In situ TEM studies of the mechanisms of crack nucleation and propagation in fully lamellar microstructures

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The mechanisms of crack nucleation and propagation of the lamellar microstructures found in pearlite were studied by in situ transmission electron microscopy. It was found that, in addition to the predominant mechanism of cleavage fracture in the cementite plates, ductile fracture of the cementite plates is another crack nucleation mechanism. The process of failure in the fully lamellar microstructure studied in the present investigation consists of four stages: dislocation pile-ups; single crack nucleation; distribution of crack nucleation; and crack growth, followed by final fracture. Through this study, a description of the lamellar microstructure fracture mechanism has been developed for numerical simulation in future studies.

Introduction

Materials with fully lamellar microstructures, e.g. pearlite in steel, the two phase structure in γ-based titanium aluminides, several high temperature alloys produced by unidirectional eutectic growth of two phase microstructures etc. have been extensively studied. This is because of their favourable mechanical properties as well as their chemical and thermal stability. Since structure–property correlations have been established for many different properties (strength, ductility, impact energy, ductile–brittle transition temperature, and fracture toughness), it is difficult to make immediate data comparisons. However, there are certain general statements that can be made with regard to the role of major structural parameters in optimising the mechanical properties of these materials. Pearlite in steel has a typical lamellar microstructure and research on its fracture and deformation has been reported. It has been generally agreed that certain microstructural features, particularly the pearlite interlamellar spacing, control cleavage fracture of the materials. The prior austenite grain size was found to have very little effect on the fracture strength of such materials.

The cleavage cracking behaviour in solids has been explained by several mechanisms. These mechanisms have been adopted generally and applied to the cleavage fracture of pearlitic eutetoid steels. A common model is one that emphasises the formation of slip bands in the ferrite between the cementite plates. These slip bands create offsets in the cementite plates, which in turn act as local stress concentrators. With increasing loading of the cementite plates, fracture would create a path with very little resistance to further deformation. This model has worked rather well for coarse pearlitic structures. For fine pearlitic structures, the deformation is more homogeneous, as the distribution of slip bands in the ferrite is much more closely spaced.

Most studies on pearlite have been based on surface fracture morphology observations, typically using scanning electron microscopy. This restricted study to a particular region, namely, the fracture surface of the material, and was quite macroscopic in perspective. It is difficult to relate the crystallographic character of the microstructure with crack nucleation and propagation. There have not been many in situ studies on fracture in these materials, and the relationship between defects in the crystals and fracture crack nucleation needs to be clarified. The most overlooked aspect is the role of the individual phases and their interfaces. This paper describes the fracture mechanisms as investigated through in situ transmission electron microscopy studies.

Experimental procedure

In order to obtain a fully lamellar microstructure, AISI 1080 steel in the form of round bar 120 mm long, 15 mm diameter, was used in this study. The standard composition is (wt-%) 0.75–0.84C, ≤0.40Mn, ≤0.35Si, ≤0.030S, and ≤0.035P. The specimen was subjected to an austenitising annealing at 880°C for 3 h in N2 atmosphere, followed by an isothermal heat treatment at 700°C for another 4 h to obtain a fully pearlitic phase with coarse lamellar microstructure. The specimen was air cooled after completing the isothermal heat treatment.

Samples were prepared for metallographic inspection by conventional metallographic means, the etchant being 3% nital. Transmission electron microscopy specimens were made from thin foils mechanically ground from the bulk material, followed by double jet polishing with a solution of acetic acid and 3% chromic acid. The electropolishing procedure was carried out at room temperature at a voltage of 50 VDC, and with a current density of about 100 mA cm⁻².

The prepared thin foils were cut to rectangular TEM tensile specimens and then were glued on the tensile testing platform attached to the FEI 100CX TEM for in situ tensile deformation study with interval tensioning. Specimens were also prepared for in situ tensile deformation in a Philips 505 scanning electron microscope.

