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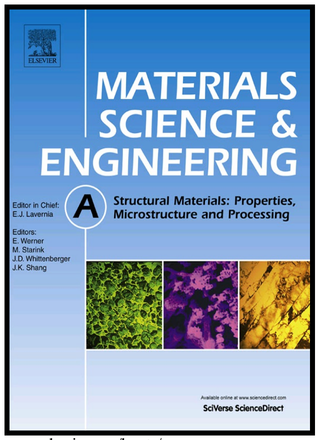

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Di Wan, Afrooz Barnoush

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Plasticity in cryogenic brittle fracture of ferritic steels: dislocation versus twinning

Di Wan, Afrooz Barnoush

Department of Mechanical and Industrial Engineering, Norwegian University of Science and Technology, Richard Birkelands vei 2B, 7491 Trondheim, Norway.

Abstract

The sub-surface deformation structure after cryogenic (77 K) brittle fracture in a ferritic steel was characterized by scanning electron microscopy (SEM). Twin-like structures were found in many grains below the fracture surface. Electron backscatter diffraction (EBSD) was used to identify the crystallography of the structures, and the twin relation in body-centered cubic (BCC) systems was indexed. The twins in the sub-surface area were characterized further by electron channeling contrast imaging (ECCI). This study clarifies the low temperature brittle fracture behavior in the ferritic Fe-3wt%Si steel consists of both cleavage and plastic deformation in the form of dislocation activities and twinning.

Keywords: cleavage fracture, twinning, scanning electron microscopy, EBSD, ECCI, BCC.

1. Introduction

It is universally acknowledged that metals can change their mechanical performances when the temperature reaches a certain low level. A famous disaster is the sinking of the RMS Titanic, in which the brittle fracture of steel at extreme cold temperature after a collision with an iceberg was a major reason [1]. As the most dangerous form of fracture, the brittle cleavage fracture is a transgranular fracture by the separation of well-defined crystallographic planes, which in ferritic steels are the {100} planes [2]. Based on the classical theory, this kind of brittle fracture is typically associated with no plastic deformation, and the loading energy is normally released by the separation of certain atomic bonds defined by crystallography. This concept was integrated into the famous Griffith model in explaining fracture toughness. From literature, major characterizations are focusing on the fractography, and the microstructural changes in the vicinity of the crack are simply ignored. This is mainly due to the challenging requirement for sample preparation. Recent development by means of focused ion beam (FIB) and lift-out method plus transmission electron microscopy (TEM) observation [3-5] is promising, but still the observation is limited to a very small area, and on the other hand this technique is very time-consuming. In this study, we shift the key point from the fracture surface to the sub-surface area. Specifically by means of scanning electron microscopy (SEM) based techniques including electron backscatter diffraction (EBSD) and electron channeling contrast imaging (ECCI), we are extending our observation to a much larger area along and below the crack, while still capable of observing single dislocation as in the case of TEM. This new method has a potential to make a paradigm shift in the study of the fracture process and as an example, we show its application for understanding the mechanism of low temperature brittle fracture.

Dislocation slipping and mechanical twinning are two common plastic deformation mechanisms. Despite the difficulties, twinning deformation in body-centered cubic (BCC) metals is reported and summarized in some review articles [6-8]. The occurrence of twinning was linked to brittle fracture in some BCC metals [9]. Cheng et al. [10] observed ferrite twins in a Fe-Mn-Al alloy by quenching the material from high temperature, and categorized them as mechanical twins induced by the thermal strain energy relief. This mechanism could contribute to a large portion of the energy release since the intrinsic stacking fault energy (SFE) in BCC materials is relatively high [11, 12].

The present study shows that the deformation structure under a cryogenic (77 K) fracture surface is associated with some plasticity in the form of dislocation activities and the formation of twin-like structures. BCC steels are one of the most widely used structural materials served in various industries subjected to low temperature, such as the oil and gas industries or marine industries in the arctic regions. To understand the fundamental deformation and fracture mechanisms under low temperature is significantly supportive to know the behaviors of the engineering structures in the extreme conditions.

