Microstructure evolution of austenitic ODS steel during high-temperature low-cycle fatique

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In this work we studied the microstructure evolution of austenitic oxide dispersion strengthened (ODS) steel during low-cycle fatigue at temperature of 650 °C under different strain amplitudes (±0.4% and ±0.7%) with a nominal strain rate of 10^{-3} s^{-1}.

The microstructure of the samples was characterized before and after cyclic straining using Tecnai 20 FEG microscope. High angle annular dark field (HAADF) scanning TEM (STEM) with energy-dispersive X-ray (EDX) spectroscopy for the determination of the composition, conventional TEM with selected area diffraction and high resolution TEM (HRTEM) with fast Fourier transformation (diffractogram) analysis for the crystallographic investigations were applied.

Figure 1 shows inverted HAADF STEM images of the samples, as received (Figure 1a) and after cyclic straining at 650°C under ±0.4% (Figure 1b) and under ±0.7% (Figure 1c) strain amplitude. The grain sizes of the as received steel in Figure 1a are in the range of 200-2000 nm. The typical observed defects in the sample are dislocations (D) or twins (TW), but most grains are almost defects free. The sample contains precipitates with mainly a bright contrast (up to 70 nm in diameter), distributed inside the grains as well as at the grain boundaries. The HRTEM and diffractogram analysis (Figures 1a and 1b) combined with detail EDX measurements (here not shown) reveal that ODS particles have mainly a composition of $Y_2Ti_{2-x}O_{7-2x}$ with pyrochlore *fcc* structure (Figure 2a), however some particles are Y_2O_3 with *bcc* structure (Figure 2b).

After cycling at ±0.4% strain amplitude (Figure 1b) more regions with defects are appeared. Due to strain, some grains manifest short dislocation (SD) or loops or stacking faults (SF). After cycling at ±0.7% strain amplitude, we observe increasing density of defects (Figure 1c).

Figure 3 shows inverted HAADF STEM images of the samples after cyclic straining at 650°C under ± 0.4% (Figure 3a) and under ±0.7% (Figure 3b) strain amplitude. In almost all grains individual dislocations appear pinned at the nanoparticle-matrix interfaces and under the effect of shear stresses, most of them are bowed out (insert in Figure 3a). This indicates an attractive particle-dislocation interaction since pinning is realized on both arrival as well as departure side of the nanoparticles. Therefore, there exists a threshold stress to detach dislocation out of this pinning, which must be overcome for the dislocation to glide; and therefore, plastic deformation to occur. This results in a retarded dislocation's mobility, and hence, inhibiting strain localization by prohibiting 3D dislocation structures formation. Moreover, dislocation pinning provides a consequent strengthening which give ODS steels their superior monotonic as well as cyclic strength over non-ODS counterparts.

As it shown in Figure 3b mostly intragranular dislocation activity is observed along with the occasional dislocations pile-up DPU (DPU). However, no noticeable three-dimensional (3D) dislocation arrangements such as the cells, ladder, vein, labyrinth and persistent slip bands were identified. These typical 3D dislocation structures are usually associated with the formation of extrusions and intrusions where fatigue cracks nucleate. Hence, their absence reveals that oxide dispersion strengthening assists in improving cyclic properties.

