

Influence of interstitial solute atom on fatigue crack growth resistance in austenitic and ferritic steels

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Influence of interstitial solute atom on fatigue crack growth resistance in austenitic and ferritic steels

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Abstract

Fatigue damage is considered to be the major cause of accidental failures in structural applications, hence many researchers in the world working on material strengthening and enhance the fatigue strength. Solid solution strengthening has attracted a lot of attentions, and thus many new high strength steels with interstitial-substitutional (i-s) interactions have been developed such as TWIP and High nitrogen (HN) steels. The major mechanisms for high work hardening capacity and ductility is due to dynamic strain aging (DSA) effect from Mn-C couples in TWIP steels, and enhanced dislocation planarity by Cr-N couples in HN steels. In addition, interstitial carbon in ferritic steels is reported to enhance the transgranular fatigue crack resistance and fatigue limit due to dynamic strain aging in ferritic steels.

However, the effect of dynamic strain aging from Mn-C, and enhanced dislocation planarity from Cr-N (i-s interaction) on fatigue crack resistance and fatigue limit has not been studied in detail. In addition, solute nitrogen would be another candidate for dynamic strain aging effect in ferritic steels that can contribute to enhance the fatigue strength which has not been studied. Therefore, this thesis is mainly focused on firstly studying the effect on Mn-C and Cr-N (i-s) interaction on the fatigue crack growth resistance in austenitic steels. Secondly studying the effect of interstitial nitrogen on the fatigue crack resistance in ferritic steels.

To this end, fundamental researches were carried out firstly, on three types of austenitic steels i.e. Fe-23Mn-0.5C (Mn-C), Fe-25Cr-1N (Cr-N) i-s interactions, and Fe-18Cr-14Ni steel without any i-s interactions with rotating bending fatigue tests. Secondly tension-compression fatigue tests were conducted on the Fe-0.0113N steel to study the effect of interstitial nitrogen. Different techniques were used in this study such as replica technique with optical microscope was selected to catch the fatigue crack propagation,

scanning electron microscope to study the fracture surface, electron back scatter diffraction analysis to study the microstructure analysis, and electron channeling contrast imaging technique used to observe the dislocation structures.

This work outlines our findings in understanding the influence of i-s interaction of Mn-C and Cr-N on the fatigue crack growth behavior and resistance in austenitic steels, and influence of interstitial nitrogen on the fatigue crack behavior in ferritic steels. The results show that as following:

- i. Fatigue limits of the Fe-25Cr-1N Fe-23Mn-0.5C and Fe-18Cr-14Ni steels were 310 MPa, 260 MPa and 110 MPa, respectively. The Mn-C and Cr-N (i-s) interaction showed a positive effect in enhancing the fatigue limit of the Fe-23Mn-0.5C and Fe-25Cr-1N steels in comparison with the Fe-18Cr-14Ni steel without any i-s interaction.
- ii. Electron channeling contrast imaging is used to observe the dislocation structure near the tips of non-propagating fatigue cracks for the three steels. The enhancement of small crack resistance in the Fe-23Mn-0.5C steel is attributed to local hardening at the crack tip owing to dislocation density increment by dynamic strain aging. In case of the Fe-25Cr-1N steel, the enhanced dislocation planarity stemming from Cr-N interaction acts as significant factor increasing crack resistance. The enhanced dislocation planarity resulted in the dislocation pile-up stress at the crack tip, preventing dislocation emission from the crack tip.
- iii. The Fe-0.0113 mass% N steel showed a fatigue limit at 150 MPa and a non-propagating fatigue crack at the fatigue limit. Intergranular and transgranular crack initiation were observed, and cracks propagated along the grain boundaries and grain interiors at stresses above the fatigue limit. The Fe-N steel did not show a high coaxing effect because of repeated crack initiation and coalescence with the main crack.

The results of our studies indicate that the effect of dynamic strain aging from Mn-C couples and enhanced dislocation planarity from Cr-N couples in austenitic steels, have a positive effect on the fatigue crack propagation resistance in comparison with the normal steel without any I-S interaction. In addition, the effect of solute nitrogen in the Fe-N ferritic steels showed the fatigue crack resistance is less in comparison with the Fe-C ferritic steels.

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Nomenclature

σ_w	Fatigue limit
ΔK_{th}	Experimental threshold stress intensity factor range
ΔK_{th_pre}	The predicted fatigue threshold by using Murakami's equation
$\sqrt{area}$	The square root of the area obtained by projecting the flaw onto a plane perpendicular to the loading axis direction

Chapter 1

General Introduction

1.1 Research background of this study

Fatigue is rather like a disease of metal. Fatigue failure is one of the largest cause of failure, with approximately 90 % of all mechanical structure failures are due to fatigue damage [1]. Fig. 1 shows examples of fatigue failures in actual applications, Fig. 1(a) shows a failure of crankshaft due to fatigue [2], and Fig. 1(b) shows a fatigue crack initiation on the bridge girder [3]. When a structure is subject to a periodical varying load, some cracks are formed at the site of some material imperfections such as voids, inclusions and crack-like defects, which usually are of microscopically small size. As fatigue cycles accumulate, these short cracks may continue to grow and coalesce, and gradually evolve to be a dominant crack, eventually reaching a critical size, beyond which catastrophic failure will happen. However, the initial further propagation of certain defects may be arrested at grain boundaries or within grains, which often occurs at low fatigue loads below a threshold.

The study of fatigue can be traced back to the last century with fatigue damage mainly divided into two parts: fatigue crack initiation and propagation. Fatigue crack propagation is further divided into small and long crack regions. Commonly, the term “small crack” in fatigue phenomena is understood from different viewpoints, such as microstructurally small cracks [4], mechanically small cracks [5], physically small cracks [6], and chemically small cracks [7]. Mechanically or microstructurally small cracks are known to be most important in terms of fatigue damage, because 70% of fatigue life is covered by small fatigue crack propagation [8]. The microstructure of the material has a substantial influence on small fatigue crack growth behavior. It is microstructural features such as texture and the distribution of effective barriers (grain boundaries, interfaces of different phases) to crack growth which brings about the anomalous or intermittent growth pattern of small cracks, the control of these barriers in terms of their size, strength and distribution has a profound effect on the fatigue resistance of materials.

In terms of actual mechanical structures, there are variety of factors such as geometry, material, manufacturing method, and environment that influence the final fatigue fracture. But major concern is mainly depending on the material properties for the fatigue

damage in practical applications. Hence for high reliability, convenience, environmental-friendliness in practical engineering, and with the advent of modern industries, research on material strengthening in improving fatigue lives has attracted a lot of attention. There are different methods of material strengthening mechanism's used for improving the fatigue lives in materials such as

1. Work Hardening
2. Precipitation hardening
3. Grain boundary strengthening
4. Solid solution strengthening and alloying

1.1.1 Work hardening

In terms of work hardening, the primary species responsible for work hardening are dislocations. Dislocations interact with each other by generating stress fields in the material. The interaction between the stress fields of dislocations can impede dislocation motion by repulsive or attractive interactions. Additionally, if two dislocations cross, dislocation line entanglement occurs, causing the formation of a jog which opposes dislocation motion. Work hardening has been reported to improve the crack closure level of fatigue crack in steels [9].

1.1.2 Precipitation hardening

Precipitation hardening method is a heat treatment technique to improve the strength. Precipitation hardening stainless steels are chromium and nickel containing steels that provide an optimum combination of the properties of martensitic and austenitic grades. Like martensitic grades, they are known for their ability to gain high strength through heat treatment and they also have the corrosion resistance of austenitic stainless steels. The precipitation hardened steels have been reported [10] to have higher fatigue strength than the normal steels because of the movement of dislocations being hindered by the precipitates.

1.1.3 Grain boundary strengthening

The strengthening in polycrystals due to grain boundaries has been experimentally established ever since Hall (Hall 1951; Petch 1953) proposed his relation between the grain size and the yield stress. The resistance of metals and alloys to fatigue crack initiation and

propagation is known to be influenced significantly by grain size. Based on a wealth of experimental results obtained on the effect of grain size on fatigue strength [11, 12], it is widely recognized that an increase in grain size generally results in a reduction in the fatigue endurance limit. On the other hand, a coarser grain structure can lead to an increased fatigue threshold stress intensity factor range, as well as a decrease in the rate of fatigue crack propagation.

1.1.4 Solid solution strengthening

In solid solution strengthening, solute atoms of one element are added to another, resulting in either substitutional or interstitial point defects in the crystal (Figure 1.2) [13]. The solute atoms cause lattice distortions that impede dislocation motion, increasing the yield stress of the material. Solute atoms have stress fields around them which can interact with those of dislocations. The presence of solute atoms imparts compressive or tensile stresses to the lattice, depending on solute size, which interfere with nearby dislocations, causing the solute atoms to act as potential barriers. There are two types of solid solutions, (1) Substitutional Solid Solution: Solute and solvent atoms are roughly of the same size and the solute atoms will replace the solvent atoms in its position in the crystal structure, and (2) Interstitial Solids Solution: The solute atoms are smaller than the solvent atoms and they will occupy interstitial positions in the solvent lattice. The major mechanisms of strengthening from solute atoms is reported due to dynamic strain aging in steels [14, 15]. In recent time solid solution strengthening has attracted lot of attention, and many new steels have been developed which have shown to increase the strength of the steel due to dynamic strain aging, hence in this study we are specifically concentrating on the solid strengthening mechanism from DSA and its effect on fatigue strength.

1.2 Dynamic strain aging

DSA is described as the interaction between the dislocation and interstitial solute atoms, strengthens the material locating the plastic zone in front of the crack tip [16]. More specifically, solute atom can either segregate to the grain boundary (GB) or diffuse to dislocation to form Cottrell atmosphere preventing further dislocation movement called as strain aging. In other words, once the dislocation emits from the fatigue crack tip, it would be temporarily arrested by obstacles and pinned by solute atoms. The aging phenomena concurrently occurs with straining. It is usually observed that that DSA can even occur at room temperature under low strain rate meaning low dislocation velocity and improve the

fatigue performance to some extent [17]. When the interstitial solute level is normal, DSA tends to occur at the varying temperature ranging from 373-573 K under normal strain rate, while it may happen at low temperature with increase in solute content [18]. In some cases, DSA tends to appear even at very high temperatures at 673-943 K with the solute atom is still strongly effective at elevated temperatures [17]. As a consequence of DSA, the growth of hardness, yield strength and tensile strength is usually observed in steels. When the DSA occurs, several generally physical manifestations are summarized as: serrations in stress-strain curve; negativity strain rate sensitivity; blue brightness; strain localization, etc.

In general, the common manifestation used to determine the DSA affect are the serrations in stress strain curve as shown in Fig. 1.3 at different temperatures [16]. At room temperature, smooth stress-strain curve is obtained, while serration or jerky flow named as Portevin-Le Chatelier (PLC) effect, often occurs at elevated temperatures as shown in Fig. 1.3. When the temperature is beyond the DSA regime, the jerky flow disappears. Moreover, the strain hardening exponent is a little larger in DSA regime than that out of DSA regime due to strain aging hardening and elongation to fracture drop in this regime which is consistent with results of many other steels [17]. It is generally thought that the carbon and nitrogen are the two primary elements controlling the DSA phenomena although many other elements such as oxygen and hydrogen [19, 20] can also be responsible for it.

In austenitic steels such as TWIP steels, the major mechanism of work hardening is attributed to DSA. Normally in FCC steels, serrations are observed in the stress-strain curves when α' -martensitic transformation (FCC $\rightarrow$ BCC or BCT), dynamic precipitation, deformation twinning, and DSA. The DSA is a dominant factor for serrations in polycrystalline Fe-Mn-C austenitic steel at around room temperature. The austenitic steels with interstitial solute atoms interaction are reported to have low stacking fault energy [21, 22]. The mechanisms responsible for this high strain hardening rate is related to the low stacking fault energy (SFE) presented by the austenitic phase. The value of the SFE controls the easiness which dislocations can carry out cross-slip. That is when the SFE is lower, it is easier to form of stacking faults and it is more difficult to cross-slip. This favors the mechanical twinning in relation to the dislocation gliding as a mechanism of plastic deformation of the steel.

In nitrogen alloyed austenitic steels very high strength is attainable. The strengthening mechanisms of N alloying are attributed to (1) interaction/pinning between dislocations and interstitial N atoms because of electrostatic attraction [23], and (2) formation of dislocation N complexes that may exert a dragging effect on dislocations [24]. The big lattice distortion

associated with interstitial N also brings about strengthening. Short range ordering involving substitutional and interstitial elements like Cr-N may also contribute to strengthening in N alloyed steels [25]. On the other hand, in austenitic stainless steels, planar dislocation structures are observed in the nitrogen alloyed steels, and planar slip is the primary deformation mechanism [26]. The formation of coplanar arrays of dislocations and occurrence of planar slip has been attributed to short range order induced by nitrogen [27]. Several researchers have reported that addition of nitrogen results in a decrease in the SFE of austenitic steels [28].

1.3 Fatigue characteristics from Interstitial solute atoms

One of the probable reasons the fatigue limit is enhanced by the Interstitial solute is due to the locking of dislocations, and it is reported that the DSA does contribute in enhancing the fatigue strength of steel. For instance Koyama et al. and Le et al. [29-32] with carbon as solute atom in an ferritic Fe-C binary alloy system reported an increase in fatigue strength, and the major mechanisms in the enhancement of fatigue strength is due to dynamic strain aging (DSA). They clarified certain points in terms of small crack growth behavior. First, transgranular cracks stopped at the grain interior, even at high stress levels, because of strain-age hardening at the crack tips within the grain interiors. Second, the failure mechanism in smooth specimens showed intergranular crack initiation and propagation, this perhaps may be because of lack of carbon segregation to the grain boundaries. Third, when the carbon content was increased from 0.006 mass% to 0.017 mass%, the Fe-C alloy showed a high coxing effect because of suppression of the intergranular fatigue crack initiation by strain aging, and increase in the threshold stress intensity factor ranges for crack growth (ΔK_{th}), because of strain-age hardening at the crack tip. As described earlier, nitrogen is another candidate for solute atom which can show strain aging effect in ferritic steels.

In terms of austenitic steels, carbon as solute atoms, there has been previous research conducted to study about the fatigue behavior. Cornette et al. [33] and Hamada et al. [34] showed that TWIP-type steels exhibit quite high fatigue strength, and the fatigue limit is 0.42–0.48 of the tensile strength. In the high-cycle fatigue (HCF) regime of Fe–0.6C–22Mn and Fe–0.3C–16.4Mn–1.5Al steels, Hamada et al. [35] reported a small amount of cyclic hardening followed by slight softening such that the stress amplitude remained close to the initial one for the majority of the fatigue life. Koyama et al. [36] reported that strain age hardening due to Mn-C couples assists fatigue crack non-

propagation. In terms of solute nitrogen alloyed austenitic steels, Vogt et al, reported that the low cycle fatigue behavior of high nitrogen austenitic and duplex stainless steels is characterized by high stress levels, a cyclic softening and an improved fatigue resistance [37]. Dai et al. reported the high cycle fatigue behavior of high-nitrogen austenitic stainless steel, Fe₂₄Mn₁₃Cr_{0.44}N, at room temperature and showed to have a superior fatigue resistance in comparison with the Fe–Cr–Ni series steel, such as 316L [38].

1.4 Aim of this research

It is known that interstitial–substitutional (I–S) atom pairs such as Mn–C and Cr–N couples in austenitic steels contribute to the superior ductility–strength balance and high work hardening capacity of their respective steels as shown in Fig. 1.4 [39–46]. The high work hardening capacity of an austenitic steel with Mn–C couples can be attributed to the stacking fault energy (SFE)-dependent deformation twinning[43, 46, 47] and dynamic strain aging (DSA) [48–50]. In particular, Mn–C coupling has been reported to promote the effect of DSA on work hardening in Fe–Mn–C-based austenitic steels[49]. In terms of Fe–Cr–N steels, the enhanced dislocation planarity due to Cr–N interaction, is known to increase the work hardening capacity, and also improving the associated strength and elongation [51]. The addition of nitrogen also leads to the introduction of strong elastic distortions into the crystal lattice, which results in solid-solution hardening.

However, the microstructural effects associated with I–S interactions having a dynamic strain aging, and enhanced dislocation planarity effects have not yet been studied in detail in the context of fatigue crack growth behavior and threshold stress intensity factor range (ΔK_{th}) for a small fatigue crack in austenitic steels. In the context of mechanical engineering to employ new steels in structures, evaluation of fatigue damage, and ΔK_{th} of small fatigue crack is most important because major accidental failure is caused by fatigue related phenomena. Hence, in this research firstly we study the effect of dynamic strain aging due to the Mn–C interaction in TWIP steel, and enhanced dislocation planarity due to Cr–N interaction in the High nitrogen steel (HNS) on the fatigue crack growth behavior and resistance in austenitic steels.

In addition, as discussed in section 1.3 solute carbon in ferritic steels is reported to enhance the transgranular fatigue crack resistance and fatigue limit due to dynamic strain aging. Solute nitrogen is considered to be another candidate to cause strain aging in the ferritic steels that can improve the fatigue properties. Distinctly the quantitative evaluation about the single effect of solute nitrogen on fatigue strength is absent in ferritic steel. Hence,

in this research secondly we study the effect of solute nitrogen on the fatigue crack growth behavior and crack resistance in a binary Fe-N ferritic steels.

1.5 Outline of this research

The these consists of six chapters. All chapters are arranged in order to achieve the main theme and objectives of the research work. The thesis is organized as follows.

In today's practical world, fatigue damage study has become a major point of research in terms of mechanical engineering since most of the failure in structures are due to fatigue. Hence for high reliability, convenience, environmental-friendliness in practical engineering, and with the advent of modern industries, many researchers are working on the material strengthening and improving fatigue strength by many different techniques. Solid solution strengthening mechanism has attracted a lot attention and many new high-strength steels with interstitial and substitutional solutes interactions (i-s) have been developed such as twinning-induced plasticity (TWIP) steels, high nitrogen (HN) austenitic steels which have been reported to have high work hardening capacity and ductility due to dynamic strain aging (DSA) effect by Mn-C couples in TWIP steels, and enhanced dislocation planarity by Cr-N couples in HN steels. However, the microstructural effects associated with i-s interactions having a dynamic strain aging, and enhanced dislocation planarity effects have not yet been studied in detail in the context of fatigue crack growth behavior and threshold stress intensity factor range (ΔK_{th}) for a small fatigue crack in austenitic steels. In addition, interstitial carbon in ferritic steels is reported to enhance the transgranular fatigue crack resistance and fatigue limit due to dynamic strain aging. Interstitial nitrogen is thus considered to be another candidate to cause strain aging in the ferritic steels that can improve the fatigue properties. Distinctly the quantitative evaluation about the single effect of interstitial nitrogen on fatigue strength is absent in ferritic steel. Hence, as the main objective of this thesis research, firstly we study the effect of dynamic strain aging due to the Mn-C interaction in TWIP steel, and enhanced dislocation planarity due to Cr-N interaction in the high nitrogen steel (HNS) on the fatigue crack growth behavior and resistance in austenitic steels. Secondly we study the effect of interstitial nitrogen on the fatigue crack growth behavior and crack resistance in a binary Fe-N ferritic steels. The thesis is organized as follows.

Chapter 1 describes a general introduction with research background and aim of this research work. The motivation of this research work is based on the current issues of to develop new materials to improve the fatigue performance during cyclic loading. Based on the further requirement for low energy consuming, safety, environment friendliness, the fatigue performance should be enhanced. Many new steels have been developed with i-s solutes interaction like Mn-C and Cr-N in austenitic steels, that are known to improve the tensile strength and elongation due to DSA effect. In addition, report has been made on interstitial carbon enhancing the fatigue life of ferritic steel. But there is no study on the interstitial nitrogen in BCC steels and Mn-C, Cr-N i-s solutes interaction in FCC steels, on the fatigue performance. Therefore, this thesis mainly focuses on the influence of i-s solutes interaction such as Mn-C and Cr-N in FCC steels, and interstitial nitrogen in BCC steels on the fatigue crack growth resistance.