2. Experimental

In this study, compact tension (CT) specimens according to the ASTM E647-13 standard were cut by electrical discharge machining (EDM) from a large-grained (grain size approx. 300 µm) Fe-3wt%Si ferritic steel. The chemical composition is the same as in [13]. Pre-cracks were initiated by fatigue loading. Pliers were used to hold the CT specimen in the holes with fixed relative position, and the specimen was put into liquid nitrogen (77 K) with the pliers jaws. Manual loading was applied through the pliers handles outside of the liquid nitrogen in an opening manner and the specimen fractured in the liquid nitrogen, as schematically described in Fig. 1. With this method, the loading was exerted when the specimen was inside the liquid nitrogen, which means the fracture happened in cryogenic environment. The reproducibility of the presented result was confirmed after loading more than 10 specimens using the same method, and the fracture morphology was confirmed as completely cleavage type in all cases. The fractography was done by a Quanta 650 SEM (Thermo Fisher Scientific Inc., USA). After fractography, the low temperature fractured part of the specimen was cut and the fracture surface was electrodeposited with Fe. The cross-section of the sample was then prepared by grinding to #4000 emery paper plus final electropolishing in H_2SO_4-methanol electrolyte. Thanks to the coating, the deformation zone near the fracture surface could be well preserved. The cross-section of the fractured sample was further investigated by EBSD and ECCI in the same SEM. With the help of ECCI, plasticity such as dislocations and twins can be investigated in bulk samples in SEM. Detailed technical information of this imaging technique can be found in [14].

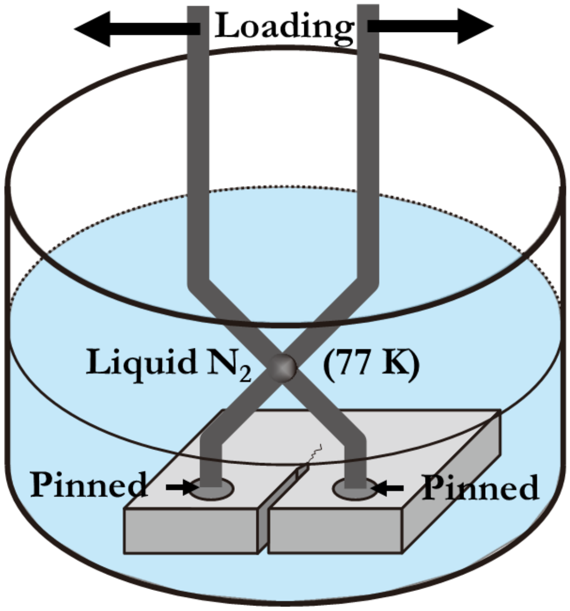

Fig. 1 Schematic description of the loading condition: CT specimens with pre-crack fractured inside liquid nitrogen by manual loading.

3. Results and discussion

Fig. 2 shows the fracture surface after low temperature loading. The whole surface has a pure transgranular brittle cleavage-like pattern. Clear river patterns can be observed over the surface. Sharp facets having an angle of roughly 90° can be seen at the end of some grains. This is common since the cleavage plane in BCC structure is {100} planes. This figure confirms the brittle cleavage behavior of the test piece under such loading conditions.

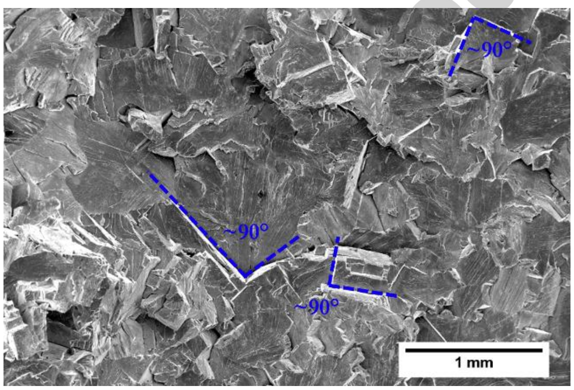

Fig. 2 Fractography showing pure cleavage type fracture of the investigated sample. The blue dash lines indicate that an approximate angle of 90° can be found between the facets.

