Microstructural changes accompanying high strain amplitude fatigue tests on a multiphase medium carbon microalloyed steel

S. SANKARAN, GOUTHAMA, S. SANGAL
Department of Materials and Metallurgical Engineering, Indian Institute of Technology, Kanpur-208 016, India

K. A. PADMANABHAN
South Asia International Institute, Mahavir Chambers, Liberty Crossroads, Hyderabad-500029, India

Due to cost effective processing, medium carbon microalloyed steels are replacing quenched and tempered alloy steels in automotive applications. To be able to substitute alloy steels, microalloyed steels must be thermomechanically processed to similar strength levels, along with an acceptable level of toughness [1]. Recently a multiphase microstructure was obtained in a medium carbon microalloyed steel through a two-step cooling and annealing procedure following controlled forging. The tensile and the low cycle fatigue (LCF) behavior in this condition is now established [2, 3]. It is known that such a high strength multiphase steel displays cyclic softening during fatigue testing, especially under high total strain amplitudes. It was also established that this multiphase steel is cyclically stronger than the same material having a pearlite-ferrite or tempered martensite microstructure [3]. In this paper, we present the results of a transmission electron microscopic study of a multiphase microalloyed steel subjected to low cycle fatigue loading at high strain amplitudes. Evidence for the microstructural changes that result from fatigue loading, recovery-assisted crystal re-orientation is presented.

The material used was an automotive grade medium carbon MA steel 38MnSiVS5. To produce the ferrite-bainite-martensite (F-B-M) microstructure, a two-step cooling and annealing (TSCA) treatment was adopted. In this process, the as-received rods of 30 mm diameter were reheated to 1200 °C for 1 hr and forged to 25 mm diameter (finish forging temperature ∼825 °C). Following forging, the rods were air cooled to 710–725 °C and quenched in water. (The temperature was always measured by a digital infrared pyrometer.) This was followed by an annealing treatment at 450 °C for 90 min.

LCF tests were carried out on a 100 kN MTS closed loop servo-hydraulic materials testing machine using specimens prepared as per ASTM E 606-80. After machining, the specimens for the LCF tests were polished with emery paper (320 to 800 grit) followed by chemical polishing in a mixture of chromic acid and sulfuric acid (500 g of chromic acid in 150 cc of sulfuric acid). To achieve a good surface finish, specimens were immersed in this bath for a minimum period of 3 hrs. LCF tests under total strain control with total strain amplitudes lying between 0.4 and 0.9% were conducted using a triangular strain waveform with zero mean strain at a constant strain rate of $2 \times 10^{-3} s^{-1}$. For TEM investigation, regions along the gauge length within 3–4 mm from the fracture surface were sampled. Fig. 1 presents the manner in which the slicing was made and 3 mm diameter discs were punched out. The sectioning/slicing operations were carried out using a slow speed diamond saw. The discs were taken from just below the fracture surface, and electrolytically thinned using a twin-jet polishing unit with an electrolyte consisting of a mixture of 10% perchloric acid and 90% acetic acid. The thinned foils were examined using a Jeol model JEM 2000FX II transmission electron microscope operating at 160 kV. From Fig. 1 it is clear that the tensile axis during fatigue loading was parallel to the plane of the disc (thin foil).

The chemical composition of the MA steel 38MnSiVS5 is given in Table I. The two-step cooling and annealing treatment produced a multiphase microstructure predominantly consisting of polygonal ferrite (PF) and tempered granular bainite (GB)/martensite (TM). The variation in the peak tensile stress with the number of cycles to failure is shown in Fig. 2 for a sample tested at a total strain amplitude ($\Delta\varepsilon_t/2$) of 0.7%. The steel did not show a stable response or a saturation stress level till fracture.