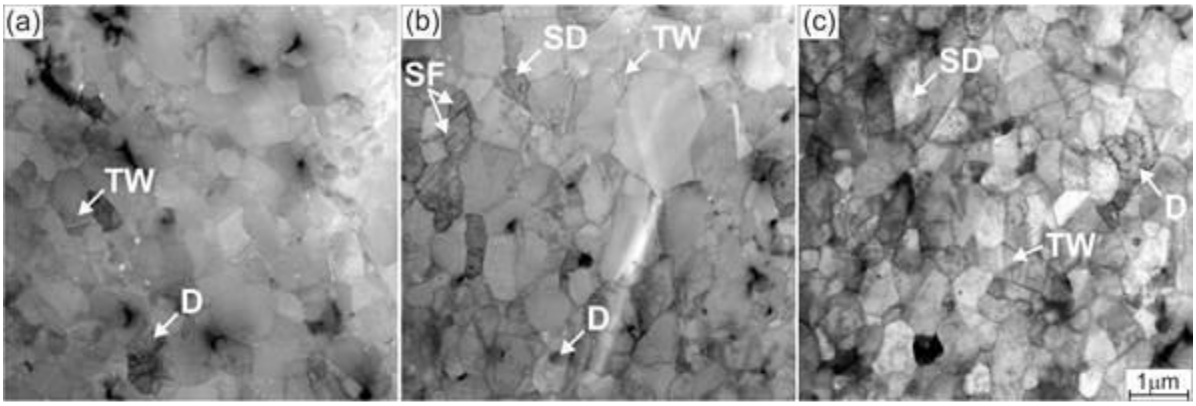

Figure 1. Inverted contrast HAADF-STEM micrographs reveal microstructures (a) before (as-received state) and (b) after cyclic straining at 650°C under ±0.4% and (c) under ±0.7% strain amplitude. By arrows are marked: D - dislocations, TW- twins, SD - short dislocations or loops, SF - stacking faults.

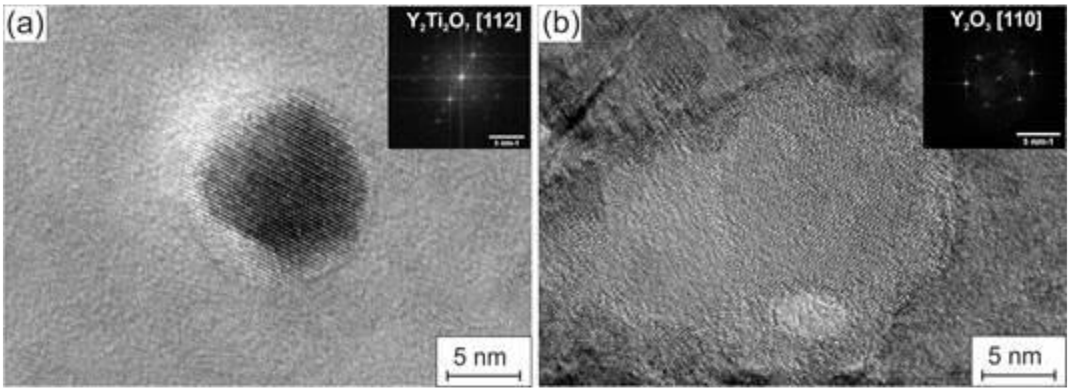

Figure 2. HRTEM images of ODS particles with corresponded diffractograms

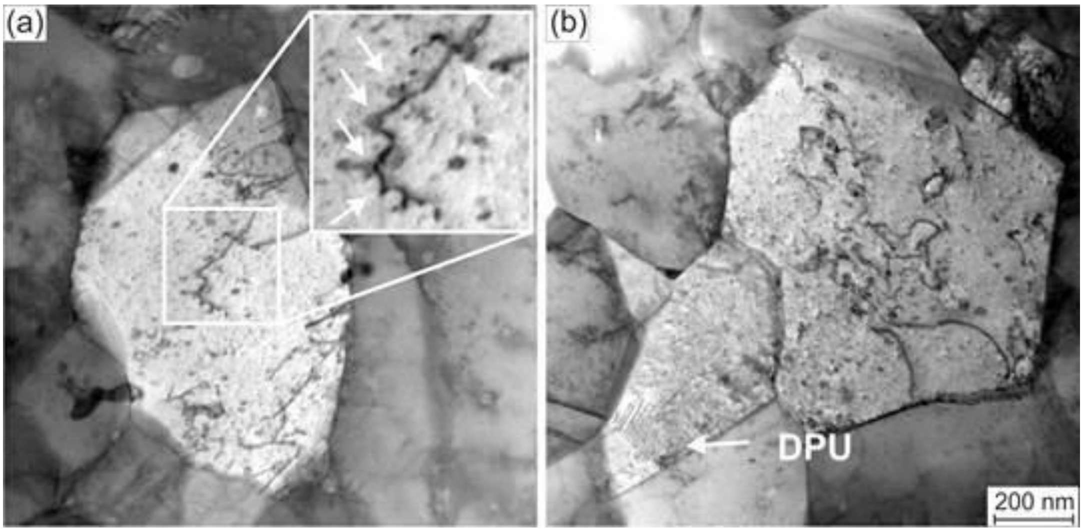

Figure 3. Inverted contrast HAADF-STEM micrographs revealing dislocations structure after cyclic straining at 650°C under ±0.4% (a) and ±0.7% (b) strain amplitudes. Insert in (a) shows pinned dislocation at various nanoparticle-matrix interfaces (arrows). In (b) by arrow is marked dislocations pile-up (DPU).