Fig. 3 is the sub-surface microstructure characterized by ECCI. An overview is shown in Fig. 3a. A plastic zone can extend into the matrix by about 100 µm. Some twin-like structures with a width up to one micrometer can be found, and these structures can influence the direction of some secondary cleavage cracks, deflecting the cracks by small steps, as shown by the yellow arrows. A magnified view of the twin-like structure is shown in Fig. 3b. Dislocation slipping lines can go through this band structure with the outgoing direction unchanged. Individual dislocation lines can be observed at the end of the twin-like structure. Secondary cracks are seen inside the grain matrix as indicated by the yellow arrows in Fig. 3a. Some of the secondary cracks are stopped by the twin-like structures, as indicated by the white arrow. Fig. 3c shows a magnified view of the end of one secondary crack. Individual dislocations are found near the crack. Fig. 3d shows an interaction area between a twin-like structure and a grain boundary (GB). Plasticity is observed emitting from the intersection zone. Heavy plastic deformation (large dislocation density) can be found near the intersection area, and it becomes less and less significant when going into the grain, as indicated by the dash-lines with arrows heading dislocation emitting directions. To clarify the nature of this twin-like structure, EBSD was used to check the crystallographic information.

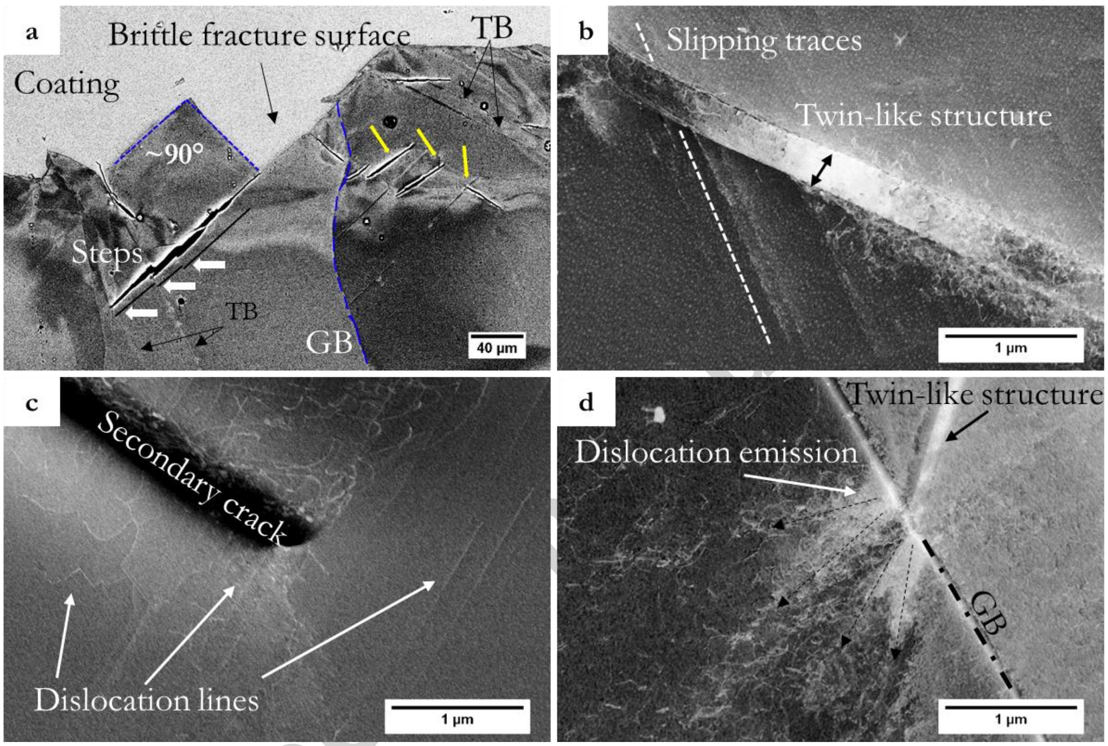

