Short Communication

Microstructure evolution mechanism near the fracture lip of 4Cr5MoSiV1 steel during deforming at 580 °C

Yaoli Wang a,b, Kexing Song a,⁎, Yanmin Zhang a,c

a School of Materials Science and Engineering, Henan University of Science and Technology, Luoyang 471023, China
b Collaborative Innovation Center of Nonferrous Metals Henan Province, Luoyang 471023, China
c Henan Key Laboratory of Advanced Non-Ferrous Metals, Luoyang 471023, China

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ABSTRACT

The failure mechanisms under thermal-mechanical conditions have always been a critical issue in hot work steels. However, previous studies mainly focused on the mechanical properties of hot work materials, while the underlying deformation mechanism remains largely unclear. Here, we investigate the crack morphology and microstructure evolution mechanism near the fracture surfaces of 4Cr5MoSiV1 hot work die steels subjected to the uniaxial tension at 580 °C. An evident deformation band was observed consisting of refined α-Fe grains with a width of about 100nm along both sides of the intergranular fracture surface. Additionally, characteristic slip band rings formed frequently, presumably due to the grain rotation near the crack. Finally, carbides (including MC, M₇C₃ and M₂₃C₆) were also considered as possible crack nucleation sources due to the existence of incoherent boundary between these carbides and the ferrite matrix.

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1. Introduction

Hot work die steel often deteriorates gradually during their applications due to deformation and fatigue during thermo-mechanical cycling, which often causes a premature failure [1]. Consequently, the failure mechanisms of hot work die steel under service conditions have attracted extensive researches [2–5]. For instance, an innovative creep-fatigue method was used to reproduce conditions at extrusion dies which gave fatigue diagrams for small-scale die samples in practical applications [3]. Gao et al. [4] used the in situ transmission electronic microscopy (TEM) tensile tests to study the face-centered cubic metals and observed the stress induced nucleation, growth and coalescence of voids. Li et al. [5] investigated the crack propagation mechanism of martensite steel via the 3D X-ray tomography and discovered both transgranular and intergranular fractures during deformation. However, most of the previous studies mainly focused on the mechanical properties of materials, while the microstructure evolution mechanisms near the crack regions remain largely unclear [6], especially for the hot die maraging steel used at high temperatures [7–9]. Here, this study focused on the microstructure evolution during the accelerated high temperature failure test (such as, tension at 580 °C with a constant rate of 1 mm/min) of maraging steel. We revealed, for the first time, the microstructure evolution mechanisms around crack regions in maraging...
steel under high temperature deformation, which critically controls the nucleation and propagation of crack at high temperature. This finding holds general implications to improving high temperature performances of engineering materials.

2. Experimental procedure

The components of the commercial 4Cr5MoSiV1 steel used in this study are (wt.%): 0.38 C, 0.35 Mn, 0.89 Si, 5.11 Cr, 1.65 Mo, 0.98 V, 0.011 P, 0.005 S and balance Fe, with N, O and H less than 50 ppm, 30 ppm and 5 ppm, respectively. Samples with a diameter of 12 mm were austenitized at 1030 °C for 30 min, followed by oil quenching to room temperature and finally tempering twice at 560 °C for 2 h followed by air cooling. Tensile samples were machined according to GB/T4338-2006 and tested at 580 °C with a SHIMADZU AG-I 250 kN operating at a constant strain rate of 1 mm/min (in accordance with that of the uniaxial tensile test). A high temperature heating device with ±3 °C error was used in the elevated temperature tests, where the samples were kept at 580 °C for 5 min before the tensile tests. After fracture, the samples were air-cooled to room temperature for further characterizations. Standard TEM samples along the tensile direction near the fracture lip were prepared by a double-jet electropolisher followed by the Ar ion milling. Microstructure characterizations were carried out in a JEM-2100 TEM operated at 200 kV. Orientation features near the fracture lip of the TEM samples were further analyzed using the Electron Backscattered Diffraction (EBSD) in a JSM-7800 F scanning electron microscope (SEM), by adopting the Transmission Kikuchi Diffraction (TKD) mode.