Results

Figure 1 shows a few dislocations in the ferrite between the cementite plates. During tensile deformation, it could be observed that dislocations initiated within the ferrite were trapped between the lamellar layers and attempted to bow in the direction of the applied stress. As the applied stress increased, these dislocations broke free and moved on their...
slip plane, until they were blocked by the ferrite/cementite interface (Fig. 2). These dislocations piled up at the interface, and they activated other dislocation sources in the adjacent lamellae, which in turn generated more dislocations. This process continued until the dislocations linked up across the entire pearlite colony with a certain orientation with respect to the colony (Fig. 3). The piling up of the dislocations at the ferrite/cementite interface created a notch on some cementite plates, marked C in Fig. 4.

In the process of tensile deformation, coarse cementite plates formed at the relatively high isothermal transformation temperatures displayed necking (indicated by B in Fig. 5) as well as kinking (indicated by A in Fig. 5). The necking could not be attributed to Rayleigh instability because the predicted wavelength of the perturbation, $2\pi/K$ where $K$ is the curvature, caused by Rayleigh instability does not correspond to the observations. It was also observed that within a pearlite colony, when a cementite plate fractured (Fig. 4), dislocations accumulated within the fractured region. However, the immediately adjacent region experienced a significant reduction in dislocation density (arrowed in Fig. 6). A similar phenomenon was observed when a notch was created in the cementite plate from the dislocations piling-up, marked C in Fig. 4.

Scanning electron microscopy examination revealed that fracture occurred almost exclusively by cleavage, with small regions of tearing along the colony boundaries (Fig. 7). The cleavage bedmarks were clearly shown on the individual facets and some could be traced back to the initiation sites (marked A).
Discussion

CRACK NUCLEATION

During most deformation processes, dislocations start moving along the most favourable slip plane when the shear stress is sufficient to generate dislocations from dislocation sources in the material. Figure 1 shows that dislocations were generated from a pinned dislocation at two ends, which bowed out and moved along under the influence of the applied stress. Figure 8 schematically illustrates the Frank–Read type mechanism, with the line AB representing the initial dislocation pinned between two obstacles. As the force applied on this dislocation increases, the curvature of BD increases to the point where it equals LAM/2, and a new dislocation is generated within the confines of a single pearlite lamella. At the same time more dislocations were generated through the same mechanism, other dislocation reactions, and/or cross slip.

As the shear stresses on these dislocations increase, they move along the slip plane (see Fig. 2) and pile up against the ferrite–cementite interface, with a specific orientation. As the density of these piled up dislocations increases, clusters of dislocations are formed. The stress exerted from the ends of these dislocation clusters generates dislocation sources in the cementite. Therefore, an increasing of number of dislocations accumulate, and finally the cluster of dislocations cross over the entire pearlite colony with a certain angle to the ferrite/cementite interfaces, as shown in Fig. 3.

Subsequently, when the piled up dislocations meet the colony boundary, the density of dislocations increases rapidly as the stress concentration formed by the clusters of dislocations cannot be relieved through activating the dislocation sources in the adjacent colonies. This is related to the different crystallographic orientation of slip planes in these colonies. The component of stress acting on the dislocation sources, which are located in the adjacent colony, is not sufficient to drive dislocation movement on the slip planes. Hence, when the density of dislocations reaches a certain threshold, under the action of both shear and applied stresses, cracks, observed in the form of a notch, are initiated in the cementite plates (Fig. 4). It is clear that the colony boundary and the interface of ferrite and cementite are different types of dislocation barriers in fully lamellar structural materials. The colony boundary can be considered to further increase the strength of the materials.

CRACK PROPAGATION

Once the first cementite plate fractures, the stress experienced by the other cementite plates increases substantially, leading to rapid fracture. Therefore, this process results in a certain statistical distribution of nucleating fracture sites in the pearlite colonies. This crack nucleating mechanism implies that the colony size (colony boundary) of pearlite is also an important factor affecting the fracture of pearlite. This result is different from some earlier studies.
Within the same pearlite colony, fracture occurs preferentially in some cementite plates, perhaps because they are more 'constrained' than other non-fractured cementite plates. The state of constraint of the fractured cementite plates is determined by their morphologies, as depicted in Fig. 9. Plates labelled A, B, and C are considered less constrained than plate D. This is because the loading force at one end of these cementite plates can be relieved to form a 'free' end through deformation of the surrounding ferrite. Therefore, plate D fractures first under the action of applied stress. As discussed in the previous paragraph, the fracture of plate D leads to a substantial increase in load for the other cementite plates, but to transfer the stress to these plates the ferrite surrounding the 'free' ends of the plates has to deform plastically first. Consequently, two scenarios may occur: (1) the ferrite deforms plasticly and thus reduces the existing stress level, in which case the cementite plate does not experience fracture; (2) there may be some plastic deformation in the ferrite, but the existence of biaxiality may constrain the ferrite under a state of hydrostatic stress. A sufficiently high stress is transferred to the less constrained cementite plates and results in fracture. The latter mechanism has more validity as numerous cementite plates within the same pearlite colony were observed to have fractured.