Fig. 3 Deformation structure below the fracture surface. (a. overview showing the sub-surface microstructure; b. twin-like structure with dislocations; c. dislocations near a secondary crack; d. intersection between a twin-like structure and a GB). The white arrows in a. indicate the steps introduced by the twin-like structures. The yellow arrows in a. indicate secondary cracks formed inside the matrix. Electronic version in color.

Fig. 4 shows the normal direction (ND) – inverse pole figure (IPF) map of an area below the cleavage fracture surface by EBSD scan. High angle grain boundaries (HAGBs) and twin boundaries (TBs) are indexed. The TBs in this map are defined as 60° with respect to <111> axis and the twin plane is defined as {112} [6]. The {112} pole figure (PF) is plotted for the red (grain matrix) and blue (twin-like structure) parts of the big grain in the lower part of the IPF map, and the poles were plotted as red and blue, respectively. It can be seen that three poles are overlapping each other as the highlighted circles in the PF. This means the red and blue parts share same {112} planes, which is the twinning plane of BCC crystals. The misorientation profile over a distance of about 5 µm across the blue part is plotted, as shown in the lower right corner of Fig. 4. A misorientation of about 60° can be seen between the red and blue part, which suggests a typical twinning relation. {100} traces are marked near the fracture surface, all of which show parallel relation to the fracture plane, suggesting cleavage separation of the {100} planes. Some regions of interest (ROIs) are highlighted in this figure and further characterized in details.