3. Results and discussion

Crack morphologies and the deformation structures near the fracture lip are shown in Fig. 1, in which a crack is clearly identified. Fig. 1a presents a panorama of a 35-μm-long crack, with a maximum width of ~400 nm. Selected area electron diffraction (SAED) patterns (Fig. 1b–e) indicate that the α-Fe

![Fig. 1 - Crack morphology and deformation band structure under uniaxial tension at 580 °C. a) Intergranular crack panorama; b–e) Bright-field TEM images and the corresponding SAED patterns of the deformation zones along a crack.](image-url)
matrix was retained near the crack tip. The two large oval cavities (zone A in Fig. 1a), might originate from the peeling off of carbides particles during the crack propagation under the tensile straining. At the crack propagation front, some deformation residues remained connected across the crack (zone B and zone C in Fig. 1a); in the meantime, evident variations of deformation morphologies were observed along the crack from zone B to zone E. The most severe plastic deformation was observed closer to the crack tip [10–12], where nanosized crystallites of \( \alpha \)-Fe formed preferentially, as evidenced by SAED pattern (Fig. 1b–c); the average size of \( \alpha \)-Fe at zone B, for instance, was less than 60 nm. The new crystal nuclei were retained down to room temperature due to the lack of growth dynamics; instead, these nuclei contributed to the plastic deformation. Therefore, the formation of nanograins of \( \alpha \)-Fe in the crack reflects the dynamic equilibrium between recrystallization and plastic deformation, where recrystallization played a dominant role near the crack tip, resulting in the formation of smaller grains. It is interestingly noted that tempered martensite slats were also observed away from the crack tip (e.g. zone C), possibly due to tensile load working on and causing the temperature away from the crack tip to be lower than near the crack tip. Grain characteristics in Fig. d and e away from the crack are similar to that observed by Wang et al. [2] in a sample which was heat treated at 1030 \(^\circ\)C for 30 min, oil cooled to room temperature and finally tempered twice at 560 \(^\circ\)C for 2 h. Further analysis demonstrates the presence of deformation bands with a width of \(~\)100 nm width along both sides of the crack, which mainly consist of \( \alpha \)-Fe nanocrystallites (Fig. 1d). The grain size of \( \alpha \)-Fe changed continuously.

Fig. 2 – EBSD-inverse pole figure maps of the deformation band along the crack. a) Grain-oriented IPF-X surface distribution map; b) Grain-oriented IPF-Y surface distribution map; c) Grain-oriented IPF-Z surface distribution map; d) Band contrast map; e) Residual strain distribution near the crack.
from tens of nanometers to hundreds of micrometers with the increasing distance from the crack surface (as evidenced by Fig. 2), and no sharp interface can be identified between the deformation band with nanocrystalline grains near the crack surface and the matrix with large grains away from the crack, similar to the observations of refined grains near the severe deformation zone in ferromagnetic metallic glass under the bending at 180 °C [13].

In addition, the ferrite formed continuously from the recovery and recrystallization process of tempered martensite and additional slips were stimulated in the matrix during the deformation at 580 °C. Given that the crack tip usually is the area with the most severe deformation [14], the dislocations piled-up at the nano-precipitate can escape from the locked positions at the precipitated phase and start to cross slip under the continuously increasing internal stress, which can be further promoted under the high deformation temperature. In this way, the glide planes can be changed and more slip bands were left in the region near the crack [15,16]. The formation of additional slips effectively releases the severe stress concentration at the crack tip, thereby delaying the crack propagation and contribute to the improvement of toughness [17]. Consequently, the elongation and cross section reduction of 4Cr5MoSiV1 steel under the uniaxial tension at 580 °C is increased by 202% and 20%, respectively, compared with those deformed at room temperature.

