3D characterization and metrology of oxide nanoparticles in ODS alloy by electron tomography

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Keywords: ODS alloy, Y-Al oxide, Electron Tomography, HAADF-STEM

Mechanically alloyed ferritic Oxide Dispersion Strengthened (ODS) alloys were extensively studied as promising materials for high temperature components operating in aggressive environments. These alloys combine a high creep resistance, due to the incorporation of Y-Al oxide nanoparticles (dispersoids) in a Fe-Cr-Al matrix, with excellent oxidation resistance at high temperatures. The studies of thermal stability of dispersoids in INCOLOY MA956 and PM 2000 type alloys showed that they are less susceptible to coarsening than the precipitates in conventional superalloys and remain stable as fine particles up to 1150ºC [1,2].

The precise qualification of the shape, size and chemical composition of dispersoids is of great importance for determination their influence on material properties at high temperatures. Images observed in TEM are two-dimensional; they are a “shadow” of the real objects, which are three-dimensional. Therefore information about the investigated object(s) and its shape, obtained from TEM is incomplete, what sometimes may lead to inaccurate or even wrong conclusions. 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.

For the tomography of the metallic materials, the BF imaging fails, because of a diffraction contrast and its changes during tilting of the specimen in TEM during image acquisition. Application of HAADF-STEM, allows to overcome this problem. HAADF detector collects only electrons which are not Bragg scattered, the recorded intensity is proportional to Z^2, moreover – image contrast depends monotonic on object thickness [3].

The samples of dispersoids were prepared as extracted double-replicas (**Figure 1a**). HAADF-TEM method was used for image acquisition. Tilt series were acquired semi-automatically at 200 kV on an FEI Tecnai G^2 microscope using FEI 3D software. All tilted images were aligned to a common tilt axis using cross-correlation; oxide Al/Y ratio was determined by EDX.

Morphology of the particles, their chemical composition, Al/Y ratio (**Figure 1b**) and its changes during isothermal annealing at 1350°C for 600 and 1000 hours were investigated. HAADF-STEM tilt series of images are presented on the **Figure 2a**. The obtained results show that several different types of nanoparticles are present in the matrix of the investigated alloy, and that there is a dependency between their chemical composition and their shape. **Figure 2b** presented 3D visualization of the oxide particles as a result of electron tomography application to particle shape determination.

DOI: 10.3217/978-3-85125-062-6-049,

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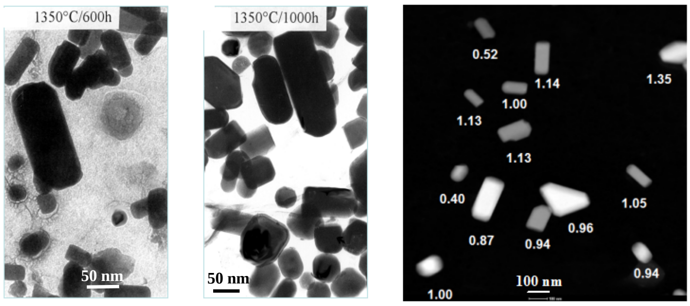
4. The financial support from the European Union under the Framework 6 program, contract for an Integrated Infrastructure Initiative, reference 026019 ESTEEM, is kindly acknowledged.

Valuable contribution of Prof. Paul A. Midgley to electron tomography investigations is gratefully acknowledged.



a) b)

Figure 1. Y-Al oxide nanoparticles, a) TEM - extracted double-replica, b) HAADF-STEM images of oxide particles and EDX Al/Y ratio.

a)

b)

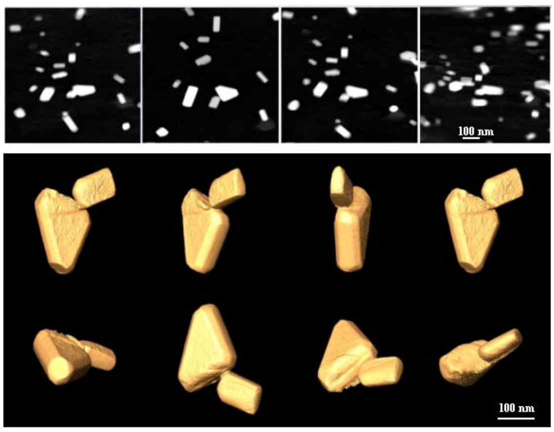


Figure 2. Y-Al oxide nanoparticles. a) HAADF-STEM tilt series of images. b) 3D visualization of oxide particles.

G. Kothleitner, M. Leisch (Eds.): MC2009, Vol. 1: Instrumentation and Methodology, DOI: 10.3217/978-3-85125-062-6-049, © Verlag der TU Graz 2009