Chapter 2 firstly reveals the procedures of the four types of steels preparation i.e. Fe-18Cr-14Ni as a reference steel without i-s solute interaction, Fe-23Mn-0.5C as a TWIP steel with Mn-C interaction, Fe-25Cr-1N as a HNS with Cr-N interaction, and Fe-0.0113N as a Fe-N binary steels in detail. The chemical compositions in this study are indicated in wt %. Next the initial microstructures of all four types of steels are shown and discussed. Next, the tensile test results are shown and discussed the variation of tensile strength and dynamic strain aging effect in the Fe-23Mn-0.5C, Fe-25Cr-1N steel, and Fe-18Cr-14Ni steels. The tensile test results for the Fe-0.0113N steel are shown at different strain rates and the results are compared with the Fe-C binary steels and interstitial-free (IF) steel.

Chapter 3 reports the fatigue crack growth behavior of the Fe-25Cr-1N, Fe-23Mn-0.5C, and Fe-18Cr-14Ni steels in terms of microstructure and crack propagation path. Specifically, we carried out rotating bending fatigue tests with a stress ratio of -1 and frequency of 30 Hz, and subsequent observations of the specimen surface by replica technique, fracture surface by scanning electron microscopy, and detailed microstructure in a vicinity of the fractured part by electron backscatter diffraction observations. Fatigue limits of the Fe-25Cr-1N, Fe-23Mn-0.5C and Fe-18Cr-14Ni steels were 310 MPa, 260 MPa and 110 MPa, respectively. The Fe-25Cr-1N, Fe-23Mn-0.5C steels showed a lower fatigue crack propagation rate and higher fatigue limit compared to the Fe-18Cr-14Ni steel without i-s solute interaction effect. The following effects on crack initiation and growth are discussed in the Fe-23Mn-0.5C austenitic steel with Mn-C interaction. As a negative effect, it is considered that deformation-induced decomposition of Mn-C couples results in local

softening, causing strain localization that assists crack initiation along $\{111\}\gamma$ slip planes. On the other hand, the ease of crack initiation along the slip planes assists crack branching, zigzag crack growth, and subcrack formation. These factors enhance roughness-induced crack closure: a positive effect. In addition, the formation of subcracks works for stress release at a crack tip: another positive effect. Discussions on dislocation structure and crack growth behavior in the Fe-25Cr-1N steel with Cr-N interaction are as follows. The short crack region exhibited a planar glide dislocation pattern, but multiple slip systems were activated as the crack lengthened. As the dislocation pattern remained planar on each slip plane, the crack propagation occurred along the $\{111\}\gamma$ slip planes, even in the long crack.

Chapter 4 reports the underlying mechanisms of the enhanced small fatigue crack resistance in the Fe-23Mn-0.5C and Fe-25Cr-1N austenitic steel. Particularly, I applied electron channeling contrast imaging near tips of non-propagating fatigue cracks in both the austenitic steel with i-s interaction and in the Fe-18Cr-14Ni steel without significant amount of interstitials. In addition, I made quantitative evaluation of the ΔK_{th} for small-fatigue-crack propagation in the case of different notch sizes and discussed the variation of ΔK_{th} with the occurrence of i-s interactions in dislocation microstructure near a non-propagating fatigue crack tip. The Fe-25Cr-1N and Fe-23Mn-0.5C steel showed enhanced crack resistance in comparison to the Fe-18Cr-4Ni steel. However, the mechanism for the crack resistance in the Fe-25Cr-1N and Fe-23Mn-0.5C steels are different.

Chapter 5 reports the fatigue crack initiation and propagation behavior of a water-quenched nitrogen-supersaturated fully ferritic steel (Fe-0.0113N). The results were compared to the Fe-C binary steels, and IF steel. Tension tests were conducted at initial strain rates of 10^{-3} s^{-1} , 10^{-4} s^{-1} , and 10^{-5} s^{-1} at room temperature, and tension-compression fatigue tests were conducted at room temperature on the Fe-0.0113N steel. The rotating bending fatigue tests with a stress ratio of -1 and frequency 30 Hz were carried out. The Fe-0.0113N steel showed a fatigue limit at 150 MPa and a non-propagating fatigue crack at the fatigue limit. The fatigue limit of Fe-0.0113N steel is lower than that of Fe-0.017C (210MPa) and comparable to that of Fe-0.0063C (150MPa), indicating a lower effect of nitrogen than carbon as an interstitial solute.

Chapter 6 is summarized for the results obtained by the present study, the Mn-C and Cr-N have positive effects on the fatigue crack growth rate and improves the fatigue limit in the austenitic steels. In terms of ferritic steel, the Fe-N steel showed lower fatigue limit in comparison with the Fe-C steel, detailed findings are listed out in the general conclusions.

1.6 List of paper appended paper during Ph.D. period: include publication

[1] Kishan Habib, Motomichi Koyama, Hiroshi Noguchi, “Impact of Mn–C couples on fatigue crack growth in austenitic steels: Is the attractive atomic interaction negative or positive?”, *International Journal of Fatigue*, 99, 1, 1–12, June, 2017.

[2] Kishan Habib, Motomichi Koyama, Toshihiro Tsuchiyama, Hiroshi Noguchi, “Fatigue crack non-propagation assisted by nitrogen-enhanced dislocation planarity in austenitic stainless steels”, *International journal of Fatigue*, 104, 1, 158–170, July, 2017.

[3] Kishan Habib, Motomichi Koyama, Toshihiro Tsuchiyama, Hiroshi Noguchi, “Visualization of dislocations through electron channeling contrast imaging at fatigue crack tip, interacting with pre-existing dislocations”, *Materials Research Letters*, 6, 1, 61-66, October, 2017.

[4] Kishan Habib, Motomichi Koyama, Toshihiro Tsuchiyama, Hiroshi Noguchi, “ECCI characterization of dislocation structures at a non-propagating fatigue crack tip: towards understanding the effects of Mn-C and Cr-N couples on crack growth resistance”, *Metallurgy and Material Transactions - A*, February, 2018 (Under Review).

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1.8 Figures



Fig.1.1 (a) Fatigue failure of engine crankshaft [2], (b) Fatigue cracking on the bridge girder [3]

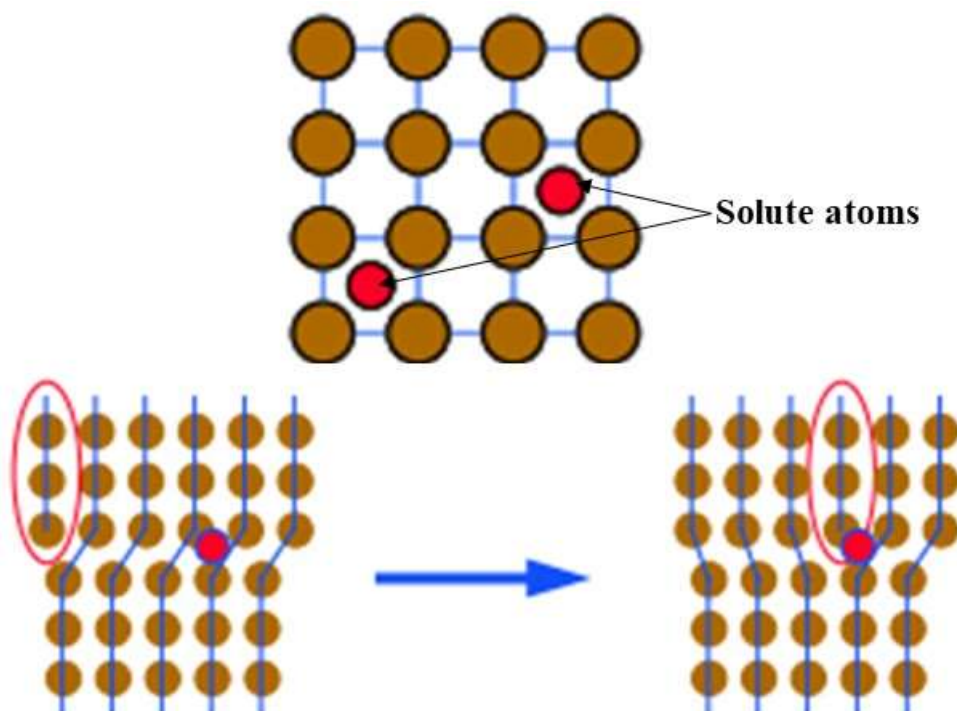


Fig. 1.2 Interstitial-Solute atom structure in the crystal lattice [13].

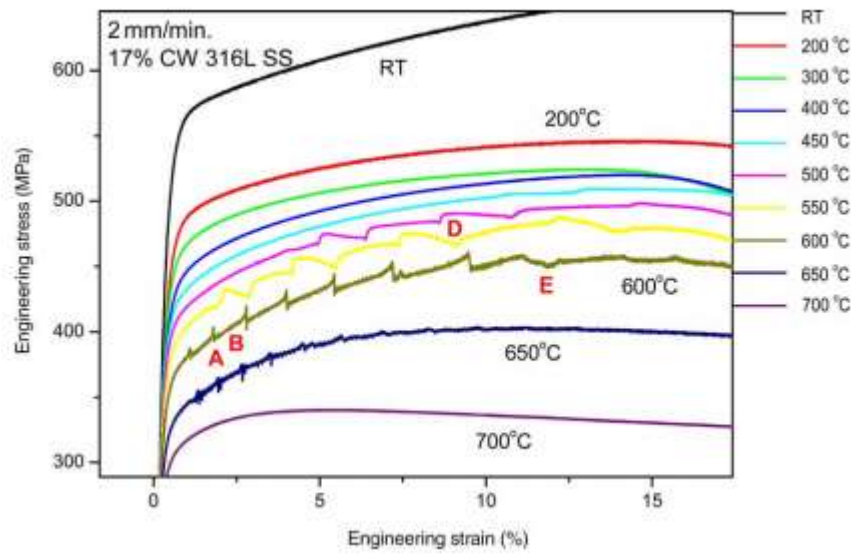


Fig. 1.3 An example of stress-strain curve representing the DSA effect at varying temperatures [16].

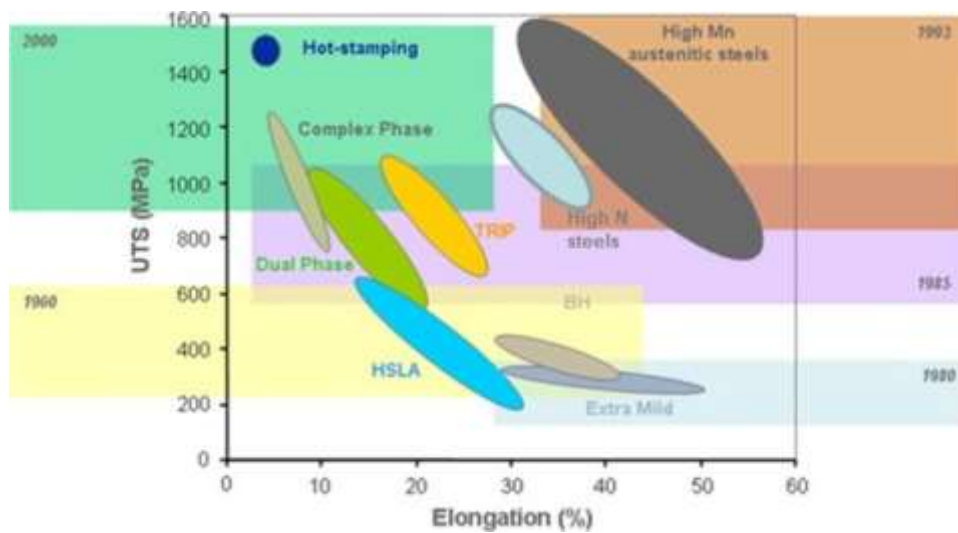


Fig. 1.4 Development of new steels for structural applications [39, 46].

Chapter 2

Model Steels Selection, Preparation, Initial Microstructure & Tensile Test Results

2.1 Introduction

Solute atoms are known to increase the mechanical properties in austenitic and ferritic steels, and the aim of this thesis is to evaluate the new generation steels with Interstitial solute atoms such as Carbon, and Nitrogen on the fatigue growth resistance. In terms of austenitic steels, such as TWIP and HNS solute atoms carbon and nitrogen plays an important role in high strength, ductility, and formability associated with their high work hardening capacity [1-3]. As discussed in Chapter 1 the major mechanisms of higher work hardening in the TWIP and HNS is due to DSA and enhanced dislocation planarity respectively. But there has been no report on the effect of DSA due to Mn-C couples and enhanced dislocation planarity due to Cr-N on the fatigue crack growth resistance. Hence in this research three types of austenitic steels are selected, firstly TWIP steel with a chemical composition of Fe-23Mn-0.5C with Mn-C couples, secondly an HNS with a chemical composition of Fe-25Cr-1N with Cr-N couples. To compare the results of the Interstitial-solute interaction on the fatigue crack behavior and resistance, a third type of austenitic steel selected is the Fe-18Cr-14Ni steel, which does not have any I-S atom interaction, has a stable austenite phase and low stacking fault energy.

In addition, interstitial solute carbon is reported to enhance the transgranular fatigue crack resistance and improved the fatigue limit of ferritic steels [4, 5]. From the viewpoint of the carbon effect, the kinetic effect is important for understanding the underlying mechanism of fatigue resistance improvement, i.e., strain-age hardening. In this context, nitrogen is expected to be another candidate to improve fatigue resistance because nitrogen is a typical interstitial solute element that shows significant capability to cause strain aging in steels [6]. However, in comparison with the carbon effect, the nitrogen effect has not been examined well in terms of fatigue crack resistance. Therefore, in this study an Fe-N binary fully ferritic steel is the other type of steel selected, and compared with the results of the Fe-C, and IF ferritic steels.

Before the fatigue test in the thesis, tensile properties for presented materials, such as yield strength, ultimate tensile strength (UTS) and elongation are to be investigated, which is better for understanding the fatigue performance in the following fatigue tests.

2.2 Model steels preparation

As discussed above, in this research three types of austenitic steels are used i.e. Fe-25Cr-1N, Fe-23Mn-0.5C, and Fe-18Cr-14Ni steels, and one type of binary ferritic steel i.e. Fe-0.0113N steel. Detail chemical composition of austenitic steels is given in Table. 2.1, and ferritic steel composition is given in Table. 2.2 in comparison with Fe-C and IF steels. The preparation method of all the steels is explained in detail below.

i. Fe-23Mn-0.5C, and Fe-18Cr-14Ni steels.

The ingots were prepared by vacuum induction melting, and were then forged and groove-rolled at a temperature of 1273 K. Next, the hot rolled bars were solution treated at 1273 K for 1 h and were subsequently water-quenched to suppress the uncontrolled formation of second phases and segregation.

ii. Fe-25Cr-1N steel

The ingot was produced by induction melting in a vacuum, homogenization at 1473 K, and hot-rolling to produce a 10 mm thick plate. The fatigue test specimen as shown in Fig. 2.1 was cut from the ingot and solution-nitriding was performed for the fatigue specimen in a furnace as shown in Fig. 2.2 [7] at 1473K for 86.4 ks in a nitrogen gas atmosphere at 0.1 MPa, followed by water quenching to ensure that no austenite decomposition or nitride precipitation occurred on cooling. As coarse-grained high nitrogen steels can exhibit brittle intergranular cracking, grain refinement was carried out on the solution-nitrided specimen by heat treatment in two stages [8]. The specimen was first subjected to isothermal heat treatment at 1173 K for 0.42 ks, followed by re-austenitization at 1473 K for 0.3 ks and water cooling. The specimen was then mechanically and electrochemically polished to remove the damage layer before fatigue testing. The tensile properties of the Fe-25Cr-1N steel have been reported to be improved as a result of grain refinement [8].

iii. Fe-0.0113N steel

The ingot was solution treated at 700° C for 3.6 ks and subsequently water quenched to suppress cementite formation and nitrogen segregation at the grain boundaries. The ingots were preserved in refrigerator at –87°C at all times except during sample preparation.

2.3 Initial microstructure

Figure 2.3 (a-c) shows the initial microstructure of the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N steels consisting of a single austenite phase before fatigue testing. The average grain sizes of the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N steels were measured to be 42 μm , 33 μm , and 22 μm , respectively, including annealing twin boundaries.

Figure 2.4 shows the initial microstructure of the Fe-0.0113N steel, consisting of a single ferrite phase before fatigue testing. The average grain size was measured to be 180 μm .

2.4 Tensile test procedure and results.

Figure 2.5 shows the diagram of the tensile test specimen used for the tensile test. Tensile test for the Fe-23Mn-0.5C, Fe-25Cr-1N, Fe-18Cr-14Ni steels were conducted at room temperature at a strain rate of 10^{-3}s^{-1} . Figure 2.5 shows the engineering stress–strain (SS) curves of the three steels. The Fe-25Cr-1N and Fe-23Mn-0.5C steels showed higher work hardening capacities and higher tensile strengths than the Fe-18Cr-14Ni steel. In addition, the Fe-23Mn-0.5C steel showed serrations in its SS curve. The 0.2% proof stress, tensile strength, and elongation are listed in Table 2.3.

Tensile test for the Fe-0.0113N steel were conducted at the strain rates of 10^{-3}s^{-1} , 10^{-4}s^{-1} , and 10^{-5}s^{-1} at room temperature. Figure 2.6(a) shows the engineering stress–strain (SS) curve for the Fe–0.0113N steel at an initial strain rate of 10^{-4}s^{-1} in comparison with the SS curves of the Fe–0.017C, Fe–0.006C, and IF steels plotted from the previous paper [9]. The 0.2% proof strength, tensile strength, and elongation values are listed in Table 2.4. Figure 2.6(b) shows the SS curves of the Fe–0.0113N steel plotted at different strain rates. No serrations are observed in the SS curve at any strain rate. Therefore, it is evident that no dynamic strain aging (DSA) occurred in the nitrogen steel at room temperature.

2.5 Effect of DSA on the tensile strength.

The serrations observed in the SS curve (Fig. 2.5) proves that there is an occurrence of DSA in the Fe-23Mn-0.5C steel. The DSA is caused by an interaction between solute atoms and moving dislocations. The possible types of interacted solute atoms in FCC metals are interstitial atoms [10], substitutional atoms [11], and interstitial – substitutional atom couples [12]. The DSA in the Fe-Mn-C steels has been reported to be attributed to the interaction between moving dislocations and Mn-C couples [12, 13]. Hence, due to strain age hardening the Fe-23Mn-0.5C steel showed higher tensile and work hardening effect in comparison with the Fe-18Cr-14Ni steel.

2.6 Effect of Nitrogen on the Tensile strength.

The S-S curve shown in Fig. 2.5 reveals that the tensile strength of the Fe-25Cr-1N steel is superior to that of the Fe-18Cr-14Ni steel. The increase in the tensile strength of the Fe-25Cr-1N steel is mainly attributed to the solid solution and grain boundary hardening that occur when the N is in the interstitial solid solution [14, 15] and also to the grain refinement of the Fe-25Cr-1N steel [8].

The Fe-0.0113N steel showed a lower tensile strength than the Fe-0.006C and Fe-0.017C steels (Fig. 2.6(a)) because of the absence of DSA effect in the Fe-0.0113N steel. No serrations were observed on the SS curve in the Fe-0.0113N steel (Fig. 2.6(b)) even at lower strain rates. This is perhaps because of the low diffusivity of nitrogen at room temperature in the Fe-0.0113N steel. Baird et al. [16] determined an activation energy of 63–84 kJ mol⁻¹ for the onset of serrations in Fe-N steel, and strain aging occurred at temperatures above 373 K.

2.7 Section Conclusion

Based on the tension test results, the Fe-25Cr-1N, and Fe-23Mn-0.5C steel showed higher tensile strength than the Fe-18Cr-14Ni steel due to the effects of solute atoms such as carbon and nitrogen in the austenitic steel. The Fe-0.0113N steel showed lower tensile strength than the Fe-0.017C and Fe-0.006C steels because of the absence of DSA effect in the Fe-0.0113N steel.