These less constrained cementite plates may undergo another type of plastic deformation. The 'free' ends of the plates may 'kink'. This begins as a result of the accumulation of dislocations around the cementite plate, as indicated at region B on Fig. 4, where dislocations line up at an angle to the lamellae. This clustering of dislocations around the free end of the cementite plate causes localised off lamellar axis force, which bends or kinks the cementite plate. Note that this situation is only prevalent near the free end of the cementite plate. The same phenomenon was observed in region A on Fig. 5, although the cementite plates were close to each other, which prevented the formation of dislocation networks. However, a localised cluster of dislocations was observed at the kinked segment of the cementite plate, marked A in Fig. 6.

Deformation of cementite plates can occur in a form that is different from the above described mechanism. From the fractured morphology of the cementite plates, necking can be seen in Fig. 5 region B, suggesting that the cementite plate has experienced plastic deformation. From the orientation of the plate with respect to the fracture faces, this particular cementite plate has been pulled to fracture. Thus, a second mode of fracture in pearlite may be tensile plastic deformation of cementite that can lead to fracture within the cementite lamella. Necking of cementite plates has previously been reported, but the thickness of the plates has to be less than 0.1 μm. However, Fig. 7 shows that the cementite plates already have thickness greater than 0.1 μm, indicating that cementite contains crystallographic defects. These defects became mobile under the influence of external stresses and therefore lead to a certain degree of plasticity, regardless of the thickness of the cementite plates.

Although there is an obvious reduction in dislocation density surrounding the crack (region C in Fig. 4), dislocations were seen to exist within the ends of fractured cementite plates (region A in Fig. 6). These dislocations are found to be oriented towards a common direction, suggesting absorption of dislocations by the crack. This phenomenon also proved the importance of dislocations piling up for cementite crack nucleation.

Based upon the above discussion and the fracture morphologies of cementite in Figs. 4 and 7, the evolution of the microcracks from the fracture of cementite plates can originate in two forms: cementite plate cleavage or ductile fracture. The cleavage of cementite plates is due to the effect of externally applied stress, which occurs when the pearlite lamellar orientation is unfavourable for the generation and multiplication of dislocations. From the morphology of the necking of the cementite plates, it is deduced that the fracture is caused by the tensile stress alone, therefore, this type of fracture is much more difficult than the cleavage fracture of the cementite plates due to the dislocation piling up.

For the types of coarse pearlite crack nucleation, in addition to the cleavage and ductile fracture of cementite plates, pearlite also undergoes the process of ferrite cleavage. Figure 10 (SEM) shows the morphology of this fracture mechanism. The cleavage of ferrite occurred along a certain crystallographic orientation, 'zigzag' passing from one lamella to another. The experimental results and atomic calculations reveal that the ferrite cleaves along the {001} plane. It is believed that the dislocations would create a crack nucleus according to Cottrell's model when the lamella in pearlite and the direction of tension is favourable to the movement of dislocations along the {110} plane of ferrite. However, this mechanism is due to within a lamellar layer, it would not cross into other lamellae. As for the cause of crack propagation, this is due to the localisation of stress after the formation of a microcrack, which activates the movement of dislocations in nearby ferrite. The exact mechanism causing the crack to propagate at about 60° along the ferrite/cementite interface is still under investigation. As the surface energy of ferrite is considerably higher than that for cementite, which implies that it requires more energy for the crack to propagate in ferrite, therefore the occurrence of this particular kind of fracture is not as common as cleavage in cementite plates.
CRYSTALLOGRAPHIC ASPECTS