A transmission electron micrograph of the low cycle fatigue tested sample displaying the typical microstructure of a polygonal ferrite grain and its selected area diffraction pattern (SADP) are given in the Fig. 3a and b, respectively. The SADP analysis indicated that it consists of an overlap of patterns from two different ferrite crystal orientations, viz., $[\bar{1}13]_\alpha$ and $[\bar{1}11]_\alpha$. The spots in the pattern corresponding to zone $[\bar{1}11]_\alpha$ are sharper compared to those from the zone $[\bar{1}13]_\alpha$ which indicates the smaller degree of inelastic scattering of electrons from the former region. This means that the region responsible for the zone $[\bar{1}11]_\alpha$ pattern is relatively strain-free. The microstructural features of the corresponding area are illustrated in Fig. 3a. Here, the central region is occupied by a grain, which shows slip band traces. We shall refer to the central region responsible for the $[\bar{1}11]$ zone pattern as the *re-oriented grain* and the region of the polygonal ferrite showing the dislocation cell structure as the *parent ferrite grain.* The symbols

TABLE I Chemical composition (wt.%)

Material	C	Si	Mn	P	S	V	N	Cr	Ti	Fe
38MnSiVS5	0.38	0.68	1.5	0.022	0.06	0.11	0.006	0.18	–	Balance

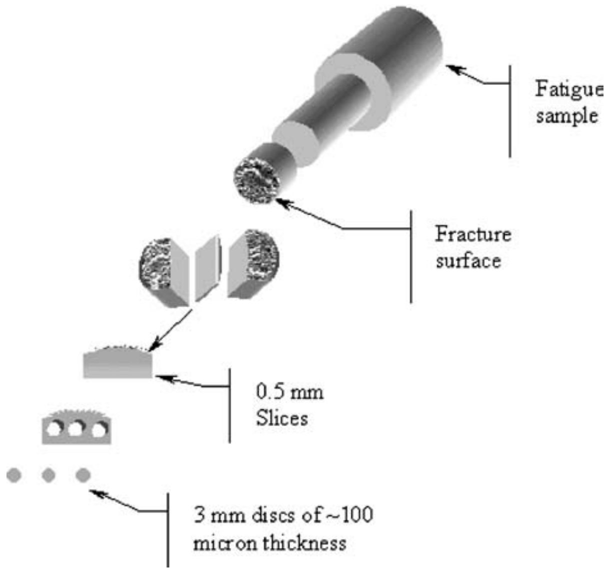


Figure 1 Schematic showing the location of the disc used for the TEM analysis.