2.8 References

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2.9 Figures and Tables

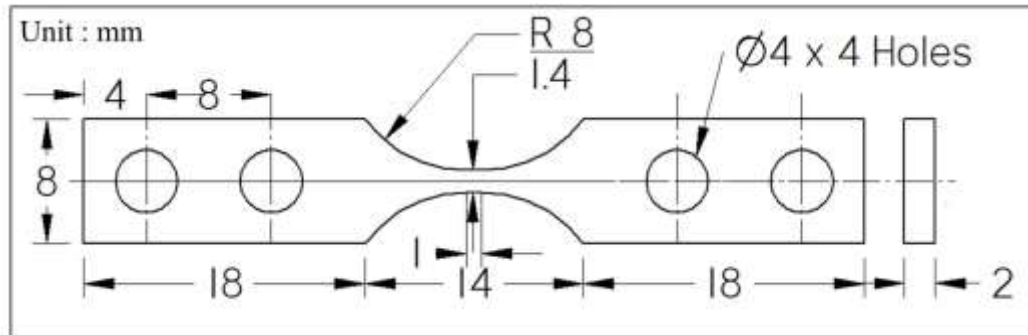


Fig. 2.1 Fatigue test specimen design for nitrogen treatment

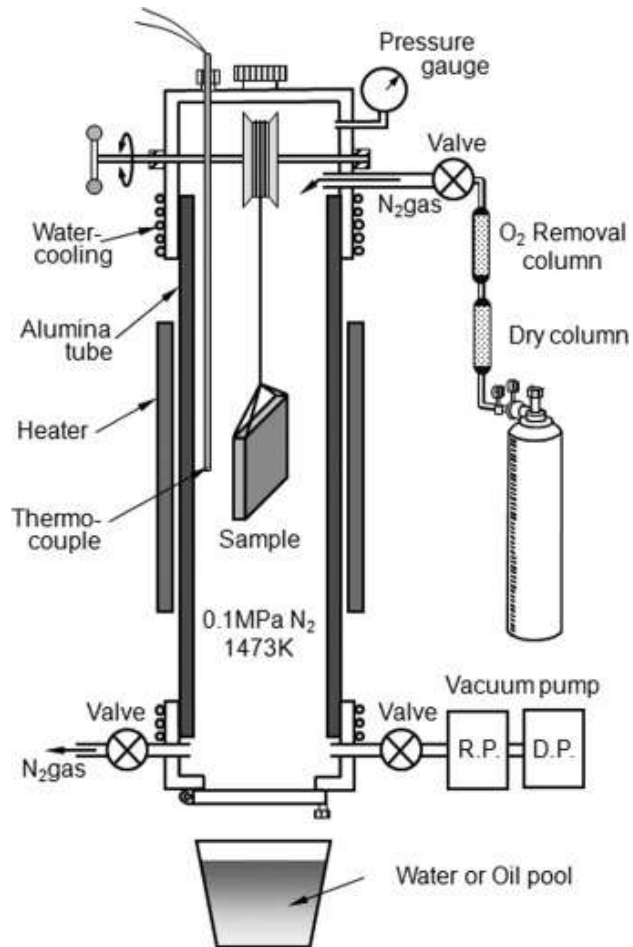


Fig. 2.2 Schematic illustration of the furnace for solution nitriding [7]

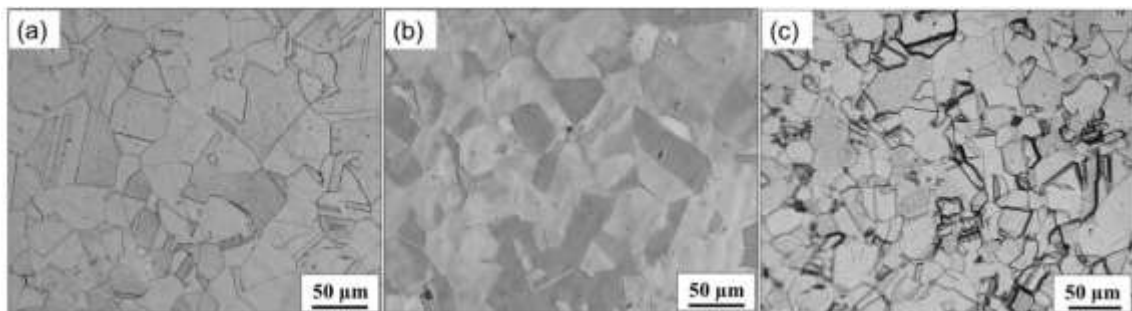


Fig. 2.3 Initial microstructure of the (a) Fe-18Cr-14Ni, (b) Fe-23Mn-0.5C, (c) Fe-25Cr-1N steels.

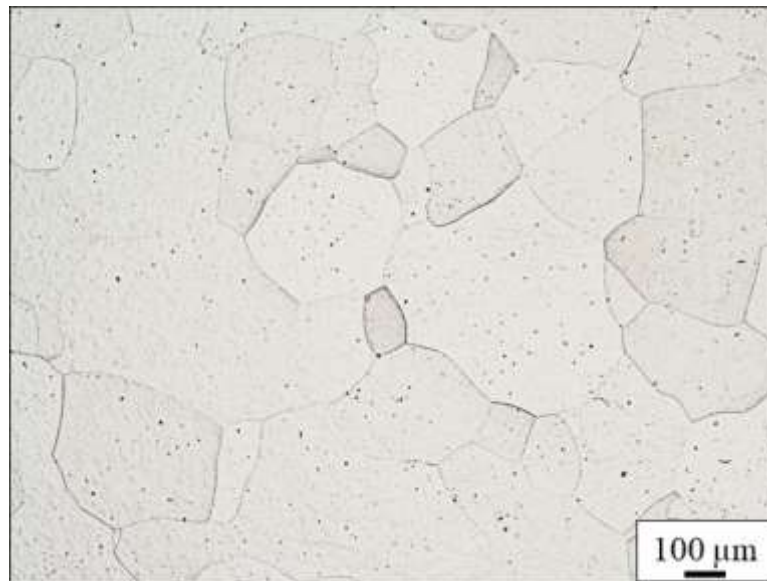


Fig. 2.4 Initial microstructure of the Fe-0.013N steels.

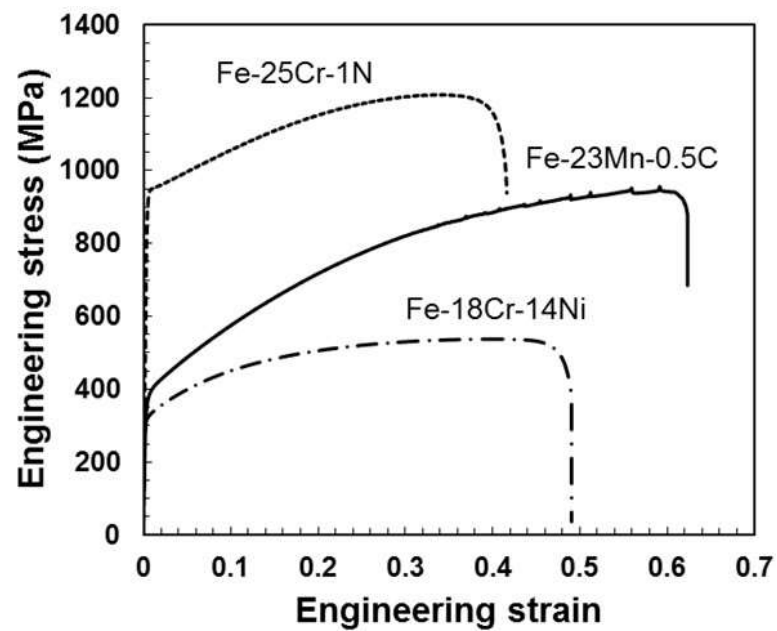


Fig. 2.5 Engineering stress–engineering strain (SS) curves of the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N steels.

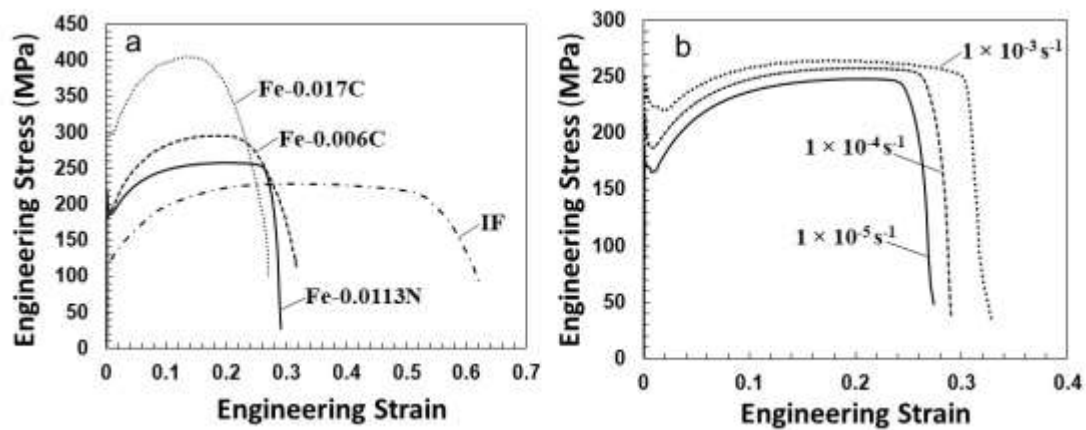


Fig. 2.6 Engineering stress–engineering strain (SS) curves. (a) SS curve of the Fe–0.0113N steel in comparison to the Fe–0.017C, Fe–0.006C, and IF steel curves. (b) SS curves of the Fe–0.0113N steel at different strain rates. The SS curves of the Fe–0.017C, Fe–0.006C, and IF steels are reproduced from a previous paper [9]

Table 2.1 Chemical compositions of three austenitic steels used in this study (mass%).

Steel	Ni	Mn	Cr	Si	C	P	S	N	O	Al	Fe
Fe-25Cr-1N	-	<0.01	25.4	0.03	0.002	0.006	0.0005	1.0	0.0036	0.039	Bal.
Fe-23Mn-0.5C	0.003	22.6	<0.001	0.027	0.51	<0.001	0.0072	-	-	-	Bal.
Fe-18Cr-14Ni	14.6	<0.001	18.8	<0.001	0.002	<0.001	0.0014	-	-	-	Bal.

Table 2.2 Chemical compositions of Fe-N ferritic steel used in this study in comparison with the Fe-C and IF steels (mass%).

Steel	N%	C%	Si%	Mn%	P%	S%	Ni%	Ti%	Al%	O*
Fe-0.0113N	0.0113	0.0011	<0.003	<0.003	<0.002	<0.0003	<0.003	<0.002	<0.002	29
IF steel [4]	0.0008	0.0019	0.009	<0.003	<0.002	<0.0003	---	0.029	0.028	15
Fe-0.0063C [4]	0.0007	0.0063	0.005	<0.004	0.0003	0.0011	---	---	0.048	<10
Fe-0.017C [4]	0.0009	0.017	<0.003	<0.003	<0.002	<0.0003	<0.003	<0.002	0.052	15

* indicates mass ppm

Table 2.3 0.2 % proof strength, tensile strength and elongation of the Fe-25Cr-1N, Fe-23Mn-0.5C, and Fe-18Cr-14Ni steels.

Steel	0.2% Proof Strength MPa	Tensile Strength MPa	Elongation %
Fe-25Cr-1N	950	1200	41
Fe-23Mn-0.5C	350	940	61
Fe-18Cr-14Ni	290	520	50

Table 2.4 0.2% proof strength, tensile strength, elongation, and Vickers hardness values of the Fe-0.0113N, Fe-0.017C, Fe-0.006C, and IF steels.

Steel	0.2% Proof Strength (MPa)	Tensile Strength (MPa)	Elongation (%)	Vickers hardness (Hv)
Fe-0.0113N	220	265	29	96.4
Fe-0.017C [9]	300	400	31	125.7
Fe-0.006C [9]	185	300	28	83.6
IF [9]	115	235	62	61

Chapter 3

Influence of Mn–C and Cr-N couples on fatigue crack growth behavior in austenitic steels

3.1 Introduction

Following the tension tests on the austenitic steels in Chapter 2, the DSA from Mn-C and enhanced dislocation planarity from Cr-N interaction showed a positive effect on the tensile strength in comparison with the normal steel without any solute atom interaction. But it is also important to analyze the effects of Mn-C and Cr-N on the fatigue damage, as discussed in Chapter 1 fatigue failure is one most important in terms of mechanical engineering for long lives structures.

Fatigue crack initiation and propagation limits are the principal factors that influence the fatigue limit, which is the most important criterion for structure designs, [1]. In steels, the crack propagation limit is particularly important in specimens with pre-existing damage such as flaws [2]. In general, strain age hardening [3-6] at a mechanically small crack tip [7], crack closure mechanisms such as plasticity-induced crack closure [8], and crack branching from the crack tip [9] are factors controlling the non-propagating fatigue behavior of steels. Therefore, in order to demonstrate the importance of Mn-C and Cr-N coupling we conducted a fatigue test to study the influence of DSA and enhanced dislocation planarity on the fatigue crack growth resistance and fatigue limit in comparison with stable Fe-Cr-Ni without any I-S interactions.

3.2 Experimental Procedure

3.2.1 Fatigue testing method

The materials used in this study were the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N (mass%) steels. Detailed chemical composition is given in Table 2.1. The fatigue tests were performed on an Ono-type rotating bending fatigue test machine at room temperature, with a stress ratio of -1 and frequency 30 Hz at room temperature. Design of fatigue test specimen is shown in Fig. 3.1(a). The specimen was fixed on a round bar jig as shown in Fig. 3.1 (b-c) to conduct the fatigue test. A hole with a diameter and depth both equal to 100 μm was drilled in the center of the specimen on the top surface to serve as a

controlled crack initiation site. The fatigue cracks were observed by optical microscopy with the replica technique. The fracture surface was observed with a scanning electron microscope (SEM) at an accelerating voltage of 15 kV. Fractured specimens were mechanically polished from the side as shown in Fig. 3.1(d), and electron backscatter diffraction (EBSD) measurements were performed at a distance of 100 μm (short crack), and 400 μm (long crack) from the bottom of the hole, with a beam step size of 50 nm, and an accelerating voltage of 20 kV. In addition, electron channeling contrast imaging (ECCI) was carried out at an accelerating voltage of 30 kV.

3.3 Experimental Results

3.3.1 Fatigue life and relevant crack growth characteristics

Figure 3.2 (a) shows a diagram of the number-of-cycles-to-failure plotted against stress amplitude for the Fe-23Mn-0.5C and Fe-18Cr-14Ni steels. The stress amplitude that does not result in failure at 2×10^7 cycles is defined as the fatigue limit in this study. The fatigue limits for the Fe-23Mn-0.5C and Fe-18Cr-14Ni steels were 260 MPa and 110 MPa, respectively. Since the differences in the fatigue limit and the fatigue life are generally explained by hardness or tensile strength, the number-of-cycles-to-failure is plotted against stress amplitude normalized by tensile strength in Fig. 3.2(b). It is clear that the fatigue limit of the Fe-23Mn-0.5C steel is higher than that of the Fe-18Cr-14Ni steel even with the normalized data set. In addition, when compared to the Fe-18Cr-14Ni steel, the fatigue lifetimes of the Fe-23Mn-0.5C steel are longer at low normalized stress amplitudes and shorter at high normalized stress amplitudes.

Figure 3.3 (a) shows a plot of the number of cycles to failure against stress amplitude for the Fe-25Cr-1N and Fe-18Cr-14Ni steels. The fatigue strength of the Fe-25Cr-1N steel is 310 MPa. The fatigue strength of the Fe-25Cr-1N steel is clearly higher than that of the Fe-18Cr-14Ni steel, even when normalized by the tensile strength as shown in Fig 3.3(b). In contrast, the fatigue life of the Fe-25Cr-1N steel at higher normalized stress amplitudes is almost identical to that of the Fe-18Cr-14Ni steel.

3.3.2 Fatigue crack growth behavior

Figure 3.4(a) shows the crack growth curves for the two steels when the ratio between stress amplitude and tensile strength was fixed at 0.37 in both cases. The fatigue crack growth rate of the Fe-18Cr-14Ni steel is faster than that of the Fe-23Mn-0.5C steel as shown in Fig. 3.4 (a). The sudden increase of the crack growth rate of the Fe-23Mn-0.5C

steel indicated by the arrows in Fig. 3.4(b) results from coalescence of cracks as discussed later. The lower fatigue crack growth rates of the Fe-23Mn-0.5C steel shown in Fig. 3.4(b) explains the longer fatigue life at relatively low stress amplitudes compared to the Fe-18Cr-14Ni steel (Fig. 3.2).

Figure 3.5 shows the crack growth curves of the two steels at the same stress amplitude to tensile strength ratio of 0.36. The fatigue crack growth for the Fe-25Cr-1N steel was slower compared to that of the Fe-18Cr-14Ni steel (Fig. 3.5(a)). A sudden increase in the crack growth rate of the Fe-25Cr-1N steel due to the crack coalescence observed, indicated by an arrow in Fig. 3.5(b). The lower fatigue crack growth rate of the Fe-25Cr-1N steel results in a higher fatigue life at low stress amplitudes compared to the Fe-18Cr-14Ni steel (Fig. 3.3). Microscopic behavior pertaining to the crack growth is presented in the following section for all three steels.

i. Fe-18Cr-14Ni Steel

Figure 3.6 shows replica images of the Fe-18Cr-14Ni steel during the fatigue test. A crack was initiated from both sides of the drill hole at 3×10^4 cycles as shown in Fig. 3.6(a). The crack subsequently propagated almost perpendicularly to the loading direction as shown in the Fig. 3.6(b-g). A considerable number of intensive slip bands formed around the crack [Fig. 3.6 (d₁, d₂, f₁, and f₂)], resulting in crack branching at many points. The tips of the branched cracks are aligned parallel to the main crack tip as indicated by the red and white arrows in Fig. 3.6 (e₁, e₂, g₁, and g₂).

Figure 3.7 shows the SEM fracture surface images of the Fe-18Cr-14Ni steel. Figure 3.7(a) shows an overview of the fracture surface, and Fig. 3.7(b) shows the crack initiation around the drill hole. The early propagation region has surface features with a river pattern as shown in Fig. 3.7(c). A subcrack was also observed as indicated by a red arrow in Fig. 3.7(c₁). The river pattern gradually disappeared with increasing crack length; and ductile fatigue striations appeared instead, as indicated by the red arrows in Fig. 3.7(d₁).

Figure 3.8 shows results of EBSD measurements performed at a location 100 μm from the bottom of the drill hole in the Fe-18Cr-14Ni fractured specimen. The EBSD images were taken from the cross-section of the region indicated by arrows in Fig. 3.8(a). The cross-sectional SEM image is shown in Fig. 3.8(b), while Fig. 3.8(c) shows the inverse pole figure map in the rolling direction (RD-IPF) that is parallel to loading direction. In the RD-IPF map, $\{111\}_\gamma$ plane traces have been highlighted as red lines. Here, some parts of the crack surface are along $\{111\}_\gamma$ planes highlighted by the yellow lines near the fracture surface. Figure 3.8(d) shows the grain orientation deviation angle map (GROD) that

corresponds to the degree of plastic deformation. In the GROD map, the maximum deviation angle is approximately 13° , and the distribution of deviation angles is heterogeneous. Figure 3.8(e) shows an image quality (IQ) map coupled with identification of $\Sigma 3$ twin boundaries. The IQ value deteriorates when two Kikuchi patterns of the parent and twin crystals are overlapped. The reduction in IQ values depicts even thin deformation twin plates [10-12]. The IQ map exhibits only annealing twins, and no plate-like microstructure such as deformation twins.