According to the Bagarytski and Pitsch/Petch relationships for the crystallography of pearlite, ferrite and cementite have the following crystallographic orientations respectively:\(^{26,27}\)

\[
\begin{align*}
(001)_{\text{Fe}_3\text{C}}/\langle 211 \rangle & \quad (001)_{\text{Fe}_3\text{C}}/\langle 521 \rangle \\
(010)_{\text{Fe}_3\text{C}}/\langle 011 \rangle & \quad [100]_{\text{Fe}_3\text{C}}/\langle 111 \rangle \\
(010)_{\text{Fe}_3\text{C}}/\langle 111 \rangle & \quad [010]_{\text{Fe}_3\text{C}}/\langle 111 \rangle \\
\end{align*}
\]

It has been reported that the cementite plates grow along the [111] direction of ferrite, and this direction is the b-axis of cementite.\(^{26}\) According to the proposed three dimensional model of pearlite formation, as (112) is the slip plane of ferrite, while (001) is the close packed plane of cementite, the atoms on the (112) plane of ferrite need to shift only a small amount to obtain the three dimensional structure of the (001) plane of cementite. Therefore, the (112) plane of ferrite and the (001) plane of cementite form the ferrite/cementite interface.\(^{26}\) From the slip planes of ferrite, such as \{110\}, \{112\}, and \{123\}, it can be computed that the slip planes \{211\} plane of ferrite need to shift only a small amount to obtain the three dimensional structure of the (001) plane of cementite. Therefore, the (112) plane of ferrite and the (001) plane of cementite form the ferrite/cementite interface.\(^{26}\) From the slip planes of ferrite, such as \{110\}, \{112\}, and \{123\}, it can be computed that the slip plane (211) in the first type of crystallographic orientation, as shown above, forms an angle of 48°-19° with the ferrite/cementite interface, while the second orientation consists of the plane (211) and the interface (521), forming an angle 47°87° (2°6° deviation). As (211) is the slip plane of ferrite, it is much easier to initiate and activate the dislocation sources and move the dislocations, compared to the same action in the non-slip planes (441) or (522) that have a ferrite/cementite interface angle of 44°.\(^{71}\) It is considered that in pearlite, the angle between the slip plane and the ferrite/cementite interface is more likely to be 48°19° or 47°87° (2°6° deviation). This explanation is consistent with the observation in Fig. 3. Although the angle between the dislocation slip plane and the ferrite/cementite interface shown in this figure is ~40°. It is believed that it deviated from 45° because the projection plane of the TEM image was not perpendicular to the axis of the slip plane and the cementite plane. This result is also similar to that of an earlier study, which showed that the shear bands have a 45° angle to the tensile axis, although the mechanism was not explained in detail.\(^{25}\)

Conclusions

*In situ* TEM tensile deformation of fully pearlitic structures has shown that two types of crack nucleation take place: cleavage of the cementite plates within the lamella; and ductile fracture of the cementite plates. Of these two mechanisms, cleavage of cementite plates is more commonly encountered, in which the effective stresses imposed by the dislocations piled up at the interface between the cementite plates and the ferrite of the pearlite lamellar nucleate cracks on the cementite plates. These dislocation pile ups or networks are also seen to link up with dislocations from adjacent lamella and cross over the whole pearlite colony, which in turn imposes an effective shear stress on the cementite plates at an orientation of about 45° (2°6° deviation). This implies that grain size may be one of the important factors controlling the strength of the fully pearlitic microstructure. Once the first cementite plate has fractured, the nucleations of cracks are then distributed on the cementite plates in the pearlite colony. These cracks rapidly propagate and link up, resulting in final failure. The processes of material failure in the fully pearlitic microstructure consist of four stages: dislocations pile up on the ferrite/cementite interface; single crack nucleation forms as one of the cementite plates leaves under the effective shear stress from the dislocation pile up; crack nucleations statistically distribute in the pearlite colonies; cracks grow and link up to fracture the material. Although most of the cementite plates failed by cleavage, some were observed to have failed in a more ductile manner, especially those oriented along the direction of the applied stress and constrained at both ends.

References