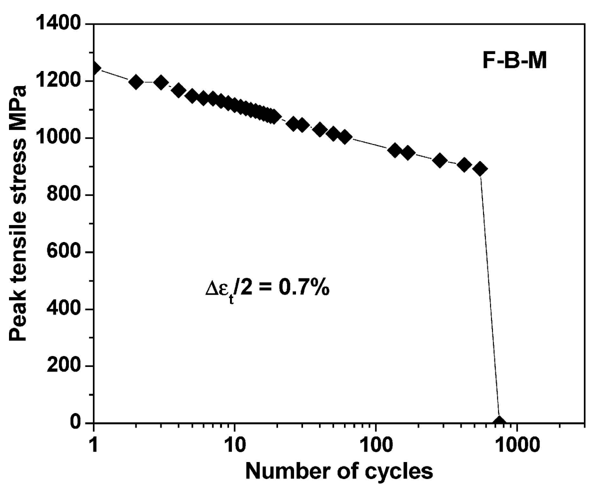
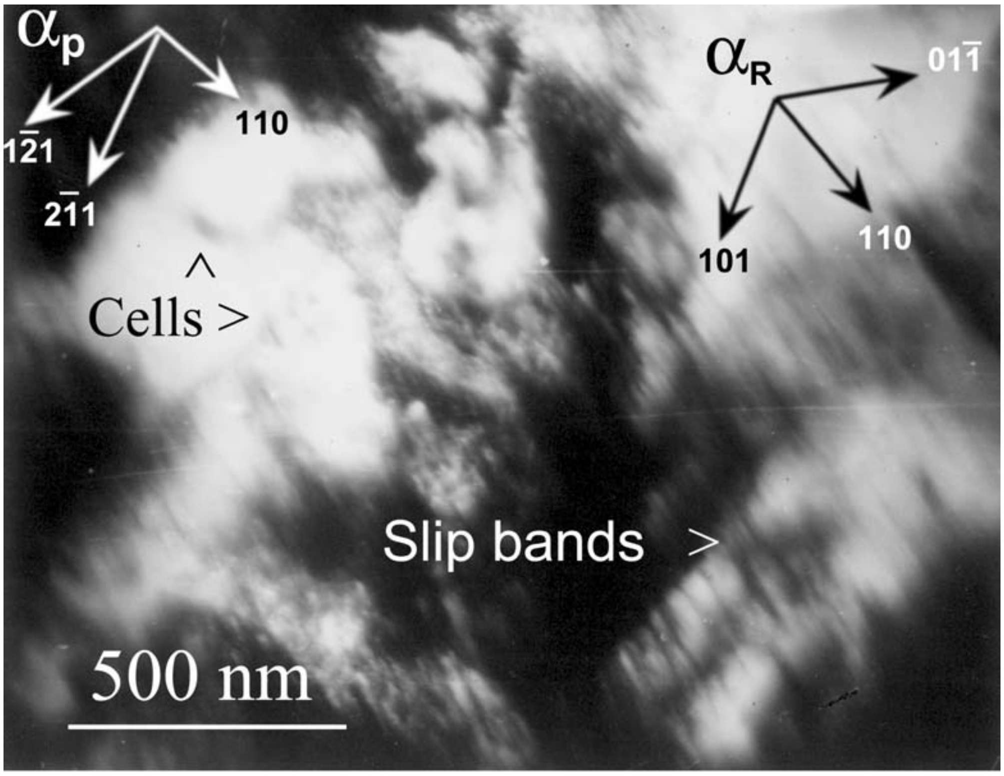


Figure 2 Cyclic stress response at a total strain amplitude $\Delta\varepsilon_t/2 = 0.7\%$ for the F-B-M microstructure.

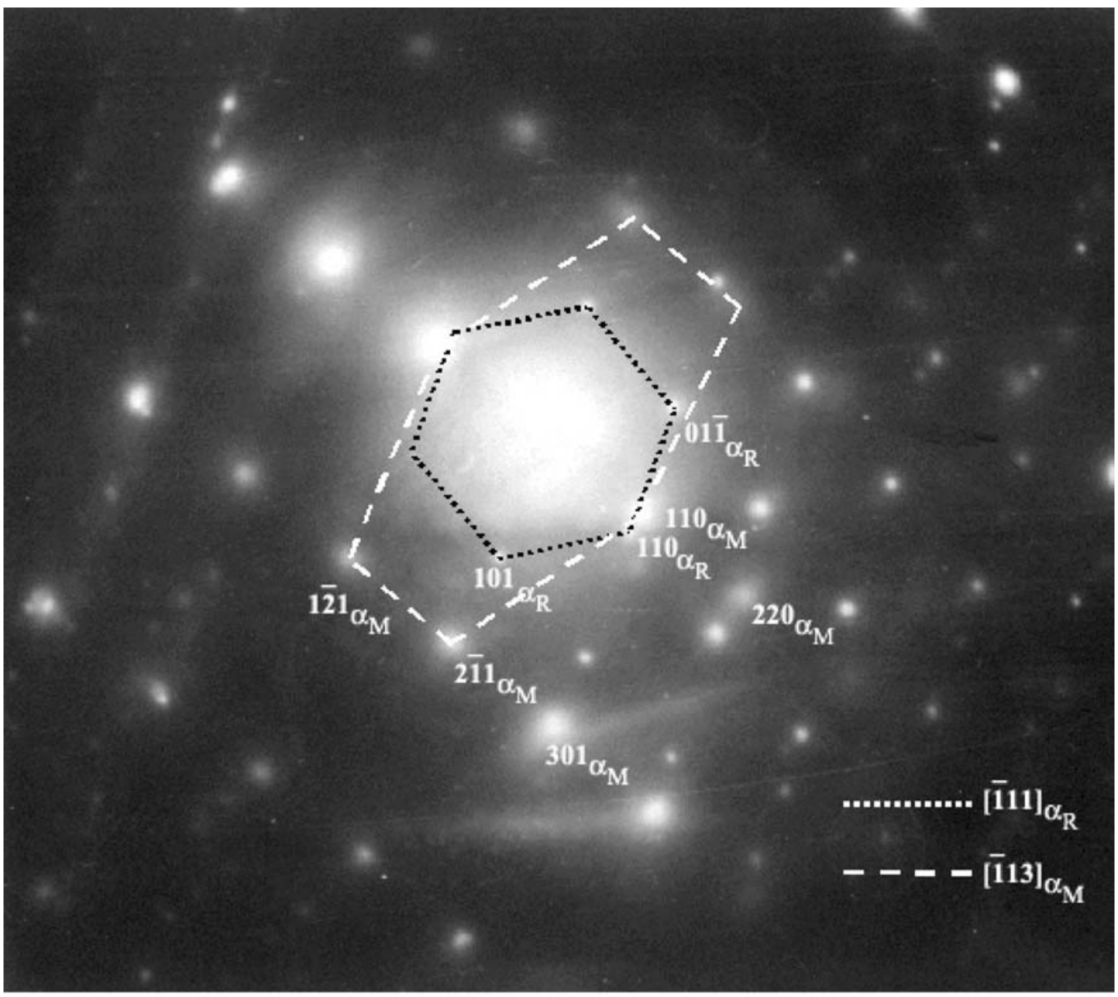
α_R and α_P respectively are used to refer to them. (The justification for these nomenclatures is presented in the next section.) The crystallographic directions associated with each of these regions, as deduced from the SADP, are indicated. The angle between $[110]\alpha_R$ and $[110]\alpha_P$ was measured to be $\sim 9^\circ$. Also note that the traces of slip bands in the *re-orientated grain* coincide with the $[110]\alpha_R$ direction.