Figure 3.9 shows the results of EBSD measurements performed at a location 400 μm from the bottom of the drill hole in the Fe-18Cr-14Ni fractured specimen. The EBSD images were taken from the cross-section of the part indicated by arrows in Fig. 3.9(a). The corresponding cross-sectional SEM image is shown in Fig. 3.9(b). In the RD-IPF map [Fig. 3.9 (c)], $\{111\}_\gamma$ plane traces have been highlighted as red lines; this indicates that the crack surface in this region does not lie along $\{111\}_\gamma$ planes at any point. In the GROD map [Fig. 3.9(d)] the maximum deviation angle is approximately 23° , which is remarkably higher than that for the short crack. In addition, the highly deformed region is homogenous along the fracture surface. Similar to Fig. 3.8(e), the corresponding IQ map shown in Fig. 3.9(e) does not show plate-like microstructures. In addition, Fig. 3.10 shows an ECC image at the crack length of 500 μm from the bottom of the drill hole. This image shows deformation twin plates depending on crystallographic orientation as indicated by yellow arrows, but the twin plate is very thin (in Fig. 3.10, the twin plates are thinner than 300 nm), and its number is much smaller than that reported in previous papers about fatigue deformation behavior of TWIP steels [13, 14].

ii. Fe-23Mn-0.5C steel

Figure 3.11 shows replica images of the Fe-23Mn-0.5C steel during the fatigue test at a stress amplitude of 350 MPa. A fatigue crack was initiated at 3×10^4 cycles at right side of the drill hole as shown in Fig. 3.11(a), while another crack was initiated at 6×10^4 cycles on the left side of the drill hole as shown in Fig. 3.11(c). The crack subsequently propagated almost perpendicular to the loading direction on both sides of the drill hole as shown in Fig. 3.11(d-g). Many subcracks initiated along the grain boundary shown by the red arrows in Fig. 3.11(d₁, e₂, f₁, and g₂). One of these subcracks coalesced with the main crack as shown by the yellow arrow in Fig. 3.11(e₁). The crack propagation path shows a zigzag pattern as shown in Fig. 3.11(d₂, f₂, g₁, and g₂). Compared to the Fe-18Cr-14Ni steel, the frequent appearance of fatigue subcrack initiation, and subsequent coalescence are

crucial features of the fatigue crack propagation in the Fe-23Mn-0.5C steel in terms of surface crack growth observed by the replica method.

Figure 3.12 shows the SEM fractographs of the Fe-23Mn-0.5C steel. Fig. 3.12(a) shows an overview of the fracture surface. Figure 3.12(b) shows that distinct striation-like features exist even in the vicinity of the drill hole as indicated by red arrows, although the typical flat feature of a crack initiation part was also observed around the drill hole. Fig. 3.12(c and d) show the early and intermediate fatigue crack propagation regions. It is noteworthy that both the striation-like and flat surface features were observed even in the relatively long fatigue crack region. Examples of the striation-like and flat surface features are indicated by red and white arrows in Fig. 3.12(c₁ and d₁).

Figure 3.13 shows the results of EBSD measurements performed at a location 100 μm from the bottom of the drill hole in the Fe-23Mn-0.5C fractured specimen. The EBSD images were taken from the cross-section of the part indicated by arrows in Fig. 3.13(a). The cross-sectional SEM image is shown in Fig. 3.13(b). In the RD-IPF map [Fig. 3.13 (c)], $\{111\}_\gamma$ plane traces have been highlighted as red lines. Here, some parts of the crack surface are along $\{111\}_\gamma$ planes highlighted by the yellow lines. In the GROD map [Fig. 3.13(d)], the maximum deviation angle is approximately 18° . No plate-like microstructure was observed in the corresponding IQ map [Fig. 3.13(e)].

Figure 3.14 shows the results of EBSD measurements performed at a location 400 μm from the bottom of the drill hole for the Fe-23Mn-0.5C fractured specimen. The EBSD images were taken from the cross-section of the region indicated by arrows in Fig. 3.14(a). The cross-sectional SEM image is shown in Fig. 3.14(b). In the RD-IPF map [Fig. 3.14 (c)], $\{111\}_\gamma$ plane traces have been highlighted as red lines. Here also, some parts of the crack surface are along $\{111\}_\gamma$ planes highlighted by yellow lines near the fracture surface. In the GROD map [Fig. 3.14(d)] the maximum deviation angle is 19° , which is nearly equal to that for the short crack. In the IQ map shown in Fig. 3.14(e), deformation twin plates were observed as indicated by yellow arrows.

iii. Fe-25Cr-1N Steel

Figure 3.15 shows the replica images of the Fe-25Cr-1N steel obtained during fatigue testing at stress amplitude of 450 MPa. A fatigue crack was initiated at 5×10^4 cycles from the left side of the drill hole (Fig. 3.15(b)) and 6×10^4 cycles from the right side of the drill hole (Fig. 3.15(c)). The cracks subsequently propagated in a direction almost perpendicular to the loading direction on both sides of the drill hole as shown in Fig. 3.15(d-g). Many

subcracks initiated near the main crack indicated by red arrows in Fig. 3.15(d₁, d₂, e₂, f₁, g₁), and some coalesced with it indicated by yellow arrows in Fig. 3.15(e₁) and (f₂). Crack branching occurred at one point, as shown by white and blue arrows in Fig. 3.15(g₂). The crack propagation was rigorous, exhibiting a zigzag pattern as observed in Fig. 3.15(e₁) and (g₂). Compared with the Fe-18Cr-14Ni steel, subcracks initiated at many points in the Fe-25Cr-1N steel and the subsequent crack coalescence and branching were the main attributes of its surface crack propagation behavior.

Figure 3.16 shows the SEM fractographs obtained from the Fe-25Cr-1N steel, where Fig. 3.16(a) gives an overview of the fracture surface. Figure 3.16(b) indicates the crack initiation site, with striation-like features observed in the vicinity of the hole (shown by red arrows) along with flat surface features (indicated by white arrows). Figure 3.16(c) shows the early propagation region, which contain subcracks and striations as indicated by red arrows in the magnified image (Fig. 3.16(c₁)). As the crack length increased, the intermediate propagation region revealed that the fracture surface was intergranular (Fig. 3.16(d)), which increased as shown in the late propagation region (Fig. 3.16(e)).

The images in Fig. 3.17 labeled (a) show the results of the EBSD measurements performed at a location 100 μm from the bottom of the drill hole in the Fe-25Cr-1N fractured specimen. The EBSD images were taken from the sample cross-section indicated by arrows in Fig. 3.17(a₁), and the associated cross-sectional SEM image is shown in Fig. 3.17(a₂). In the RD-IPF map (Fig. 3.17(a₃)), the $\{111\}_{\gamma}$ plane traces have been highlighted by red lines; the map reveals that in some regions the crack surface did occur along the $\{111\}_{\gamma}$ planes, indicated by yellow lines near the fracture surface. The GROD map (Fig. 3.17(a₄)) gives a maximum deviation angle of approximately 10° , and shows that the deviation angle distribution is heterogeneous. Plate-like microstructure was not observed in the corresponding IQ map, as shown in Fig. 3.17(a₅).

The images in Fig. 3.17 labeled (b) show the results of the EBSD measurements performed at a location 400 μm from the bottom of the drill hole in the Fe-25Cr-1N fractured specimen. The EBSD images were taken from the sample cross-section indicated by arrows in Fig. 3.17(b₁), and the corresponding cross-sectional SEM image is shown in Fig. 3.17(b₂). The RD-IPF map (Fig. 3.17(b₃)) shows the $\{111\}_{\gamma}$ plane traces highlighted by red lines; they indicate that the crack surface in this region also appeared partially along the $\{111\}_{\gamma}$ planes. The GROD map (Fig. 3.17(b₄)) gives a maximum deviation angle of approximately 9° , which is almost identical to the value obtained from the short crack. No plate-like microstructure was in the corresponding IQ map, as shown in Fig. 3.17(b₅).

Figure 3.18 shows the ECC images of the Fe-25Cr-1N steel. Figure 3.18(a) shows the dislocation structure at the short crack (100 μm from the bottom of the drill hole), which is controlled by planar glide as shown in Fig. 3.18(a₁). As the crack lengthens (500 μm from the bottom of the drill hole, Fig. 3.18(b)), multiple slip systems are activated as shown in Fig. 3.18(b₁). However, it should be noted that the pattern of dislocations on each slip plane remains planar, even in the long crack region.

Figure 3.19 shows a further ECC image of the Fe-25Cr-1N steel at a grain boundary located far from the fracture edge. The image shows the pile-up of planar dislocations at the grain boundary. The local contrast variation near the grain boundary (outlined by dashed yellow lines) indicates a large stress concentration at the boundary, due to the elastic field associated with the dislocation pile-up.

3.4 Discussion

3.4.1 Crack branching, subcrack formation, and crack coalescence

In the Fe-18Cr-14Ni steel, the most important characteristic of fatigue crack propagation was crack branching as shown in Fig. 3.6. The fatigue crack propagated continuously in the grain interior. Only when a crack meets grain boundaries or deformation bands, does crack branching occur along the grain boundary as shown in Fig. 3.6(e₂, g₁, g₂) and along the internal microstructure bands such as slip bands, as shown in Fig. 3.6(e₁). Accordingly, the fracture surface also showed a secondary crack (Fig. 3.7c₁) in the short crack region. Crack branching along the grain boundaries and deformation bands is a common crack propagation behavior in FCC steels with low SFE [15]. These facts resulted in smooth crack propagation behavior as shown in Fig. 3.4. The microstructure-dependent propagation behavior disappeared when the crack length was increased as shown in Fig. 3.9. Accordingly, typical fatigue striation features were observed in Fig. 3.7d₁.

In contrast, the Fe-23Mn-0.5C steel experienced subcrack formation as shown in Fig. 3.11. The number of subcracks was significantly higher than that of the Fe-18Cr-14Ni steel. Also, the formation of a considerable number of fatigue subcracks in the Fe-23Mn-0.5C steel has been reported in our previous study [16]. One reason why the subcracks form easily is the occurrence of DSA arising from the Mn-C couples. Localized strain banding associated with the DSA causes lattice defect accumulation, leading to subcrack formation. Specifically, dislocation depinning from a solute atmosphere or decomposition of Mn-C couples cause local strain softening [17-19], which increases local density of dislocation

and vacancies. An extra stress required for the dislocation depinning or decomposition of Mn-C couples would be provided by formation/movement of stress concentration sites during fatigue such as surface relief formation and main fatigue crack propagation. Relevant discussion for the subcrack formation mechanism is given in section 3.4.3. The occurrence of subcracks has both positive and negative effects on the fatigue crack growth. In terms of negative effects, subcrack coalescence with the main crack, accelerates fatigue crack growth as shown in Fig. 3.4(c) and 3.11(e₁). Accordingly, the fracture surface shown in Fig. 3.12(d₁) exhibited both striation and quasi-cleavage-like regions. In terms of positive effects, stress redistribution near a crack tip [20] and roughness-induced crack closure [21, 22] decelerates the crack growth. Specifically, stress release occurs when a subcrack is aligned parallel to the main crack [20]. The roughness-induced crack closure results from zigzag crack propagation through crack coalescence shown in Fig. 3.11 (f₂, g₁, and g₂). It is also noteworthy that ductile fatigue striations were observed even at a very small crack [Fig. 3.12(b)]. The striation formation indicates that an alternating crack blunting and re-sharpening mechanism occurs [23, 24], which enables plasticity-induced crack closure [25]. The plasticity-induced crack closure acts as a factor that decelerates fatigue crack propagation, particularly in a small-crack region. Therefore, in the following sections, we discuss the details of subcrack formation behavior and small fatigue crack growth behavior in terms of crystallography and the effects of Mn-C interaction.

In contrast, the Fe-25Cr-1N steel also exhibited subcrack formation, as shown in Fig. 3.15. The number of subcracks was significantly higher than was observed in the Fe-18Cr-14Ni steel. Subcrack formation can be caused by the enhancement of dislocation planarity by the Cr-N interaction. Specifically, the Cr-N interaction suppresses cross-slip and enhances planar slip, leading to subcrack development on the surface [26]. Subcrack formation has both positive and negative effects on the growth of fatigue cracks. Subcrack coalescence with the main crack can accelerate fatigue crack growth, as shown in Fig. 3.5(b) and Fig. 3.15(e₁) and (f₂). However, a stress distribution near the crack tip [27] and roughness-induced crack closure [28, 29] can decelerate the crack growth. Specifically, stress release occurs when a subcrack is aligned parallel to the main crack [27], decreasing the driving force for crack growth. The roughness-induced crack closure phenomenon results from zigzag crack propagation, through crack coalescence, and deflection as shown in Fig. 3.15(e₁, f₂, g₁, g₂).

3.4.2 Subcrack formation mechanism: importance of $\{111\}_\gamma$ slip plane cracking

In the Fe-18Cr-14Ni steel, as indicated by yellow lines in Fig. 3.8(c), the microstructural fatigue crack propagation path was along $\{111\}_\gamma$ planes at some points in the short crack region; however, the feature of crack propagation along $\{111\}_\gamma$ planes disappeared in the long crack region [Fig. 3.9(c)]. Since deformation twins were not observed at the vicinity of the fracture surface in the Fe-18Cr-14Ni steel at the short crack region [Figs. 3.8(e)], the crack propagation along $\{111\}_\gamma$ planes are not attributed to deformation twinning. The $\{111\}_\gamma$ slip plane cracking arises from the accumulation of dislocations on a single $\{111\}_\gamma$ plane [30, 31] that would provide damage sources such as vacancies and dislocation dipoles during the fatigue process. However, as the maximum GROD values increased with increasing crack length [Figs. 3.8(d) and 3.9(d)], the plastic zone size and maximum plastic strain at a crack tip increase with increasing crack length. This is likely to activate another $\{111\}_\gamma$ slip plane because of work hardening on the primary slip plane. Therefore, the crystallographic features of the fatigue crack propagation path are smeared out when the fatigue crack length is large, as shown in Fig. 3.9(c). Furthermore, the large plastic strain at the crack tip creates a stretching zone, causing ductile fatigue striations [23] as observed in Fig. 3.7(d₁).

In contrast, the Fe-23Mn-0.5C steel showed that the fatigue crack propagation behavior was along $\{111\}_\gamma$ planes at both short and long crack regions as shown in Figs. 3.13(c) and 3.14(c). Although deformation twins were observed in the long crack region shown in Fig. 3.12(e), the crack propagation path was not along the twin plates. This fact indicates that the $\{111\}_\gamma$ fatigue cracking in the Fe-23Mn-0.5C steel is not directly correlated with the twin formation. The occurrence of $\{111\}_\gamma$ fatigue cracking even in the long crack region indicates the underlying difference of fatigue crack propagation behavior between the Fe-18Cr-14Ni and Fe-23Mn-0.5C steels. The fatigue crack propagation along the $\{111\}_\gamma$ planes at both short and long crack regions is attributed to the ease of fatigue crack initiation associated with DSA-induced slip localization. In fact, in a previous study [16], fatigue cracking was observed to be initiated easily in the Fe-23Mn-0.5C steel when the stress amplitude was high, because of local softening stemming from the decomposition of Mn-C couples. Similar to the effect of high stress amplitude, a large amount of plastic strain at a long crack tip can cause plastic strain localization, which assists fatigue crack

initiation in the vicinity of a crack tip or mode II fatigue crack growth. Through mode II fatigue crack growth or coalescence between the initiated crack and the main crack, the crack propagates along $\{111\}_\gamma$ even for a long crack length. In this context, a fatigue crack propagates when the plastic strain at the crack tip achieves a critical strain for the fatigue crack initiation. Namely, the plastic strain around the crack surface does not increase over the critical strain, which is a reason of no remarkable increase in crack tip strain with increasing crack length. Accordingly, the maximum GROD value did not increase with crack length as indicated in Figs. 3.13(d) and 3.14(d).

In contrast, the Fe-25Cr-1N steel exhibited fatigue crack propagation along the $\{111\}_\gamma$ planes in both the short and long crack regions, as shown in Fig. 3.17(a₃) and (b₃). The occurrence of crack propagation along the $\{111\}_\gamma$ planes in both the long and short crack regions is the major difference between the fatigue crack propagation behavior of the Fe-18Cr-14Ni and Fe-25Cr-1N steels. This behavior can mainly be attributed to the enhanced dislocation planarity associated with the Cr-N interaction, which leads to subcrack formation. Figure 3.18 shows the dislocation planarity at both the short and long cracks; at the short crack (Fig. 3.18(a)) the dislocations have a simple planar array, but at the long crack (Fig. 3.18(b)) multiple glide systems are activated. Note that the slip character remains planar on each slip plane, resulting in the formation of subcracks (even at the long crack) as shown in Fig. 3.15(g₁) and (g₂). This is due to the dislocation pile-up at the grain boundary (Fig. 3.19), and as a result the fracture surface exhibits intergranular fracture at the long crack (Fig. 3.17(b₁)). This result is completely different to the behavior observed in the Fe-18Cr-14Ni steel. As seen in Fig. 3.17(a₄), the degree of strain evolution/localization in the Fe-25Cr-1N steel was small compared to the Fe-18Cr-14Ni steel. This can be explained by two factors. The first relates to the subcrack formation and subsequent crack coalescence. When the crack growth occurs via crack coalescence, the plastic strain around the crack surface corresponds to a critical strain associated with the crack initiation, which must be lower than the plastic strain evolution arising from the crack opening for growth resulting in a significant crack length to occur. Second, the enhanced dislocation planar array in the vicinity of the crack surface facilitates mode II fatigue crack growth; this refers to the crack propagation path occurring along the $\{111\}_\gamma$ planes, even in the long crack region. The dislocation planar glide-driven crack growth causes highly localized dislocation accumulation along the slip plane. Therefore, when a crack propagates along the $\{111\}_\gamma$ slip plane, the plastic zone size around the crack surface must be small. Furthermore, as shown in Fig. 3.17(b₄), the plastic strain around the crack surface did not

increase with crack length. This can also be explained by the reasons mentioned above. In terms of crack coalescence, the strain level is predominantly controlled by the critical strain required for the crack initiation. We can consider the mode II crack growth stemming from the dislocation planarity, which was maintained even at the long crack. From this viewpoint, the plastic strain level would increase with crack length, for the most part. However, compared with the Fe-18Cr-14Ni steel (which exhibited mode I crack growth in the long crack region), the dependence of the crack length on the plastic zone size and detectable strain level must be lower. This is due to the residual dislocation planar array observed in Fig. 3.18(b₁), which allowed mode II crack growth to occur even in the long crack region.

3.4.3 Role of Mn-C and Cr-N interaction on fatigue crack growth: its crack length dependence

The $\{111\}_\gamma$ slip plane cracking requires two conditions: (1) dislocation accumulation on a primary slip plane and (2) suppression of activation of the secondary slip plane. First note that the DSA of Fe-Mn-C austenitic steels has been reported to occur through Mn-C coupling [32-34]. When a crack is short, work hardening occurs on the primary slip plane at the crack tip through the formation of Mn-C couples, which pins dislocations emitted from the crack tip. For instance, as a plausible mechanism of the dislocation pinning, Lee et al. reported [34] that motion of a leading partial dislocation mandatorily changes a position of interstitial carbon from an octahedron site to a tetrahedron site in the austenitic phase of Fe-Mn-C steels. This crystallographic effect of the partial dislocation motion on carbon position has also been pointed out in a previous work [35]. The carbon atoms located at the tetrahedron sites in the stacking fault part of the extended dislocation easily move to one of the octahedron sites that lie adjacent to Mn atoms having an attractive force before the pertaining trailing partial sweeps the same position where the carbon atoms are located. The preferential movement of carbon atoms to the octahedral sites lying adjacent Mn atoms increases Mn-C couple density in the stacking fault part, which pins extended dislocations strongly. This suppresses dislocation accumulation on the primary slip plane due to a reduction in dislocation mobility. In addition, this effect activates dislocation motion on a secondary slip plane to accommodate stress concentration at the crack tip, which contributes to crack blunting and re-sharpening accompanied by ductile fatigue striation. The fracture surface shown in Fig. 3.10(b) demonstrates corresponding shallow striation-like features even in the vicinity of the drill hole. However, dislocations emitted from the crack tip on the secondary slip plane are also pinned by the Mn-C couples, similar to the

case of dislocations on the primary slip plane. Therefore, when the crack becomes long, further plastic shear stress and subsequent deformation are applied on the primary slip plane, which is required to accommodate a high stress arising from the crack opening. The extra deformation causes decomposition of the Mn-C couples existing on the primary slip plane [19]. The decoupling of attractively interacting atoms softens the shear strength on the primary slip plane, localizing dislocation motion [36-38]. In other words, dislocations preferentially accumulate on the primary slip plane, causing $\{111\}_\gamma$ slip plane cracking even in the long crack region. Hence, it can be concluded that the Mn-C couples play ambivalent roles, depending on fatigue crack length. Because of the combined effect of this crack length dependence and stress amplitude, the Fe-23Mn-0.5C steel showed greater normalized fatigue limit and fatigue lifetimes at low normalized stress amplitudes, and shorter fatigue lifetimes at high normalized stress amplitudes than those of the Fe-18Cr-14Ni steel as shown in Fig. 3.2(b).