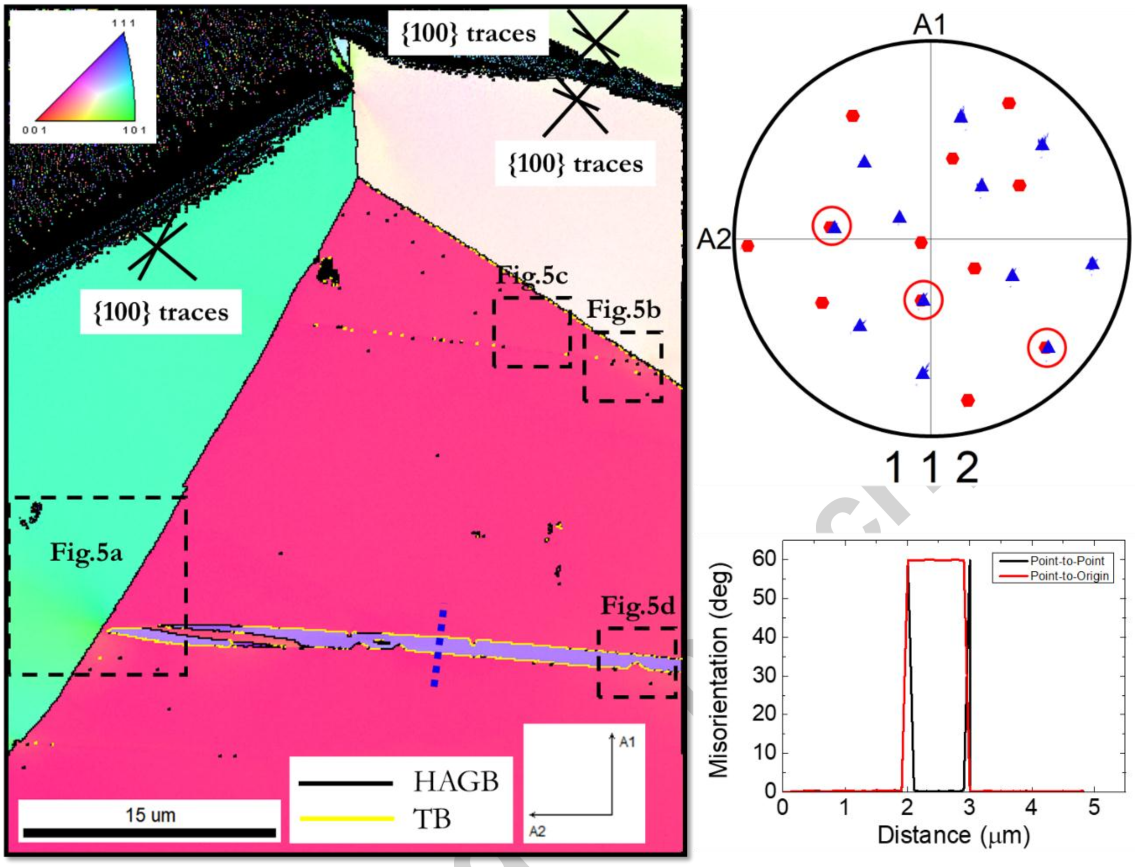

Fig. 4 ND-IPF map and the corresponding {112} PF of the red grain and the blue part inside the red grain of the area below the fracture surface. {100} plane traces are marked near the fracture surface. TB can be indexed between the blue and the red parts. Three overlapping poles are highlighted in the pole figure. Misorientation profile is plotted over the highlighted blue dot-line in the IPF map.

Fig. 5 shows a close-up investigation of the twin-like structures highlighted in Fig. 4. A large area of plastic deformation in the neighboring grain can be observed in Fig. 5a. The plasticity was emitting from the intersection between the twin-like structure and the HAGB. In Fig. 5b, a plastic zone was found also inside the grain containing the twin-like structure. Clear individual dislocations can be observed near the plastic zone. These individual dislocations can generally be found in the grain, as in Fig. 5c. From the EBSD information, most of these dislocation lines are parallel to the <111> directions of this grain, suggesting a large fraction of screw dislocation components. In Fig. 5d, an imperfection in the twin-like structure is presented. A triangular shaped zone is observed connecting the boundary, and arrays of dislocations are emitted from the intersecting points on the straight boundary. The arrays are also parallel to the <111> directions. From this figure, the largest width of this twin-like structure is measured to be about 1 μm.