Fatigue softening is a manifestation of the microstructural changes that take place during cyclic loading. The multiphase (ferrite-bainite-martensite) steel consists of predominantly non-equilibrium (metastable) microstructures, which display severe softening at high total strain amplitudes (see, e.g., Fig. 2) during fatigue testing till fracture [3]. In a study on a low carbon steel [4], two basic dislocation processes were identified as responsible for the changes in the substructure that lead to fatigue softening: (a) dislocations may cluster together, moving from areas of low density to areas of high density exposing relatively dislocation-free soft regions to fatigue deformation; and (b) annihilation of dislocations by cross-slip of screw dislocations and climb of edge dislocations. In the present study, evidence for microstructural modification that could have played independent and additional role in determining the magnitude of strain softening, *local ferrite grain reorientation,* was obtained.

The observation of a cell structure and dislocation-free regions (Fig. 3a) confirm the operation in multiphase microstructures of the earlier identified processes of fatigue softening [4] points (a) and (b) mentioned in the previous paragraph. In early studies on fatigue in polycrystalline metals [5] it was observed that in certain grains with the '*softest*' orientation for plastic deformation intense bands were formed and slip got localized leading to the so called '*persistent slip bands (PSBs)*'. When the polygonal ferrite in the multiphase microstructure gets cyclically strained, a cell structure forms and dislocation cross slip takes place freely. In such a dynamic environment, there is a possibility that certain grains or sub-grains may have the so called 'softest' orientation, which will experience deformation under cyclic loading. During a fatigue test, dynamic recovery takes place (as may also be deduced from the continuous softening in the peak tensile stress—number of fatigue cycles curve, Fig. 2) and a portion of the polygonal ferrite grains that are in the softest orientation deform into *a new orientation*, which we refer to as 'a *re-oriented* grain'. As only one well defined slip system is observed, one can deduce that for this region in the microstructure the primary slip direction was almost parallel to the easy slip direction with respect to the tensile axis of the test sample. This argument is supported by the fact that, as shown in Fig. 3a, slip band traces coincide with the trace $[110]\alpha_R$, which falls on the great circle of the stereogram containing the $[\bar{1}11]\alpha_R$ direction. Also note that the angle between $[110]\alpha_P$ and $[110]\alpha_R$ is only 9°, which lies within the angular spread of 15°, conventionally accepted as the limit for the misorientation angle between subgrains. This view is also consistant with the idea that reorientation occurred during fatigue testing. The possibility that the region simply has more than one grain which has led to the overlapping pattern seen in Fig. 3b is eliminated by the fact that in such a situation the diffraction spots would have similar systematic intensity gradients. Instead, we find the set of spots corresponding to the $[\bar{1}11]_\alpha$ zone to be rather sharp which is indicative of not only its strain free/recovered state, but also of its origin. The lattice rotation realised during dynamic recovery, especially at such a high total