Moreover, Fe-Mn-C TWIP steel was recently found to show fatigue crack non-propagation at a fatigue limit, even though a TWIP steel without carbon did not [16]. The role of the Mn-C coupling that activates secondary slip planes may play an important role in the fatigue crack non-propagation. Plasticity-induced crack closure requires local volumetric expansion at a crack tip along the loading direction to evolve residual compressive stress. As long as dislocation slip deformation is considered, atomistic displacements are given only by shear, which indicates that normal strain evolution along the loading direction is a result of shear straining on two or more slip systems. Hence, the DSA-enhanced activation of dual slip planes assists to provide normal plastic strain evolution along the loading direction at a crack tip, which brings about compressive residual stress at the crack tip, i.e. plasticity-induced crack closure. One effect of the closure can be to inhibit or stop fatigue crack propagation. Therefore, the DSA assists fatigue crack non-propagation by not only generating hardening at a crack tip, but also by activation of a secondary slip plane. The non-propagation of the main crack and its subcracks enhances the fatigue limit and fatigue lifetimes at low stress amplitudes. This viewpoint is believed to be crucial in order to understand fatigue crack non-propagation mechanism in Fe-Mn-C austenitic steels.

The austenite in both the Fe-25Cr-1N and Fe-18Cr-14Ni steels was stable, and possessed characteristics typical of low stacking fault energy materials, such as twin and extended dislocations. However, a significant difference between the two steels was found in the nitrogen-enhanced dislocation planarity. The dislocation planarity tends to depend

on the stacking fault energy and the short-range ordering of solute atoms. However, the enhanced dislocation planarity observed in the Fe-25Cr-1N steel is an exceptional result, produced by the short-range ordering associated with the Cr-N interaction. This is hypothesized to play a major role in the fatigue crack non-propagation behavior, in a similar manner to that discussed in relation to crack growth resistance in section 4.4.2. Specifically, the enhanced planar dislocation glide causes crack branching, deflection, and subcrack formation, facilitating the roughening of the crack surface. This effect enhances the roughness-induced crack closure, which can improve the crack non-propagation limit. In addition, the high dislocation pile-up stress stemming from the suppression of cross-slip prevents dislocation emission from the crack tip, which may also increase the limit of crack non-propagation.

3.5 Section Conclusions

A comparative study on fatigue crack growth in Fe-25Cr-1N, Fe-23Mn-0.5C and Fe-18Cr-14Ni steels was conducted to elucidate the effects of Mn-C and Cr-N i-s interaction on fatigue resistance. From the results, the following conclusions can be made:

- (1) The Mn-C and Cr-N i-s interaction showed a positive effect in enhancing the fatigue limit of the Fe-23Mn-0.5C and Fe-25Cr-1N steels in comparison with the Fe-18Cr-14Ni steel without any i-s interaction.
- (2) The Mn-C interaction in the Fe-23Mn-0.5C steel showed following effects on crack initiation and propagation
 - Deformation induced decomposition of Mn-C couples results in local softening, causing strain localization that assists crack initiation along $\{111\}\gamma$ slip planes.
 - Due to ease of fatigue crack initiation along the slip planes, showed subcrack formation, crack coalescence, and zig-zag crack propagation behavior. These factors enhance the roughness induced crack closure, a positive effect on fatigue crack growth.
 - Formation of subcracks works for stress release at the crack tip: another positive effect.
- (3) The Cr-N interaction in the Fe-25Cr-1N steel showed following effects on fatigue crack initiation and propagation
 - The short crack region exhibited a planar glide dislocation pattern, but multiple slip systems were activated in the long crack system.

- As the dislocation pattern remained planar on each slip plane, the crack propagated along $\{111\}\gamma$ slip plane in the long crack region.
- The dislocation pile-up at the grain boundaries caused grain boundary subcracks, which offers stress release at the crack tip.

3.6 References

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3.7 Figures

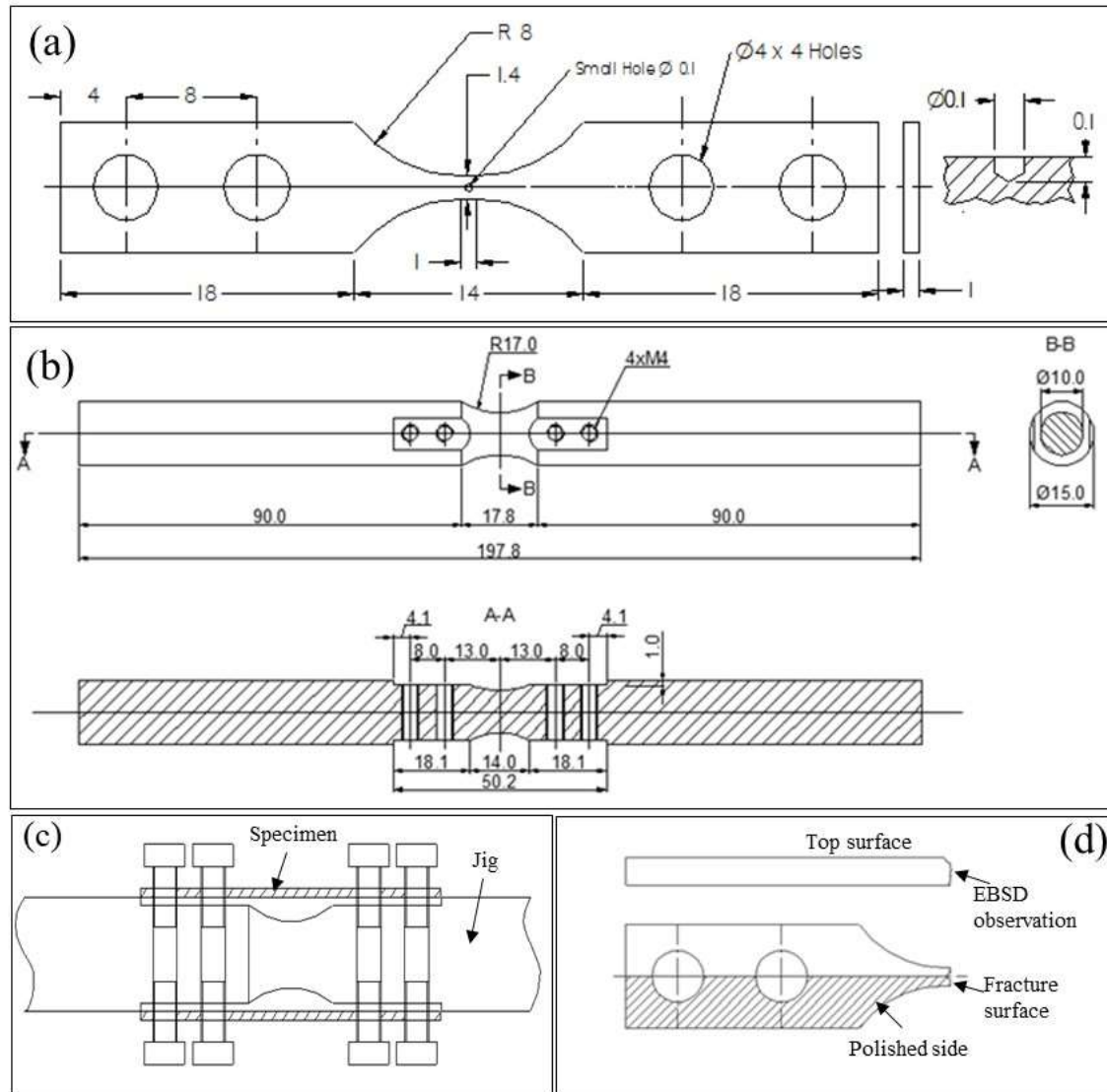


Fig. 3.1. Schematics of (a) a fatigue test specimen, (b) a jig for rotating bending fatigue testing, and (c) a configuration of the specimen fixed on the Jig. (d) A region of EBSD observations. The hatched region was removed by mechanical polishing. The unit of length is mm.

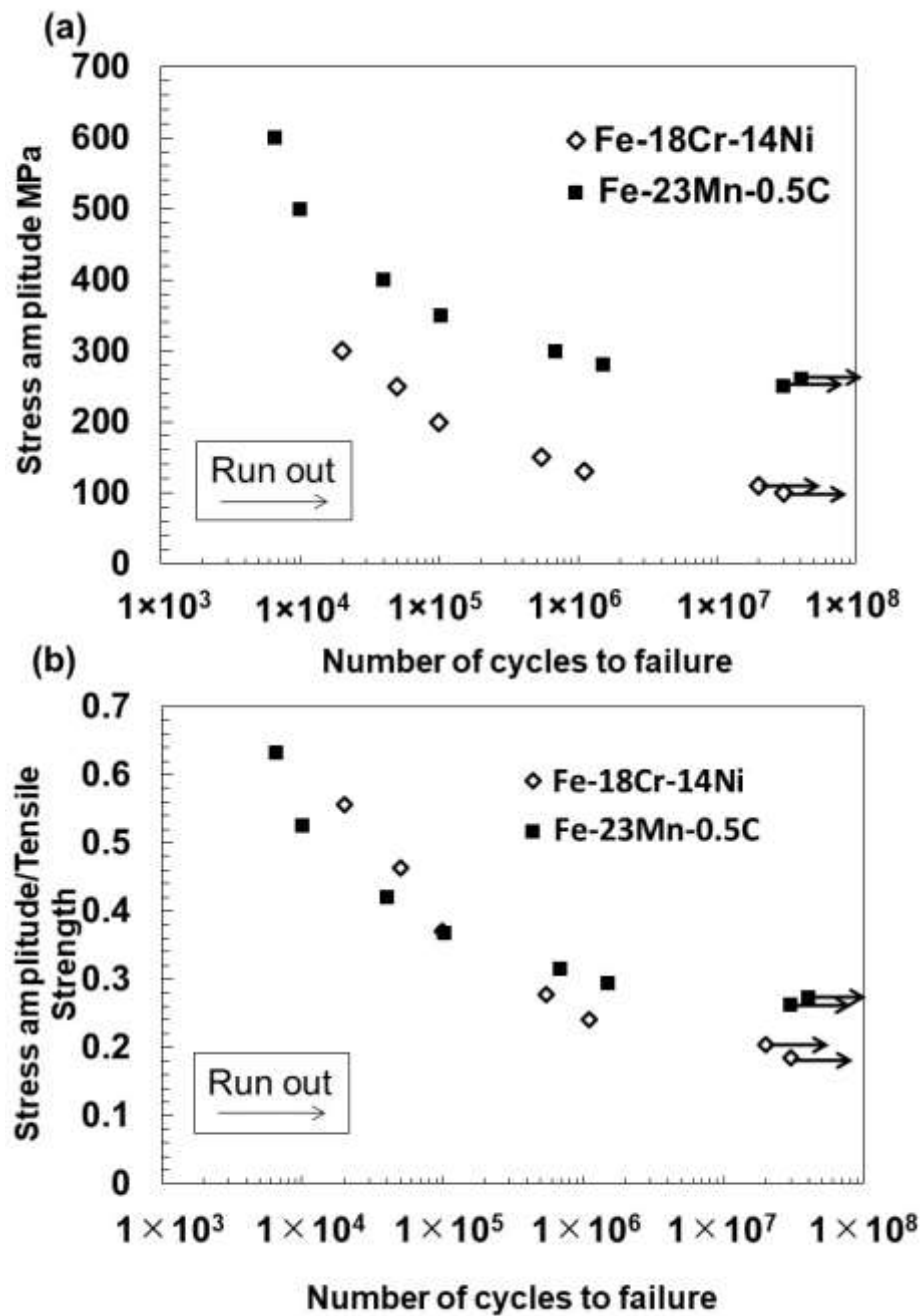


Fig. 3.2. (a) Stress amplitude versus fatigue lifetime curves of the Fe-23Mn-0.5C and Fe-18Cr-14Ni steels. (b) Number of cycles to failure plotted against stress amplitude normalized by tensile strength.

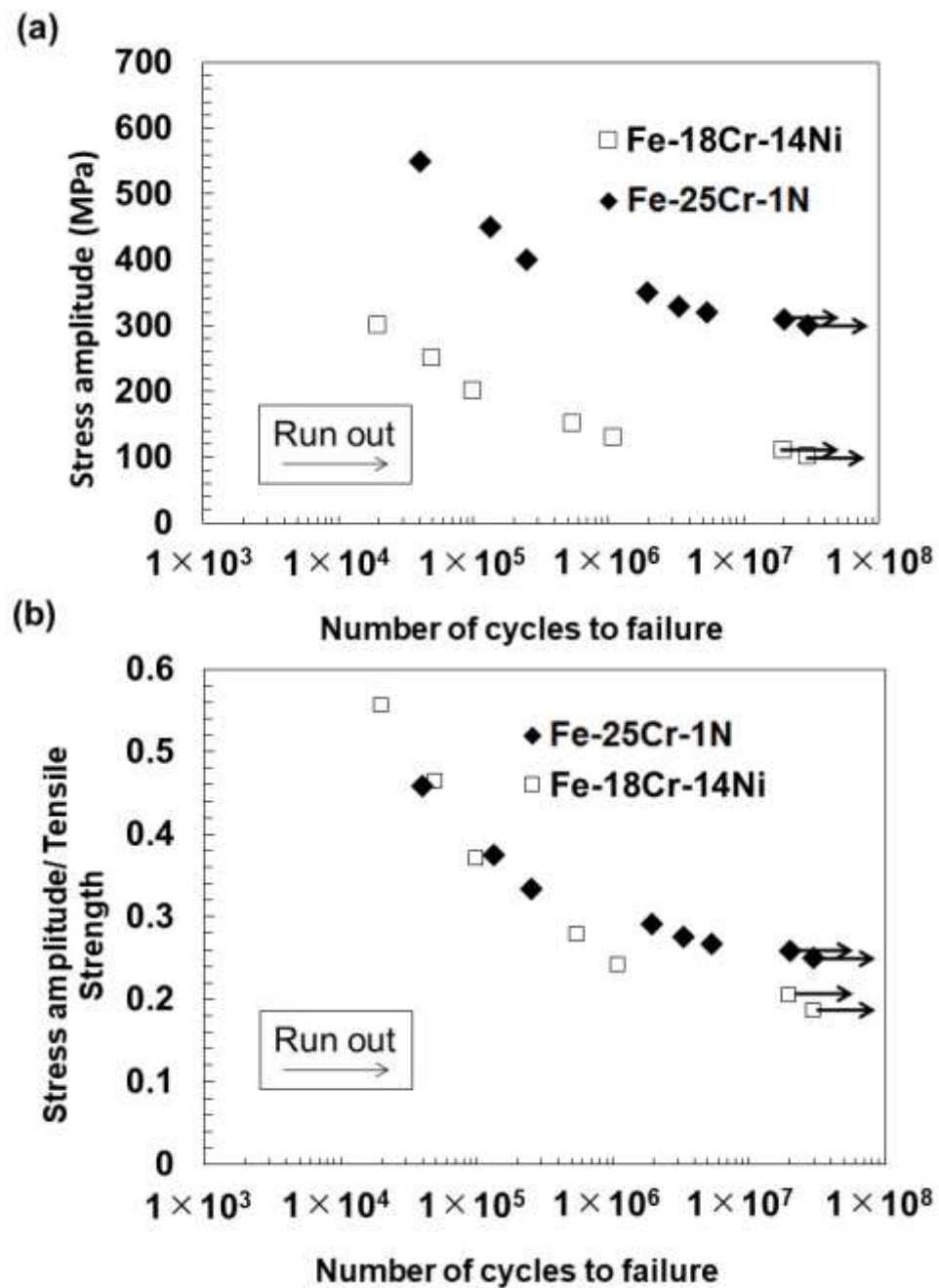


Fig. 3.3 (a) Stress amplitude versus fatigue life for the Fe-25Cr-1N and Fe-18Cr-14Ni steels. (b) Number of cycles to failure plotted against stress amplitude normalized by the tensile strength.

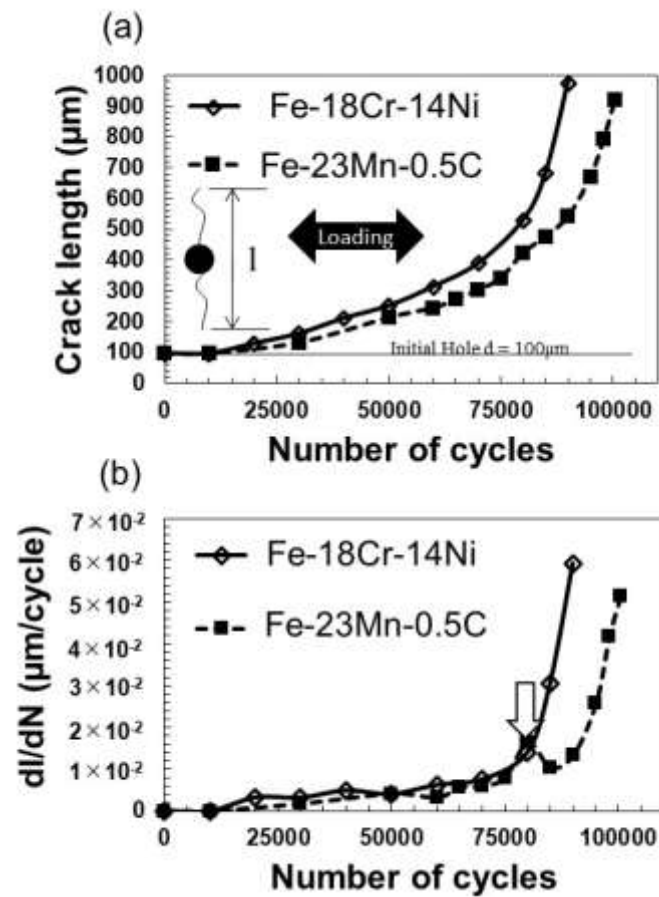


Fig. 3.4. Fatigue crack growth rate curves of the two steels at the same ratio between stress amplitude and tensile strength. (a) Crack length versus number of cycles. (b) Fatigue crack growth rate versus number of cycles. Stress amplitude of the Fe-23Mn-0.5C and Fe-18Cr-14Ni steels are 350 and 200 MPa, respectively.