The same specimen shown in Fig. 1 was also examined by EBSD in SEM, as shown in Fig. 2. It is clear that both the intergranular and transgranular propagations of crack occurred during the crack propagation in the 4Cr5MoSiV1

Fig. 3 – TEM images demonstrating three different types of carbides in the 4Cr5MoSiV1 steels under the tensile testing at 580 °C. a–b) Bright-field image and the corresponding electron diffraction pattern of M23C6; c–d) Dark-field image and the corresponding electron diffraction pattern of M23C6; e–f) Bright-field image and the corresponding electron diffraction pattern of MC.
steel (Fig. 2a–c). Close examination further confirms the observation of prevalence of nanosized grains (~60 nm) after recrystallization near the crack surface, especially in the region where the intergranular propagation of crack occurred (Fig. 2a–d), consistent with our TEM observations. Meanwhile, a high strain zone exists near the crack surface due to the transition from to a locally higher-stress state to a high residual strain after fracture, as demonstrated by the distribution of residual strain distribution in Fig. 2e, which agrees well with the observations in the ferromagnetic metallic glass [13] and the nanograins formation mechanism under the action of mechanical stress was well revealed in the paper [18].

In addition, the morphology and structure of carbides near the crack were also examined via the SAED (Fig. 3b, d and f), which indicates the existence of nanosized $M_2C_3$, $M_23C_6$ and MC, respectively. Fig. 3a shows a spherical carbide with the size of 300 nm, which is identified to be the $M_2C_3$ according to the diffraction pattern in Fig. 3b. The elongated diffraction spots should originate from the large number of stacking faults that existed in the carbide, as suggested in the previous report [19]. Carbides of $M_23C_6$ type with fine sizes were usually formed along the ferrite/martensite boundary. The spherical MC with the size of ~150 nm is also captured (Fig. 3e), which bears a semi-coherent relationship with matrix, as indicated by the SAED patterns in Fig. 3f, which is consistent with the previous studies [4,20]. For the spherical carbides, the peeling off of them can occur easily under mechanical loading (see zone A in Fig. 1a), given the incoherency of carbides/α-Fe interfaces and the relatively large precipitate sizes such that severe dislocation pile-up at the interface occurred frequently (see Fig. 3e), resulting in high deformation incompatibility on two sides of the carbides/α-Fe interfaces and thereby the interface delamination.

Since the slip systems in each grain of the polycrystalline 4Cr5MoSiV1 steel are completely different, only those with the most favorable orientations can be activated under mechanical loading. Here we observe, for the first time, the occurrence of dislocations on different slip systems (even some concentric rings of the slip band) of 4Cr5MoSiV1 steel at the crack tip during deforming at $580^\circ$ (zone B in Fig. 1b). Fig. 4 schematically shows the formation mechanism of dislocation slip on different systems. Upon deformation, dislocations first nucleated from a preferential site (e.g., Frank-read source) inside grain 1; thereafter, consecutive nucleation and slip of dislocations on the preferential planes in grain 1 induce the formation of a slip band under the uniaxial tensile stress (Fig. 4a). The slip band intersecting with the grain boundary (GB) can result in severe stress concentration at the GB, which stimulates the dislocation emission in the adjacent grain (Fig. 4b) [21,22]. Similarly, each time the slip band intersect with the GB, additional slips can be activated in the neighboring grain under external loading. As the deformation process going on, these grains can be bent, rotated or subdivided due to the dense dislocation activities, resulting in the grain refinement (Fig. 4b–d). Eventually, a slip band ring form as a result of grain rotation and nano-crystal formation near the crack.

4. Summary

In conclusion, the failure mechanism of 4Cr5MoSiV1 hot work die steel under thermal-mechanical condition has been investigated. The formation mechanism of crack can be explained in terms of dynamic equilibria of recrystallization and plastic deformation. An evident deformation band was observed along both sides of the intergranular fracture surface, consist-
ing of refined α-Fe grains with a width of about 100 nm. The characteristic slip band rings formed frequently, presumably due to the grain rotation near the crack, while the carbides (including MC, M₇C₃ and M₂₃C₆) were also considered as possible crack nucleation sources due to the existence of incoherent boundary between these carbides and the ferrite matrix.

Conflicts of interest

The authors declare no conflicts of interest.

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