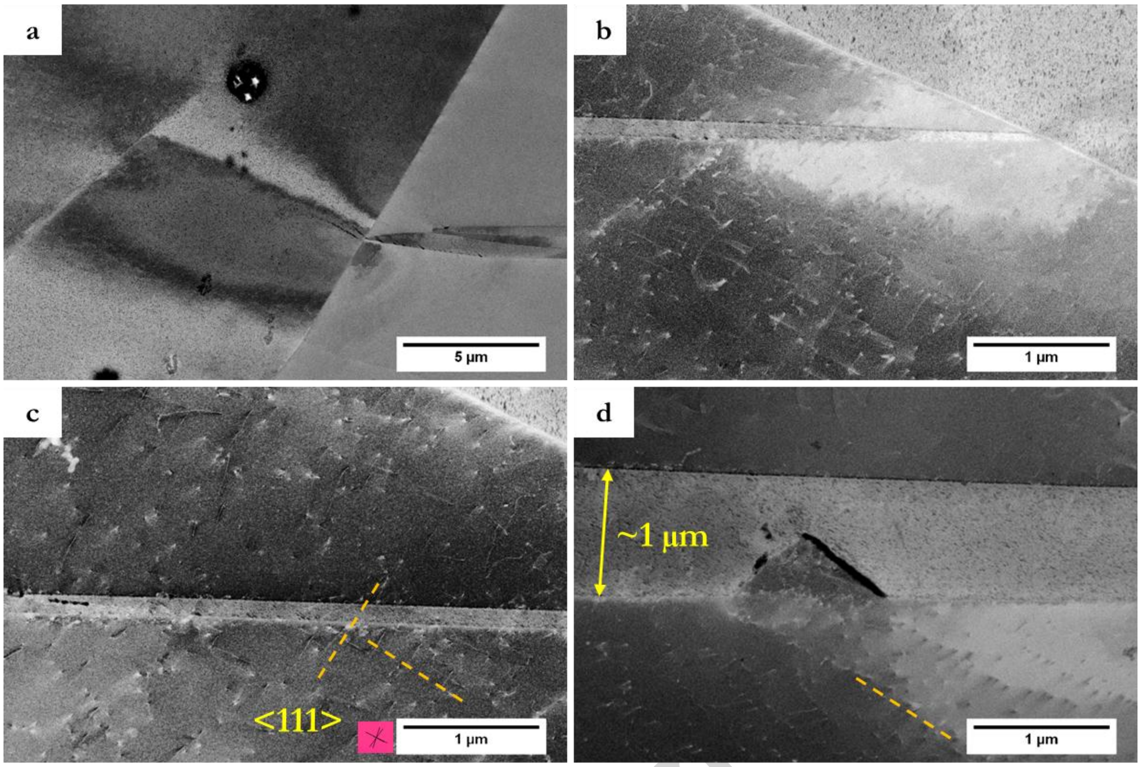

Fig. 5 Detailed investigation of the twin-like structure. (a. induced plasticity in the neighboring grain; b. plasticity near the intersection zone between the twin-like structure and a grain boundary; c. dislocation structure near the twin-like structure; d. imperfection in the twin-like structure. The lines in the red rectangular in c. and the yellow dash lines indicate the <111> directions indexed by EBSD. All corresponding ROIs are marked in Fig. 4.)

As is pointed out by Chen [2], a plasticity should be expected locally to trigger the cleavage fracture of ferritic steels at low temperature. The energy release, as well as the stress redistribution, is strongly associated with the distribution of this plastic zone. The local plastic flow is a necessary precursor to cleavage and yielding, which might be induced by dislocation slipping or twinning [15]. In the present investigation, local plasticity was observed generally spread near the fracture surface. Both dislocation slip and twinning are found contributing to the local plasticity (c.f. Fig. 3). According to the results in both Fig. 3 and Fig. 5, the twins could also influence the plasticity in the surrounding area.

The low temperature deformation behavior of pure iron has been studied using small-scale mechanical testing (micro-pillar bending) and molecular dynamics (MD) simulations [16, 17]. It is suggested that the dislocation slipping behavior at low temperature is mainly controlled by the screw components due to the intrinsic core structure. As shown in Fig. 5c, the dislocation lines are generally parallel to the <111> directions of the grain, and since the perfect dislocations in BCC lattice have the burgers vector of ½ <111>, it could be concluded that the dislocations here are containing a large fraction of screw components.

As is generally investigated and modelled, the screw dislocation motion in BCC materials is mainly realized by the double kink mechanism [18-21]. Since this mechanism is a thermally activated process, the screw dislocation motion depends strongly on the temperature. When the temperature decreases, the thermal activation energy of screw dislocation motion is also decreasing. According to the dislocation dynamics simulation results [21], at low temperature the dislocations prefer to form long straight lines. When looking at the investigated dislocation lines inside the grain such as Fig. 3c and Fig. 5b-d, long straight dislocation lines parallel to the <111> crystallographic directions are found, which give proof to the simulation results in [21]. We could thus conclude that although pure cleavage brittle fracture happened to the investigated specimen under cryogenic temperature, some plastic activities were still happening in the grains near the fracture surface in the form of screw dislocation motion. However, if we start talking about the twinning behavior in the investigated material, it is considered as a less popular topic that was paid less attention.