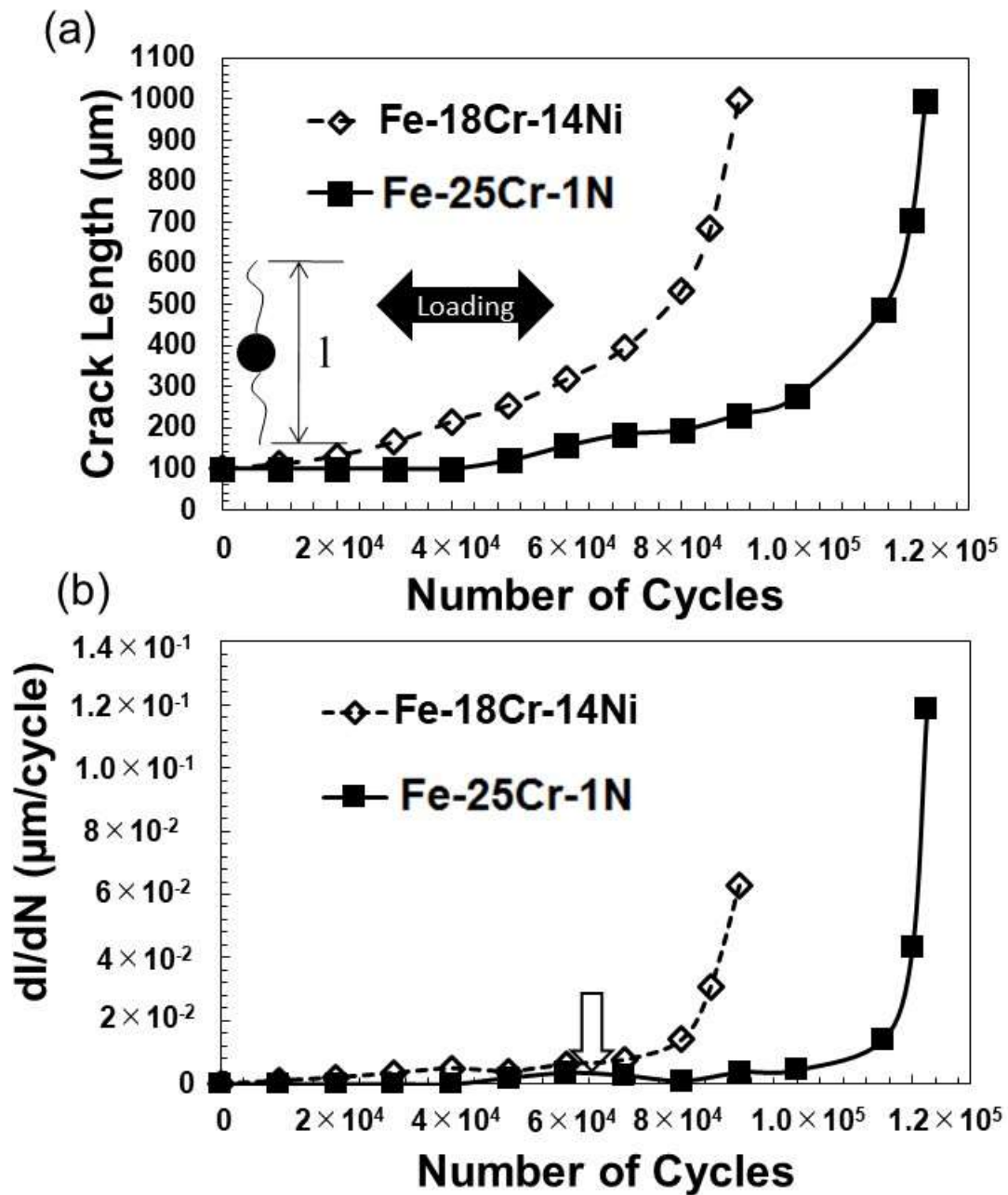


Fig. 3.5 Fatigue crack growth rate curves for the two steels at the same stress ratio of 0.36: (a) crack length versus number of cycles, (b) crack growth versus number of cycles.

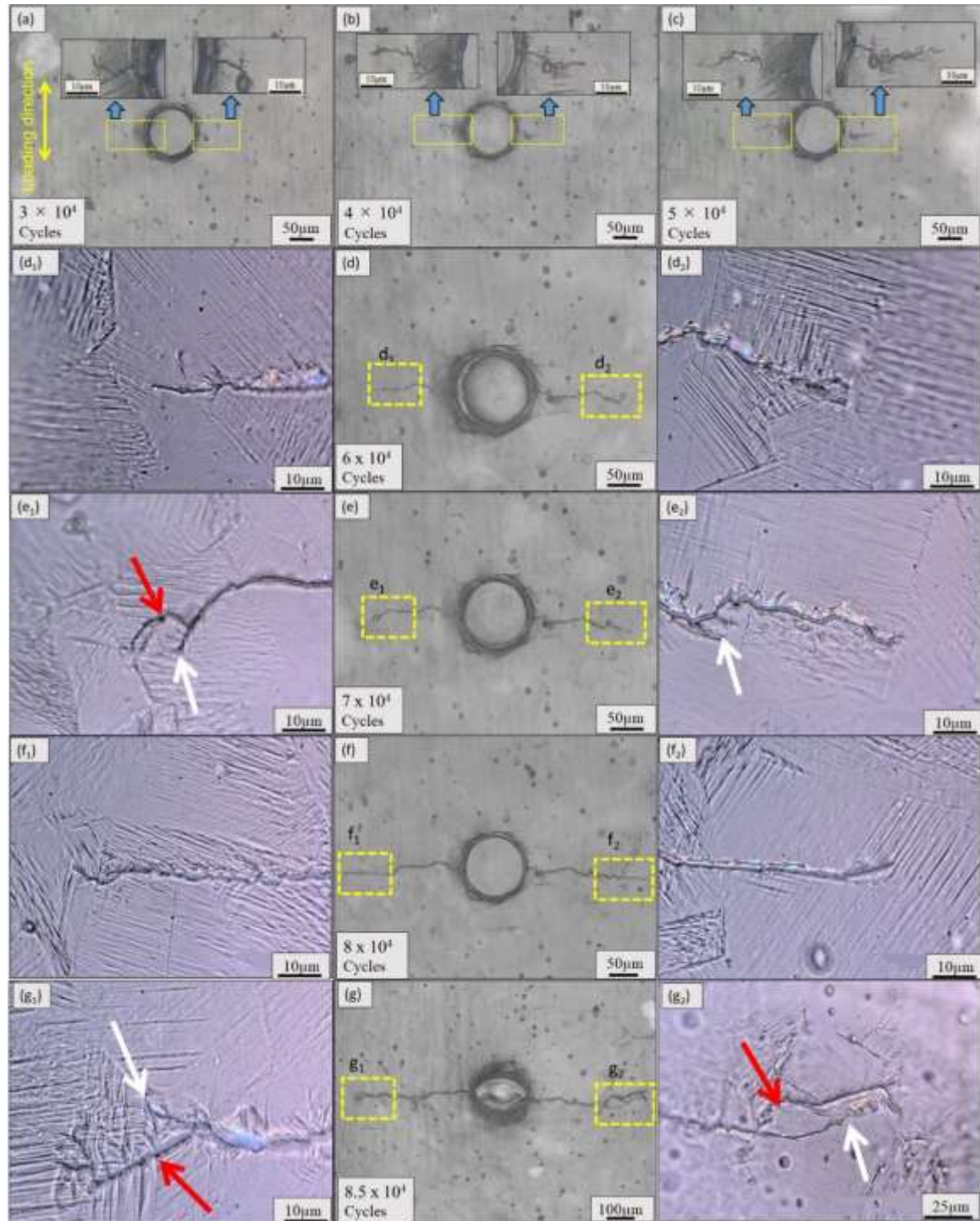


Fig. 3.6. Optical micrographs of the Fe-18Cr-14Ni steel during the fatigue test at a stress amplitude of 200 MPa: (a) 3×10^4 , (b) 4×10^4 , (c) 5×10^4 , (d) 6×10^4 , (e) 7×10^4 , (f) 8×10^4 , and (g) 8.5×10^4 cycles. The red and white arrows indicate main and sub cracks, respectively.

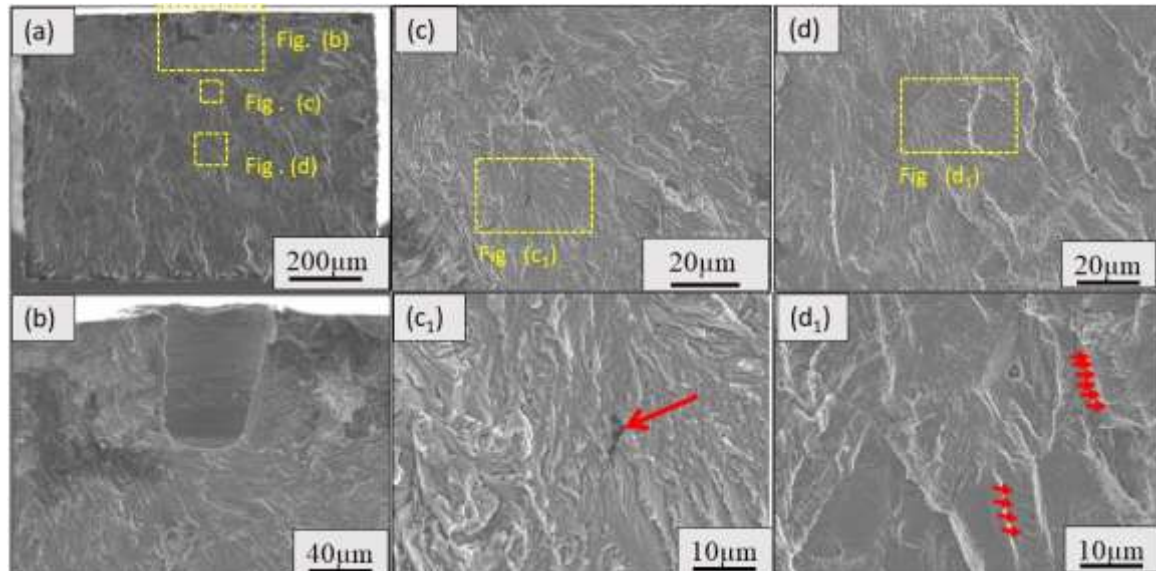


Fig. 3.7. SEM images of the fatigue fracture surfaces of the Fe-18Cr-14Ni steel tested at 200 MPa. (a) Overview of the fracture surface. (b) A region where crack was initiated from the drill hole. (c) Early, and (d) intermediate propagation regions outlined by the dashed lines in Fig. (a). The red arrow in Fig. (c₁) indicates a secondary crack. The small red arrows in Fig. (d₁) indicate ductile fatigue striations.

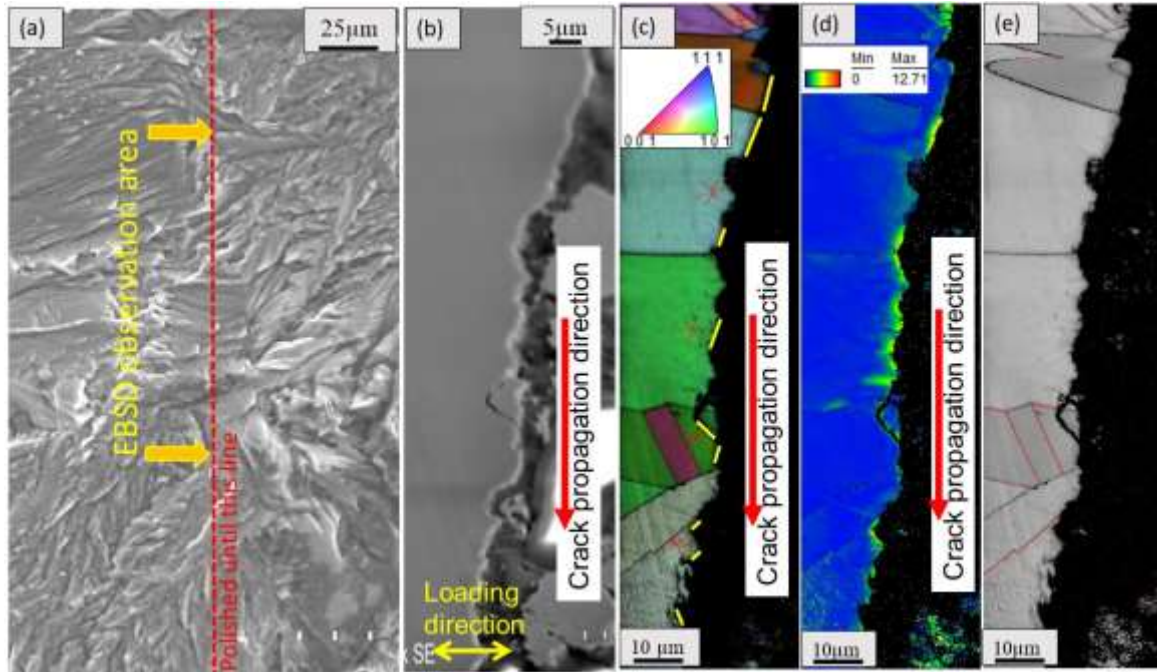


Fig. 3.8. A set of cross-sectional EBSD analysis results of a short crack region 100 μm from the bottom of the drill hole in the Fe-18Cr-14Ni steel tested at 200 MPa: (a) Fracture surface, (b) SEM image, (c) image quality + RD-IPF map, and (d) grain reference orientation deviation angle (GROD) map, (e) Image quality map with identification of $\Sigma 3$ twin boundaries. The yellow lines in (c) indicate regions where the crack surface is along one of $\{111\}_{\gamma}$ planes. The red lines in (e) indicate $\Sigma 3$ twin boundaries.

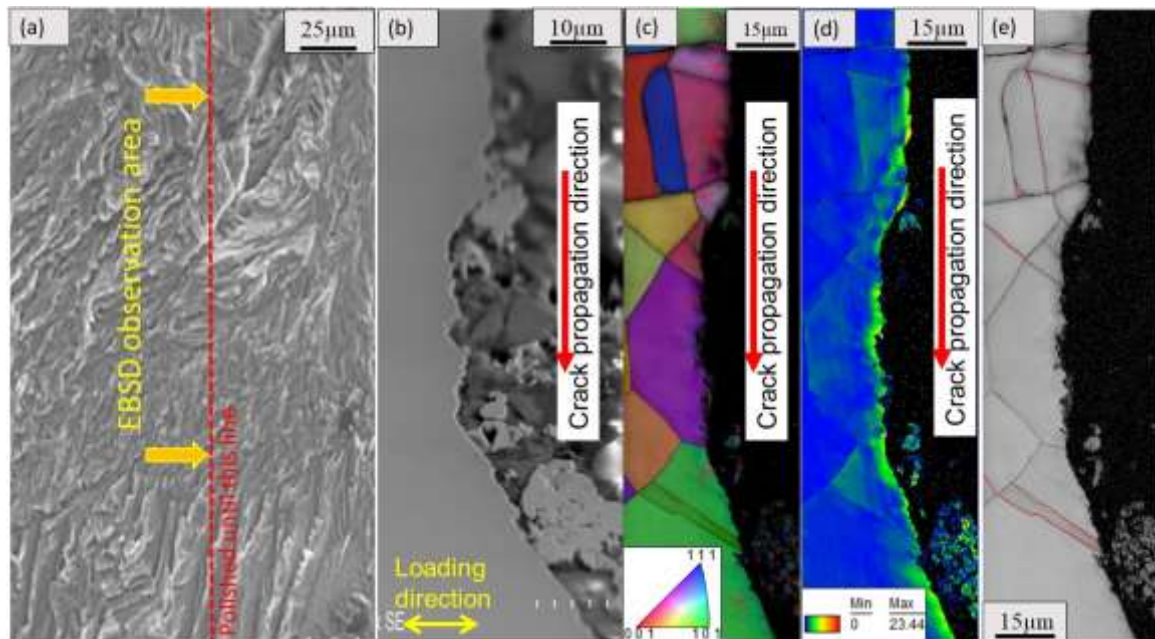


Fig. 3.9. A set of cross-sectional EBSD analysis results of a long crack region 400 μm from the bottom of the drill hole in the Fe-18Cr-14Ni steel tested at 200 MPa: (a) Fracture surface, (b) SEM image, (c) image quality + RD-IPF map, and (d) GROD map. (e) Image quality map with identification of $\Sigma 3$ twin boundaries. The red lines in (e) indicate $\Sigma 3$ twin boundaries.

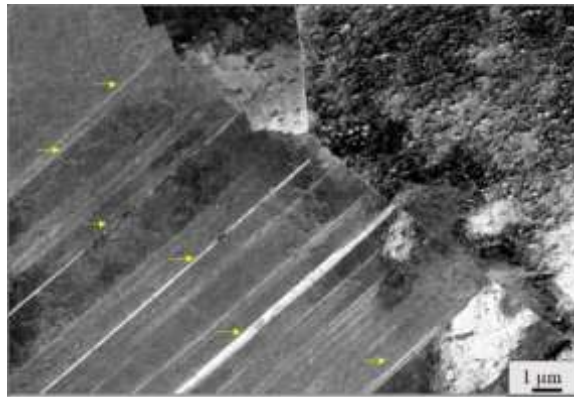


Fig. 3.10. An ECC image in the Fe-18Cr-14Ni steel at 500 μm from the bottom of the drill hole tested at 200 MPa. The yellow lines indicate twin plates.

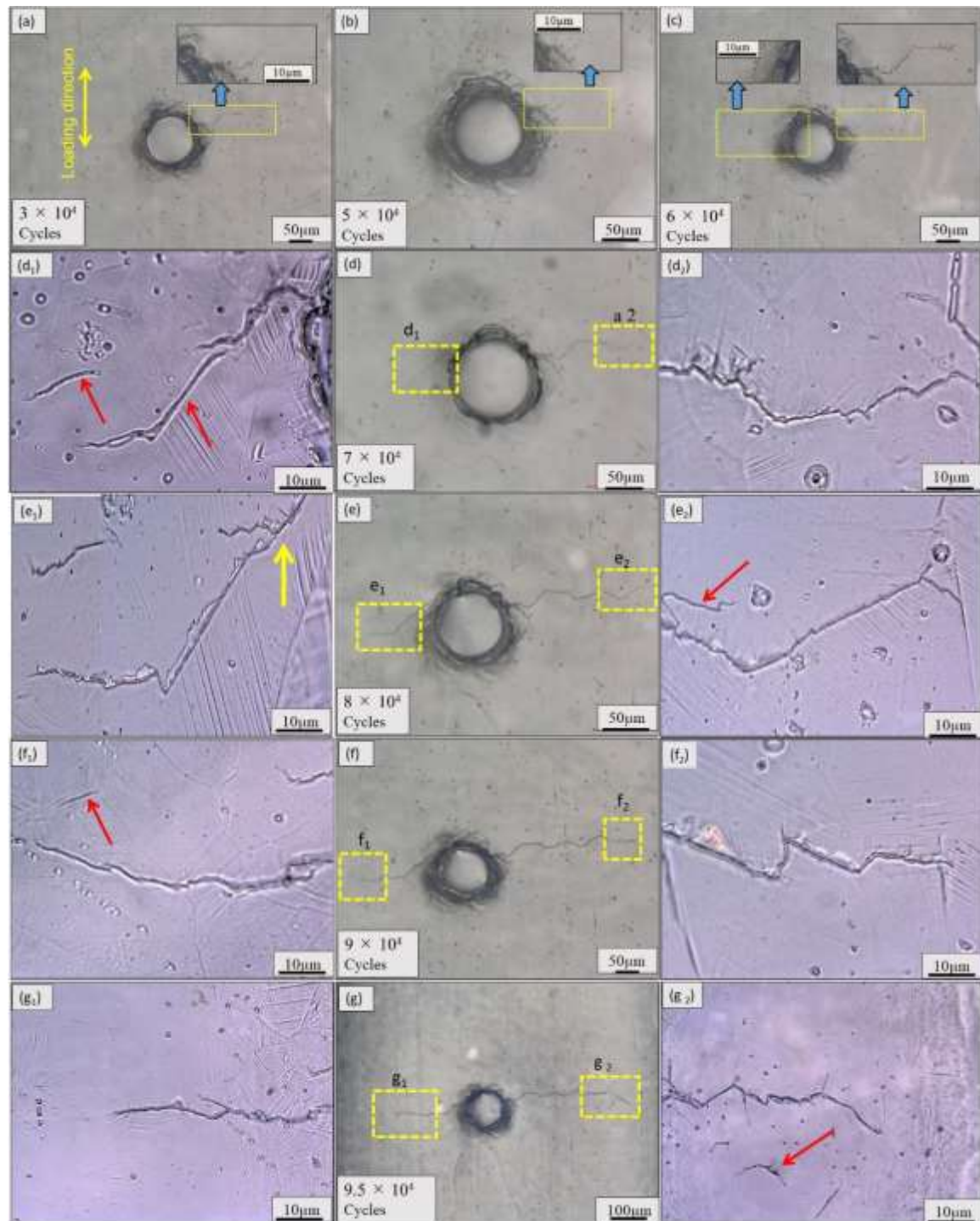


Fig. 3.11. Optical micrographs of the Fe-23Mn-0.5C steel during the fatigue test at a stress amplitude of 350 MPa: (a) 3.0×10^4 , (b) 5.0×10^4 , (c) 6.0×10^4 , (d) 7.0×10^4 , (e) 8.0×10^4 , (f) 9.0×10^4 , and (g) 9.5×10^4 cycles. The red arrows indicate subcracks and the yellow arrow indicates a point of crack coalescence.

