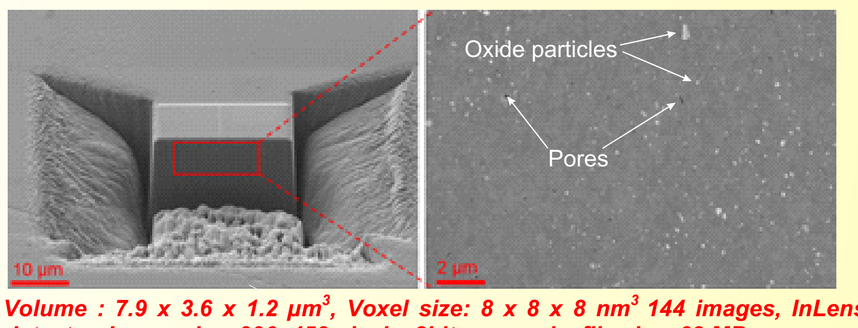




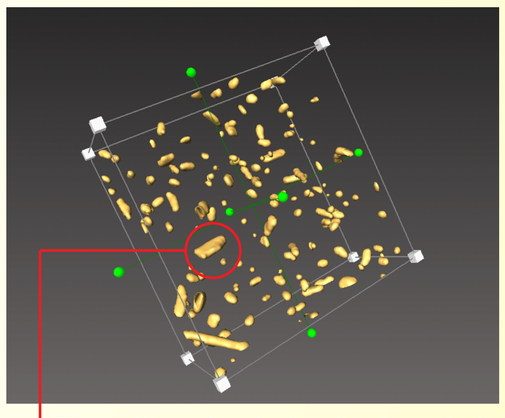
3D visualization of reconstructed volume containing oxide nanoparticles



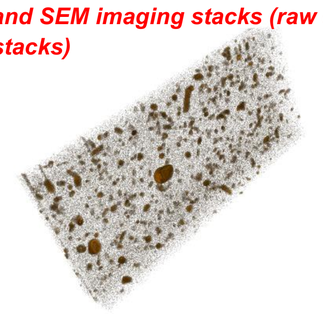
3D visualisation of reconstructed oxide particles



Volume : 7.9 x 3.6 x 1.2 µm^3, Voxel size: 8 x 8 x 8 nm^3 144 images, InLens detector, Image size: 996x453 pixels, 8bit grayscale, file size: 62 MB

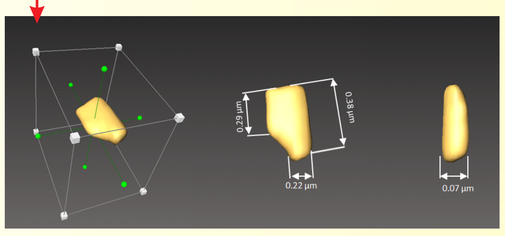


3D visualization of FIB serial slicing and SEM imaging stacks (raw data stacks)

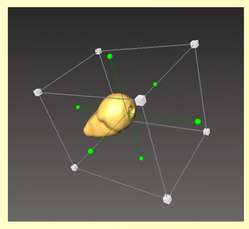


Volume : 7.9 x 3.6 x 1.2 µm^3

Three-dimensional visualisation of shape and spatial distribution of oxides particles in Incoloy MA956. Voxel size in the raw data volume is 8 x 8 x 8 nm



3D visualisation of reconstructed single yttria oxide particle in Incoloy MA956



3D visualisation of reconstructed single pore in Incoloy MA956

FIB/SEM Tomography

FIB (Focused Ion Beam) tomography is based on a serial sectioning procedure employing a FIB/SEM dual beam workstation Zeiss NEON 40EsB CrossBeam equipped with a FIB column (Ga ions). Repeated removal of layers as thin as a few nm allows to explore at total a volume of some µm^3 with a voxel size as 8 nm × 8 nm × 8 nm. 3D mapping of dispersoids by serial FIB slicing and SEM imaging was performed using a 30 kV Ga+ ion beam to get a precise in-situ milling. The SEM images at accelerating voltage 1.7 kV were taken with using Inlens detector. The 3D reconstruction and visualization led to observe several types of dispersoids present in the matrix and allowed for precise measurement of their size. To visualize the 3D reconstruction, the Avizo 6 and FIJI 1.44p software was used. Filtering and shift correction performed on the images using cross-correlation method allowed for 3D visualization of the reconstructed volume.

SUMMARY

HAADF-STEM tomography methods provide quantitative data about microstructure, size, shape and distribution of the particles and serve complementary information about microstructure features to TEM investigated.

FIB/SEM tomography has become an important tool for visualization and metrology of precipitates in micro- and nanometer scale.

The results achieved confirm the ability of HAADF-STEM and FIB/SEM tomography to get 3D reconstruction of the objects of 100 nm or even smaller. Such 3D reconstructions can serve as a basis for quantitative analyses of complex structures in materials down to the nano-scale.

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