Conventionally, twinning in ferrite is relatively difficult since the intrinsic stacking fault energy (SFE) is extremely high that can reach up to ~1 J/m^2 [11, 22]. The MD simulations indicate that twinning can happen in pure Fe micro-pillar when the temperature is lowered to 15 K [16, 17]. However, the twinning phenomenon has already been observed at 77 K in the Fe-3wt% bulk specimen in the present study. Several reasons could be attributed to this discrepancy. Das [23] reported Si can decrease the stacking fault energy of an alloy, which is an important parameter of the twinning possibility of a material. During unexpected brittle fracture, the stress concentration at the crack-tip will be much higher than the static loading condition. And deformation twins in BCC materials can be induced by this heavy deformation [6]. Based on the facts stated above, the twinning phenomenon can happen more easily in the studied material than the prediction to pure iron [16, 17]. Although previous works found proof of twins in ferritic materials [24-26], they focus only on the fracture surface and no detailed crystallographic information was given. Instead, we used advanced high resolution SEM techniques to characterize the twin structure and successfully correlated the structure with crystallography. The <111>-60° twin boundary with a common {112} plane was confirmed and thus giving explicit proof to the twinning behavior in the studied BCC material.

Reid [9] summarized the relation between twinning and brittle fracture. The relation is different from case to case, but most cases fall in these four categories: 1. twinning and brittle fracture are independent processes; 2. twins are nucleated by cracking; 3. cracks are nucleated by twins, but the cracks may or may not continue propagation; 4. twins provide preferred path for crack propagation. According to Fig. 3a, most of the secondary cracks are located away from the twins, and some are stopped by the twins. No strong proof can correlate the primary crack (corresponding to the fracture surface) and the twin structures. It is thus inferred that the twins would not induce the crack nucleation in the studied case. From the same figure, the cracks are not along the same direction as the twins, although they are located in the same grain. Thus the twins in the studied case could not provide a preferred path for the crack propagation. From literature, some proof of twinning in ferrite could be found on fractographs after cyclic loading at 123 K [25] or monotonic tensile loading at 77 K [24]. Except that the twins were found together with cleavage fracture, there is no unique association between twinning and fracture in BCC metals and alloys [9].

A possible mechanism of the twin nucleation was described in detail in Ref. [22]. One ½ <111> screw dislocation is considered to dissociate into three 1/6 <111> fractional dislocations with a configuration symmetric to the <111> screw axis. This configuration is stable under zero stress state. However, the fractional dislocations will translate on the most stressed {112} plane under external loading, and form a three-layer stacking fault. This is considered as the nucleus of the twin, and further dislocation motion on the {112} plane would be considered as twin growth. The screw dislocation motion requires a thermal activation due to the dissociated core structure, thus the dislocation mobility is strongly reduced at low temperature. Therefore, the twinning mechanism mentioned above could contribute partly to the plastic energy release from the external loading. This mechanism is in good agreement with some proposed mechanisms and the corresponding experimental results [27-29].

4. Summary

To sum up, we did combined high resolution ECCI and EBSD characterizations on a ferritic specimen that was fractured at cryogenic temperature (77 K). Some of the conclusions could be drawn:

- The cryogenic cleavage fracture of the studied ferritic Fe-3wt%Si steel was associated with plastic deformation near the fracture surface in the form of dislocation emission and twinning.
- For the first time, a <111> 60° type twin boundary with common {112} plane relation in the studied material was characterized by the combined ECCI + EBSD technique. The characterizations provide both detailed crystallographic information and microstructure feature with resolution up to single dislocation line level. This technique is promising in the analysis of the fracture process zone in a much larger area than conventional TEM investigations.
- The twinning mechanism in BCC structure was discussed, which was considered to be related with the dissociation of <111> screw dislocations and the dislocation motion on stressed {112} planes.

A promising outlook of this work is to construct a proper experimental setup that is able to control the load and testing environment (e.g. temperature control) as well as record the mechanical data for further analysis. This is ongoing in the authors' research group and will be the prospect of the future work.

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Author contributions

D. Wan conducted all experimental work, data analysis, interpretation and prepared the manuscript. A. Barnoush is the supervisor of this work. Both authors discussed the manuscript and approved for submission.

Competing interests statement

The authors do not have competing interests.

Data availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time due to technical or time limitations.

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