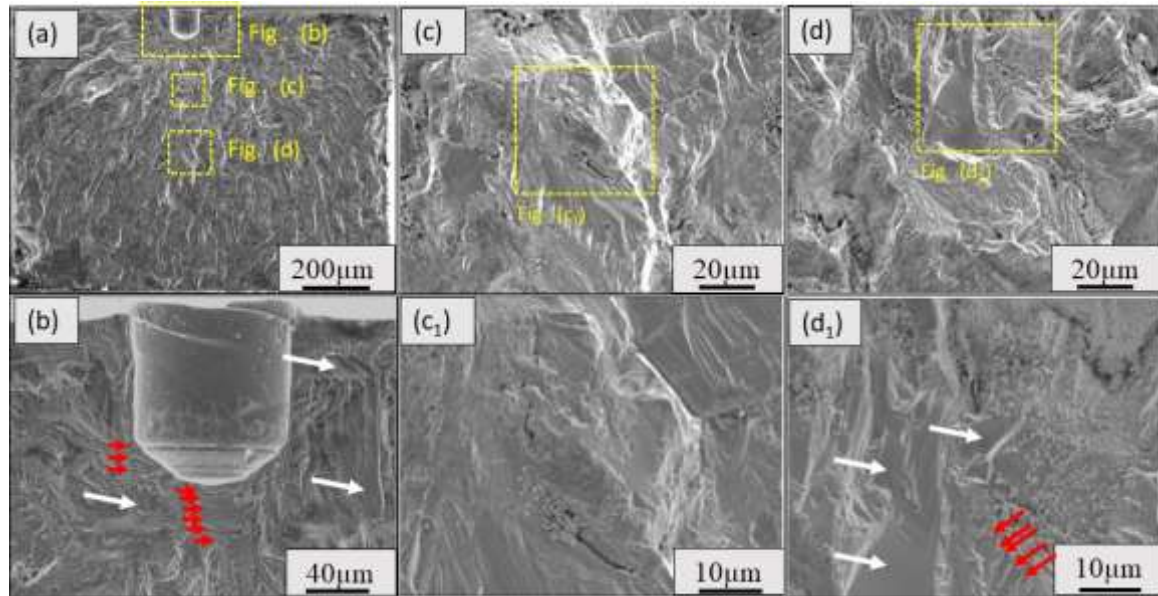


Fig. 3.12. SEM images of the fatigue fracture surfaces of the Fe-23Mn-0.5C steel tested at 350 MPa: (a) an overview of the fracture surface, (b) a region where a crack was initiated from the drill hole, (c) early, and (d) intermediate propagation regions outlined by the dashed lines in Fig. (a). The red arrows indicate striation feature, and the white arrows indicate flat regions.

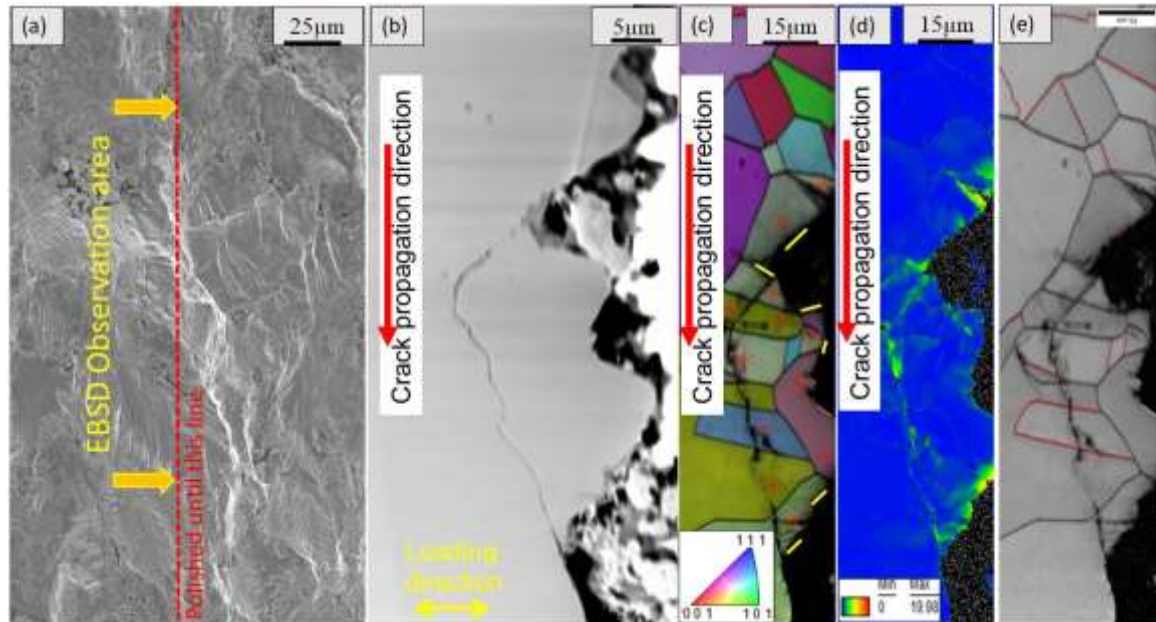


Fig. 3.13. A set of cross-sectional EBSD analysis results of a short crack region 100 μm from the bottom of the drill hole in the Fe-23Mn-0.5C steel tested at 350 MPa: (a) fracture surface, (b) cross sectional SEM image. (c) image quality + RD-IPF map, and (d) GROD map, (e) Image quality map with identification of $\Sigma 3$ twin boundaries. The yellow lines in (c) indicate regions where the crack surface is along one of $\{111\}_{\gamma}$ planes. The red lines in (e) indicate $\Sigma 3$ twin boundaries.

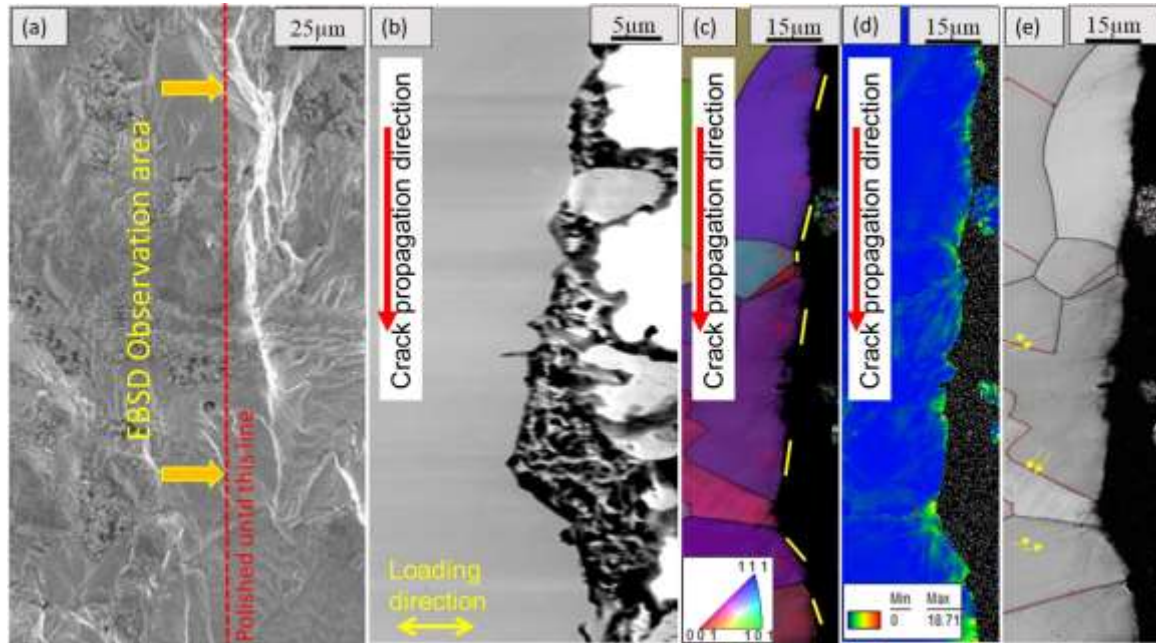


Fig. 3.14. A set of cross-sectional EBSD analysis results of a long crack region 400 μm from the bottom of the drill hole in the Fe-23Mn-0.5C steel tested at 350 MPa: (a) fracture surface, (b) cross sectional SEM image. (c) image quality + RD-IPF map, and (d) GROD map, (e) Image quality map with identification of $\Sigma 3$ twin boundaries. The yellow lines in (c) indicate regions where the crack surface is along one of $\{111\}_{\gamma}$ planes. The red lines and yellow arrows in (e) indicate $\Sigma 3$ twin boundaries and twin plates, respectively.

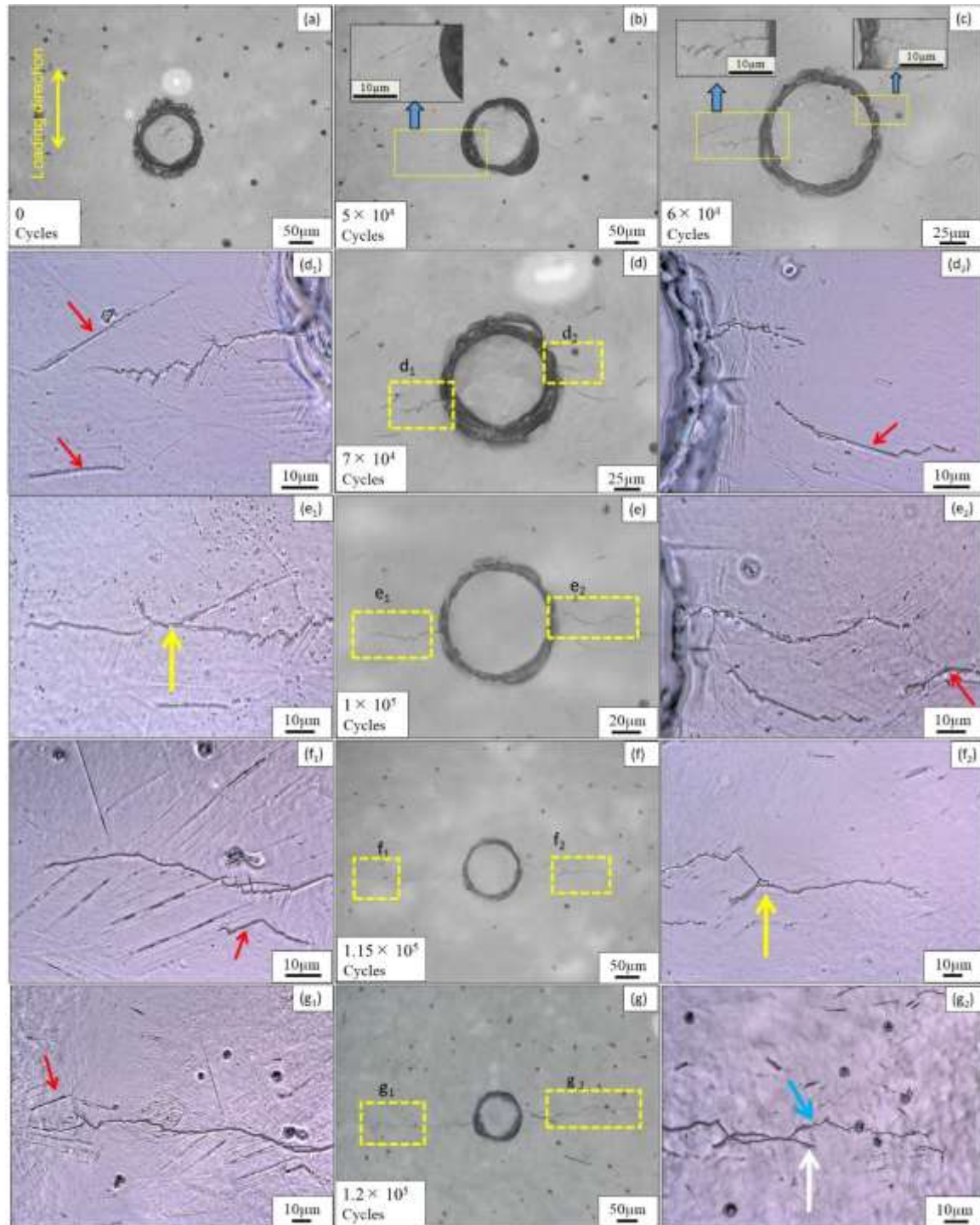


Fig. 3.15 Optical micrographs of the Fe-25Cr-1N steel replicas taken during fatigue testing at 450 MPa: (a) 0, (b) 5×10^4 , (c) 6×10^4 , (d) 7×10^4 , (e) 1×10^5 , (f) 1.15×10^5 , and (g) 1.2×10^5 cycles. The red arrows indicate subcracks and the yellow arrow indicates a point of crack coalescence. The blue and white arrows in image (g₂) indicate the main crack and subcracks, respectively.

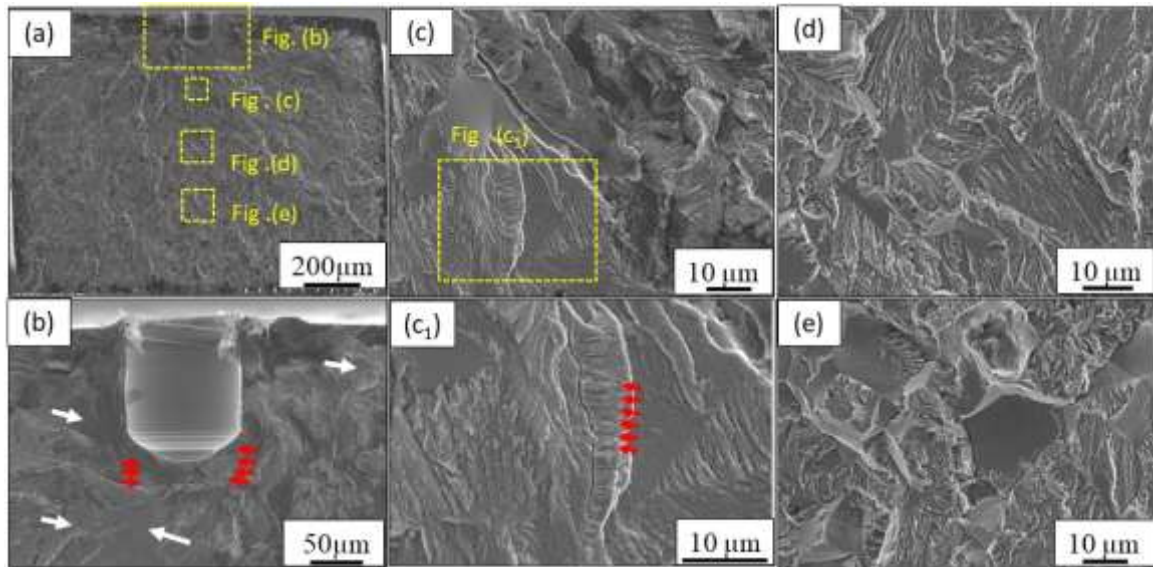


Fig. 3.16 Scanning electron microscopy (SEM) images of the fatigue fracture surface for the Fe-25Cr-1N steel tested at 450 MPa: (a) overview of the fracture surface, (b) site of the crack initiation emanating from the drill hole, (c) early, (d) intermediate, and (e) late propagation regions as indicated by dashed lines in (a). Striation features are indicated by red arrows, and flat regions by white arrows.

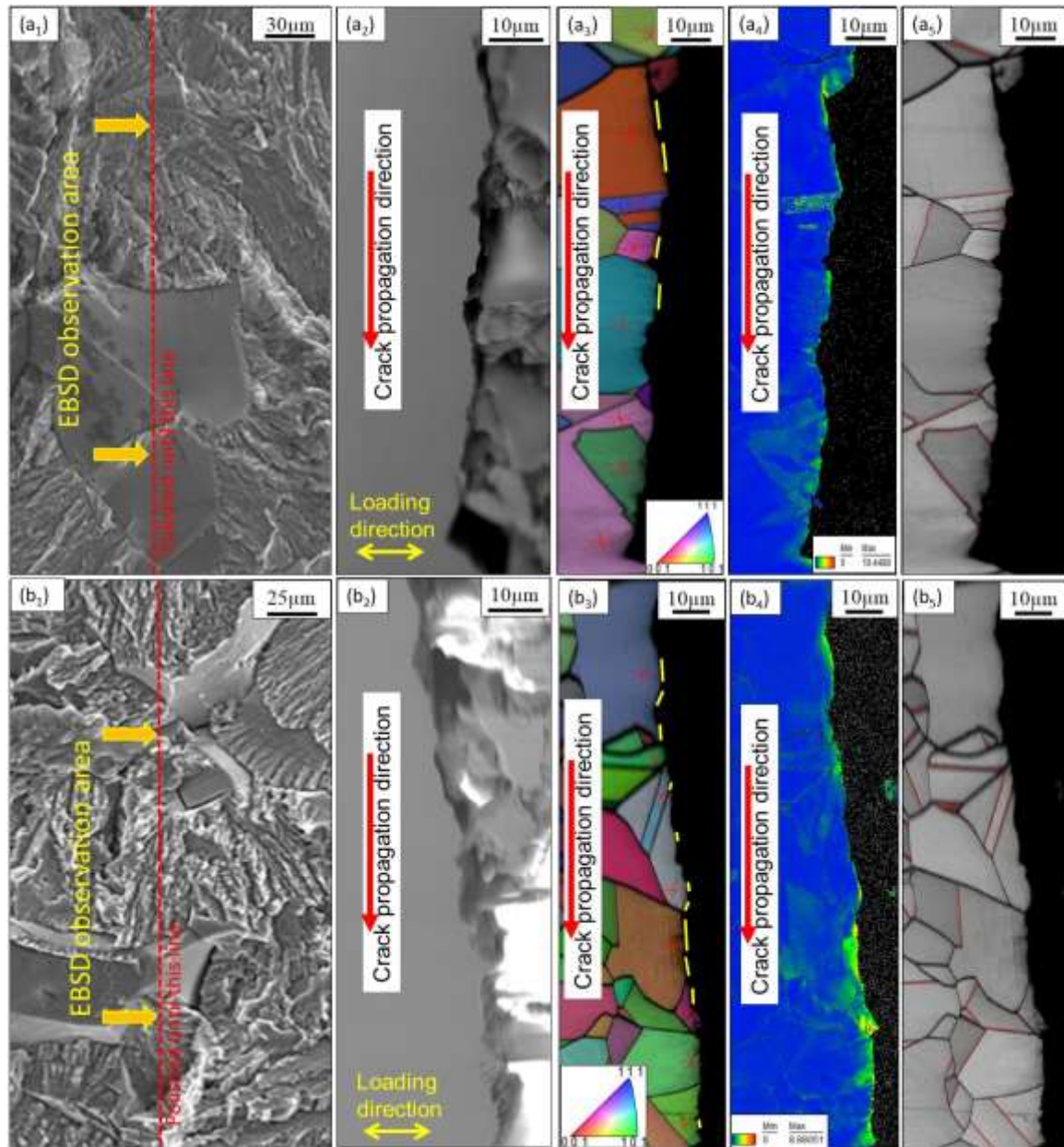


Fig. 3.17 Electron backscatter diffraction (EBSD) images of the Fe-25Cr-1N steel. Analysis at the short crack (100 μm from the bottom of drill hole) is indicated by the label (a), and at the long crack (400 μm from the bottom of drill hole) by the label (b): (a₁ and b₁) fracture surface, (a₂ and b₂) scanning electron microscopy (SEM) image, (a₃ and b₃) image quality maps in the rolling direction occurring parallel to the loading direction (IQ + RD IPF), (a₄ and b₄) grain reference orientation deviation angle (GROD) maps, and (a₅ and b₅) IQ map with identification of the $\Sigma 3$ twin boundaries. The yellow lines in (a₃) indicate regions where the crack surface follows one of the $\{111\}_{\gamma}$ planes. The red lines in (e) indicate the $\Sigma 3$ twin boundaries

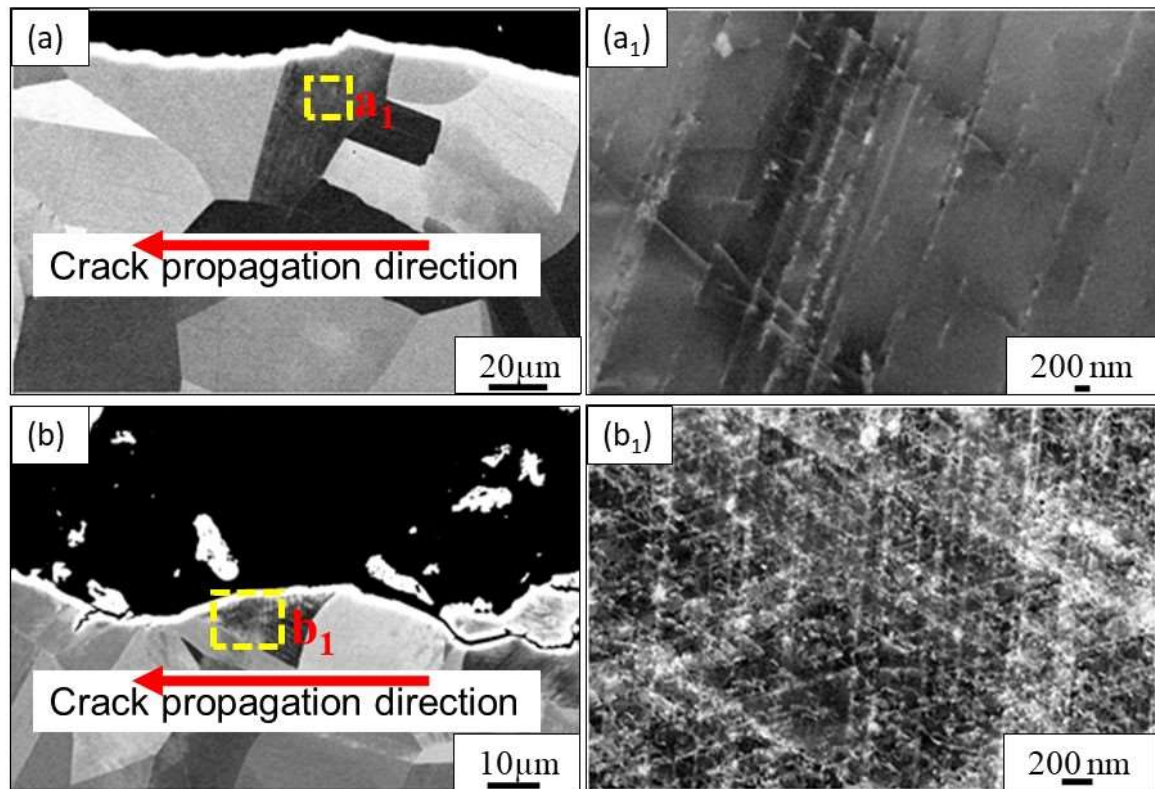


Fig. 3.18 Electron channeling contrast (ECC) images of the Fe-25Cr-1N steel, showing dislocation structure at: (a) the short crack (100 μm from the bottom of the drill hole) and (b) the long crack (400 μm from the bottom of the drill hole). Images (a₁) and (b₁) show magnifications of the short and long cracks.