(a)



(b)

Figure 3 Transmission electron micrographs of a sample fatigue tested to failure at a total strain amplitude of 0.7%: (a) dislocation substructure showing cell formation and slip bands and (b) diffraction pattern showing matrix ferrite orientation $[\bar{1}13]_\alpha$ and reoriented ferrite grain with direction $[\bar{1}11]_\alpha$.

strain amplitude value of 0.7% under cyclic loading, suggests that fatigue strain induced grain/subgrain reorientation is possible and this is what is seen in Fig. 3a. By this reorientation, a situation of a crystal oriented for single slip, considered most ideal for PSB formation is achieved which is of importance in determining the mechanical response during fatigue of a polycrystalline system. The possibility of a reorientation taking place during recovery was hinted at in the past based on X-ray texture analysis of titanium aluminide subjected to a recovery anneal [6], but no concrete evidence could be provided.

Slip reversibility has been found to be a desirable property for fatigue resistance. Planar slip alloys with fine, narrow, slip bands are found to be more resistant to fatigue than materials exhibiting wavy slip [7]. The reoriented grains, with their ideal orientation for cyclic deformation developed due to their formation during cyclic loading, may have an inherently higher fatigue resistance, as then strain localization is avoided. However, the conditions that lead to their formation and their potential in offsetting the fatigue softening in a multiphase system need to be identified in the future.

PSBs have been clearly observed only in single crystals and large grained materials. In a recent study on cyclic softening of ultra-fine grained copper [8] PSBs could not be observed. But an SEM analysis of the surface damage in specimens tested at low plastic strain amplitudes showed typical extrusions and intrusions usually associated with PSBs. In the present study the PSBs were not seen.

The tendency of a microstructure to soften depends on how far removed it is from equilibrium. A large deviation leads to greater rates of softening because the driving force is the energy stored within the material [4]. Effects of number of cycles and stress amplitude on the amount of softening are similar to those of time and temperature on the amount of recovery. Therefore, softening during fatigue may be regarded as cyclic-load-assisted recovery [4]. Earlier investigations [9] have shown that a fatigue strain can be accompanied by recovery by cross slip and dislocation climb. This will give rise to strain softening (dynamic recovery) even at room temperature (in copper alloys). Stored energies of some of the phase mixtures have been reported for a 0.2C steel (total alloy content in that steel was 2%) ferrite and cementite (70 J mol^{-1}), bainite and paraequilibrium cementite (785 J mol^{-1}), martensite (1214 J mol^{-1}) [10]. In the present case the stored energy in the respective phase mixtures is expected to be slightly more as the carbon content of the present alloy is 0.38% and the total alloy content is 2.938%.

In conclusion, a multiphase microstructure, ferrite-bainite-martensite, displayed cyclic softening till fracture (with no saturation) at high total strain amplitude of 0.7%. Transmission electron microscopy of a sample fatigued at this high strain amplitude revealed evidence for usually observed dislocation processes that lead to fatigue softening (dislocation clustering, formation of dislocation free regions and dislocation annihilation). In addition, microstructural modification that can play an independent role in determining the mechanical response, dynamic recovery induced reorientation of ferrite grains was observed.

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