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Microstructure and Defect Characterization Using Advanced STEM Techniques: 4D-STEM and WB DF STEM

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Rapid advances in Scanning Transmission Electron Microscopy (STEM) techniques offer material scientists many advanced tools to investigate fine microstructure features of engineering alloys and gain deep insights of underlining deformation and strengthening mechanisms. In this paper, applications of two advanced STEM techniques: 4D-STEM and weak-beam dark-field (WB DF STEM), are presented.

In 4D-STEM, convergent beam electron diffraction (CBED) patterns or nano beam electron diffraction (NBED) patterns at each probe position of a two-dimensional STEM image are recorded [1]. Using 4D-STEM data, nanometer resolution strain mapping in large scale can be achieved [2] Conventional defect characterization is normally conducted in TEM mode using parallel beam illumination. Recently, a new defect imaging technique: WB DF STEM, was developed [3]. As compared with conventional WB DF TEM imaging, WB DF STEM technique can provide similar high resolution. Moreover, bend contours can be efficiently suppressed and thicker specimens can be imaged in WB DF STEM mode.

In this work, 4D-STEM test was conducted in a FEI Titan 80-300 equipped with a Gatan K2-IS direct electron detection camera. All 4D-STEM datasets were processed using Gatan Digital Micrograph software package and custom MATLAB codes. Polycrystalline nickel-based superalloy Inconel 718 and ME3 were used in current 4D-STEM study. WB DF STEM test was carried out using a FEI Tecnai TEM/STEM operating at 200keV. An equiatomic NiCoCr solid solution alloy was used in WB DF STEM study. Details related to the mechanical testing of this alloy and TEM sample preparation can be found elsewhere [4].

Figure 1 shows an example of strain mapping using 4D-STEM to reveal the distribution of misfit strain between γ'' precipitates and γ matrix in nickel-based superalloy Inconel 718. Strain maps in Figure 2 shows the increase of negative misfit strain between γ' precipitates and γ matrix in superalloy ME3 with increasing test temperature using in-situ 4D-STEM technique. Figure 3 shows examples of using WB DF STEM in imaging deformation substructures in NiCoCr alloy. The WB DF STEM images were captured using g $=\{2\bar{2}0\}$ type diffraction vectors with the center of 3g set to satisfy Bragg's condition. Due to the low stacking fault energy of this alloy, compact dislocations can easily dissociate into partial dislocations as showing in Figure 3 a). The reaction of dissociated dislocations on different slip planes can form Lomer-Cottrell (L-C) locks as indicted in Figure 3b). The formation of deformation twinning and deformation induced hexagonal close-packed (HCP) phase in this alloy is also due to the cross slip of dissociated partial dislocations. Those deformation substructures act as strong barriers to dislocation slip and can further partition grains, resulting in dyanmic Hall-Petch strengthening and contiributing to the exceptionally large work hardening rates and ductitlity of this alloy [4,5].

References:

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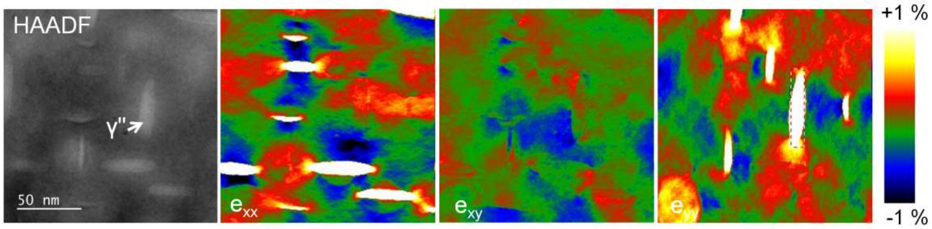

Figure 1. 4D-STEM strain maps of nickel-based superalloy Inconel 718

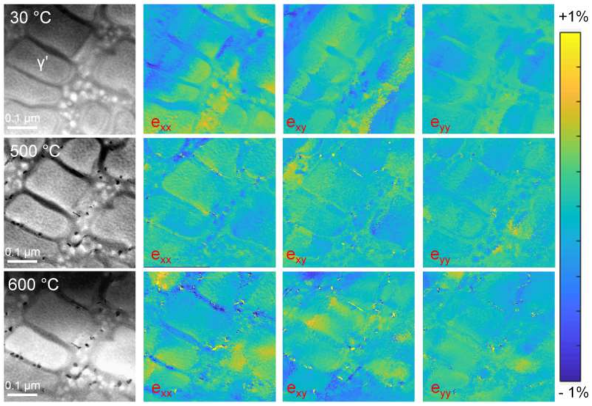

Figure 2. In-situ 4D-STEM strain maps of nickel-based superalloy ME3 at different temperature

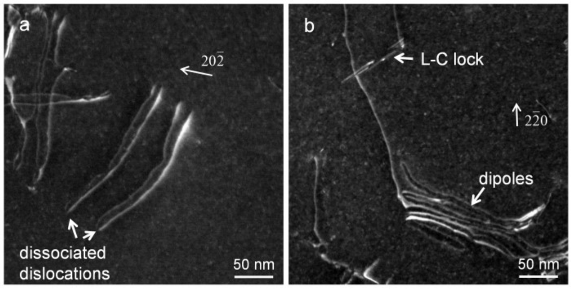

Figure 3. WB DF STEM characterization of deformation substructures in NiCoCr alloy: a) dissociated dislocation, and b) L-C lock and dislocation dipoles.

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