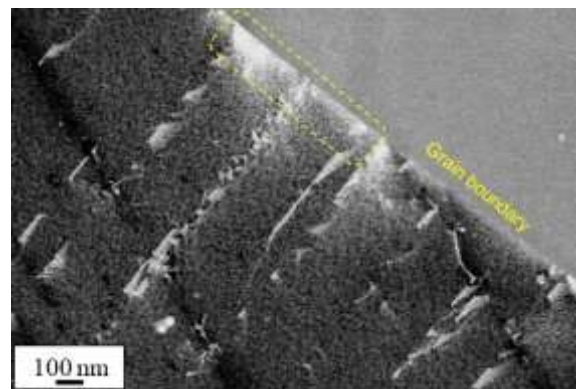


Fig. 3.19 An electron channeling contrast (ECC) image of a grain boundary in the Fe-25Cr-1N fractured specimen. The outlined region shows the elastic contrast associated with the dislocation pile-up stress concentration.

Chapter 4

Understanding the effects of Mn-C and Cr-N couples on crack growth resistance

4.1 Introduction

In Chapter 3, we reported that Mn-C and Cr-N interactions showed a positive effect on the crack growth behavior. However, the influence of the crack resistance associated with Mn-C and Cr-N couples on crack propagation rate was mainly discussed in the context of crack morphologies without detailed characterization of crack tip microstructures, and exhibited roughness-induced crack closure. We expect that the I-S interactions positively affect crack tip microstructure, which trigger fatigue crack non-propagation. Specifically, in the Fe-23Mn-0.5C steel, the fatigue limit would be enhanced by dislocation accumulation stemming from the dislocation pinning effect. In contrast, the dislocation pile-up stress of the dislocation planar array may suppress crack opening in Fe-25Cr-1N steel at the fatigue limit.

The occurrence of the dislocation motion at a fatigue crack tip plays a crucial role on the crack growth. Specifically, the motion of a crack tip dislocation controls the work hardening in the vicinity of the crack tip [1], formation of compressive residual stress at the crack wake [2], and plastic deformation of the crack tip [3]. However, the nature of the effects of the crack tip dislocation on the crack resistance has not been clarified yet. This is due to the fact that the microstructure observation at a crack tip indeed requires crack-length-specific, microstructurally local, stress-concentrated, and dislocation-resolved characterization techniques.

A strong candidate for this purpose is electron channeling contrast imaging (ECCI). ECCI enables the observation of large region [8, 9], identical [10, 11], and dislocation-resolved [12-14] microstructures in a bulk specimen. In addition, the availability of the bulk specimen is expected to have a direct correlation with the replica method that has been used for fatigue crack growth observations. Hence in this chapter, we describe the potential of ECCI for the inspection of crack tip dislocations of a non-propagating fatigue crack and derive its effect on the crack closure and ΔK_{th} for the three types of austenitic steels.

4.2 Experimental Procedure

4.2.1 Fatigue testing and characterization

The materials used in this study were the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N (mass%) steels same as employed in Chapter 3. The fatigue test specimens were cut by electric discharge machining, as shown in Figure 4.1(a), and the damage layer was removed by mechanical and electrochemical polishing. In order to evaluate the fatigue crack non-propagation limit and its reproducibility, two types of artificial flaws were introduced into the specimen at its center. Type-A flaws were introduced using a combination of drilling and focused ion beam (FIB) milling, whereas type-B flaws were introduced by FIB milling only. The corresponding dimensions of the type-A and B flaws are shown in Figures 4.1(b) and (c), respectively. Procedure of the fatigue test is similar to the procedure explained in Chapter 3 section 3.2.1.

After fatigue crack non-propagation was confirmed, microstructures at the crack tip were observed by electron backscatter diffraction (EBSD) and ECCI. After the fatigue tests and before the EBSD and ECCI observations, the specimens of all three steels were mechanically polished with colloidal silica for ambient time to remove the affected layers. The EBSD measurements were performed with a beam step size of 50 nm and an accelerating voltage of 20 kV. ECCI was performed using a solid-state four-quadrant backscattered electron (BSE) detector at an accelerating voltage of 30 kV, probe current of 10 nA, and working distance of 3 mm.

4.2.2 Prediction of fatigue life by Murakami's equation

In order to estimate the fatigue life and crack growth stress intensity threshold factor range for the three steels, Murakami's equation was used, which only requires the Vickers hardness HV, and $\sqrt{area}$ of the flaw to predict the small notch crack threshold [8].

$$\Delta K_{th} = 0.65 \times 2 \sigma_w \sqrt{\pi \sqrt{area}} \quad (R = -1) \quad (1)$$

$$\Delta K_{th_pre} = 3.3 \times 10^{-3} (HV + 120) (\sqrt{area})^{1/3} \quad (R = -1) \quad (2)$$

where σ_w and $\sqrt{area}$ are the fatigue crack non-propagation limit and the square root of the area respectively. The square root of the areas is obtained by projecting the flaw onto a plane perpendicular to the loading axis direction. The ΔK_{th_pre} term is the predicted ΔK_{th} . This equation is not only used to predict fatigue crack non propagation limit from a non-metallic inclusion, but also used to predict the small crack ΔK_{th} for various materials.

Murakami et al. [8] reported the accuracy of the prediction of this equation for various conventional steels.

4.3 Experimental Results

4.3.1 Fatigue life, ΔK_{th} , and relevant non-propagating crack growth characteristics.

Figure 4.2 shows the ΔK_{th} results of the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N steels with different notch sizes. In this study, fatigue limit is termed at the stress amplitude which does not result in failure at 2×10^7 cycles. Two specimens of each type of steel were tested to determine the fatigue limits. The fatigue limits of the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N steels are listed in Table 4.1. The ΔK_{th} values shown in Fig. 4.2 were estimated based on the fatigue limits. The threshold stress intensity factor (ΔK_{th}) values are determined to be the values under which the crack growth is not observable and it shows a non-propagating crack phenomena.

Table 4.1 shows the predicted and experimental ΔK_{th} for the three materials with the different notch sizes. The ΔK_{th} values of the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N steels were determined to be 2.45, 6.02, and 6.76 $\text{MPa}\sqrt{m}$ respectively for type-A notch and 2.46, 5.36, and 6.21 $\text{MPa}\sqrt{m}$ respectively for type-B notch. ΔK_{th} of the Fe-18Cr-14Ni steel is 25% less than the Murakami's equation prediction, ΔK_{th} of the Fe-23Mn-0.5C steel is 30% higher the Murakami's equation prediction, and ΔK_{th} of the Fe-25Cr-1N steel is within $\pm 10\%$ from the Murakami's equation prediction as shown in Fig. 4.2.

Figure 4.3 shows the fatigue crack length plotted against number of cycles for the non-propagating cracks of all three materials. The crack length in the Fe-18Cr-14Ni steel continuously increased until 1.75×10^7 cycles and then stopped to propagate until 2×10^7 cycles. In contrast, the Fe-25Cr-1N showed a steep crack length increment until 7.5×10^6 cycles, later the crack length was constant until 2×10^7 cycles. The Fe-23Mn-0.5C steel showed a small increment of crack length until 1.5×10^7 cycles, later the crack length was constant until 2×10^7 cycles. In addition, the Fe-23Mn-0.5C steel showed the lowest non-propagating crack length in comparison with the other two steels. Characteristics of non-propagating crack for all three materials are discussed in next section with replica, EBSD, and ECC images.

4.3.2 Microscopic crack growth features with associated dislocation pattern.

i. Fe-18Cr-14Ni steel

Figures 4.4(a)–(c) show the replica images of the Fe-18Cr-14Ni steel with the type-A notch as obtained during the fatigue test at 100 MPa. The length of the small fatigue crack increased continuously. Figures 4.4(d)–(i) show the results of the EBSD measurements corresponding to the crack shown in Figure 4.4(c). Specifically, Figures 4.4(d) and (e) show the inverse pole figure (IPF) maps in the rolling direction parallel to the loading direction (RD-IPF maps). The RD-IPF maps show that the crack propagated transgranularly and stopped propagating in the grain interior on both sides of the hole. Figures 4.4(f) and (g) show the grain reference orientation deviation (GROD) maps, which correspond to the degree of plastic deformation [9, 10]. In the GROD maps, the maximum deviation angle is approximately the same in the cracks on both the side, and the distribution of the deviation angles is heterogeneous. Figures 4.4(h) and (i) show image quality (IQ) maps with identified $\Sigma 3$ twin boundaries. The IQ value decreases when two Kikuchi patterns of the parent and twin crystals are overlapped. The decrease in IQ values indicates even thin deformation twin plates [11–13]. Only annealing twins, and no plate-like microstructures such as deformation twins, are observed in the IQ maps.

Figure 4.5(a) shows the ECC image of the Fe-18Cr-14Ni steel corresponding to the crack shown in Figure 4.4(d). Figure 4.5(b) shows the dislocation structure in the vicinity of the crack tip. The dislocations are tangled with each other, and dislocation cells or their precursors are formed. The dislocation structure is hereafter referred to as the dislocation cell. The cell size is in the range of 400–800 nm, and only a few dislocations are observed in the dislocation cells, as shown in Figure 5.5(c).

ii. Fe-23Mn-0.5C steel

Figures 4.6(a)–(c) show the replica images of the Fe-23Mn-0.5C steel with the type-A notch as obtained during the fatigue test at 245 MPa. Cracks initiated on both sides of the hole and fracture did not occur until 2×10^7 cycles. Subcracks were also observed, as indicated by the red arrows in Figure 4.6(c). Figures 4.6(d)–(i) show the EBSD images corresponding to the cracks outlined by the dashed lines in Figure 4.6(c). The cracks propagated transgranularly and stopped propagating at the grain boundary depicted in the RD-IPF map (Figures 4.6(d) and (e)). In the GROD maps (Figures 4.6(f) and (g)), the maximum deviation angle is approximately 10° and the distribution of the deviation angles around the crack is homogeneous. Figures 4.6(h) and (i) show IQ maps with identified $\Sigma 3$

twin boundaries. Only annealing twins, and no plate-like microstructures such as deformation twins, are observed in the IQ maps.

Figure 4.7(a) shows the ECC images of the Fe-23Mn-0.5C steel corresponding to the crack shown in Figure 4.6(d). Figures 4.7(b) and (c) show the dislocation structure in the vicinity of the crack tip. It is noteworthy that dislocation density in Figure 4.7(c) is higher than that of the Fe-18Cr-14Ni steel shown in Figure 4.7(c). Specifically, the inter-dislocation spacing is less than 100 nm in most parts of the ECC image in Figure 4.7(c).

iii. Fe-25Cr-1N steel

Figures 4.8(a)–(c) show the replica images of the Fe-25Cr-1N steel with the type-A notch as obtained during the fatigue test at 275 MPa. Cracks initiated on both sides of the hole, and the cracks did not propagate from 1×10^7 to 2×10^7 cycles, as shown in Figures 4.8(b) and (c). Figures 4.8(d)–(i) show the EBSD images corresponding to the cracks outlined by the dashed lines in Figure 4.8(c). The cracks propagated mostly in a transgranular manner, as shown in the RD-IPF maps (Figures 4.8(d) and (e)). In the GROD maps (Figures 4.8(f) and (g)), the maximum deviation angle is approximately 7° , and the distribution of the deviation angles around the crack is homogeneous. Figures 4.8(h) and (i) show the IQ maps with identified $\Sigma 3$ twin boundaries. Only annealing twins, and no plate-like microstructures such as deformation twins, are observed in the IQ maps.

Figure 4.9 shows the ECC images of the Fe-25Cr-1N steel corresponding to the cracks shown in Figures 4.8(d) and (e). Figure 4.9(a₁) shows the dislocation structure in the vicinity of the crack tip. This figure reveals that multiple slip systems are activated in the vicinity of the crack tip but the dislocations pattern on each slip plane is still planar. Figure 4.9(a₃) shows the dislocation intersection point far from the crack tip. The dislocation spacing increases with increasing distance from the intersection point, owing to dislocation pile-up stress. Figure 4.9(b) shows another example of a dislocation structure observed on the other side of the crack tip. Figure 4.9(b₁) shows the tip of the main crack and the branched crack (indicated by a white arrow) in the vicinity of this crack tip. Figures 4.9(b₂) and (b₃) show the tips of the main crack and subcrack, respectively. The dislocations are planar along the slip planes on which the crack tip lies. The dislocations emitted from the main crack interact with the pre-existing dislocations, similar to the phenomenon observed in Figure 4.9(a₃), whereas the tip of the branched crack directly meets the pre-existing dislocations after crack deflection, as indicated by the red arrow in Figure 4.9(b₃).

4.4 Discussion

4.4.1 Effects of solute atoms on crack tip microstructure

We observed that in the Fe-18Cr-14Ni steel, the dislocations at the crack tip were tangled with each other and dislocation cells were formed, as shown in Figure 4.5(c). The Fe-23Mn-0.5C steel showed a higher dislocation density at the crack tip than that of the Fe-18Cr-14Ni steel (Figure 4-7(c)). This higher dislocation density in the former steel can be attributed to deformation twinning [14] and DSA [15]. However, according to Figures 4.6(h) and (i), twinning was not observed near the crack. Therefore, the higher dislocation density at the crack tip in the Fe-23Mn-0.5C steel is attributed to the DSA caused by the Mn-C interaction. The DSA in Fe-Mn-C TWIP steels occurs not through multiple jumps of carbon atoms but rather through only a single jump of carbon atoms, which implies that dislocations are locked even in the vicinity of the crack tip [15]. If it is assumed that the dislocations are not depinned, additional dislocation multiplication is required to induce further plastic deformation, which results in the high dislocation density at the crack tip. In fact, a high increase rate of the dislocation density per unit strain has been reported in Fe-Mn-C Hadfield steel [16]. High-dislocation-density substructures act as strong barriers to dislocation glide at the crack tip.

In contrast, an important microstructural characteristic of the Fe-25Cr-1N steel is present on planar dislocation arrays. As can be seen in Figure 4.9(c), the dislocation array at the crack tip in the Fe-25Cr-1N steel is much more planar than that in the Fe-18Cr-14Ni steel. The dislocation structure in austenitic steel depends on the ease of cross-slip. The planar arrangement of dislocations on slip planes occurs in two cases: (1) when the SFE of the material is low [17, 18] and (2) in the presence of the short range ordering of Cr-N [19] in the alloy. It has also been reported that addition of nitrogen to austenitic stainless steel induces the planar arrangement of dislocations [20-23]. Thus, our new findings are that the nitrogen-enhanced dislocation planarity can remain even at the fatigue crack tip, which enhances resistance to dislocation motion.

4.4.2 Microstructure effects on fatigue crack closure and non-propagation

In the Fe-18Cr-14Ni steel, many slip bands are formed around the crack, as shown in Figures 4.4(a)–(c), which results in crack initiation along the slip bands [24]. In general, fatigue damage evolution in or along slip bands causes the fatigue crack initiation, and also assist subsequent propagation of the crack [25, 26]. Accordingly, the crack initiation and

crack propagation along slip planes observed in the Fe-18Cr-14Ni steel are also explained by the same mechanism. Therefore, motion and distribution of dislocations at a fatigue crack tip in a grain-size-scale are key to reveal underlying mechanism of resistance to small crack growth.

In the Fe-23Mn-0.5C steel, a non-propagating fatigue crack is observed (Figures 4.6(a)–(c)); strain age hardening at the fatigue crack tip facilitates the occurrence of non-propagation of a fatigue crack [27]. Specifically, as discussed above, DSA results in the formation of the high-dislocation-density substructure at the crack tip (Figure 4.7(c)). This substructure prevents the motion of crack tip dislocations, which, in turn, enhances the fatigue crack resistance. It is also noteworthy that the GROD maps in Figures 4.6(f) and (g) show that the crack tip and wake are homogeneously deformed with a higher plastic strain than that in the case of the Fe-18Cr-14Ni steel as shown in Figures 4.4(f) and (g). This fact indicates that the total plastic strain in the crack wake in the Fe-23Mn-0.5C steel is higher than that in the Fe-18Cr-14Ni steel, which leads to a higher compressive residual stress that activates plasticity-induced crack closure (PICC) [28] in the Fe-23Mn-0.5C steel. PICC can reduce the crack opening displacement, which consequently results in an early contact between the surfaces of the cracks and a reduction in the actual driving force for crack propagation [29]. Hence, the strain hardening at the crack tip due to the high dislocation density at this tip and the PICC together lead to the non-propagation of the fatigue crack in the Fe-23Mn-0.5C steel.

The Fe-25Cr-1N steel also shows a non-propagating fatigue crack (Figures 4.8(a)–(c)), but its underlying mechanism is different from that in the Fe-23Mn-0.5C steel. First, it is noteworthy that the dislocation density at the crack tip in the Fe-25Cr-1N steel does not show any drastic increase, as shown in Figure 4.9(a₁). In addition, the GROD maps in Figures 4.8(f) and (g) show that the size of the plastic zone in the crack wake in the Fe-25Cr-1N steel is smaller than those in the other two steels. However, the Fe-25Cr-1N steel shows a crack non-propagation phenomena, as depicted in Figures 4.8(a)–(c). Specifically, the crack propagation stopped inside the grain, as shown in Figures 4.9(a₁) and (b). Highly planar dislocations are observed around the crack tip (Figures 4.9(a₁) and (b₁)), and the dislocation motion is impinged by the dislocation arrays present on other slip planes indicated by the blue arrows in Figure 4.9(a₂). According to our previous study [30], planar dislocation arrays result in non-propagation of a fatigue crack because of dislocation pile-up stress caused by the impingement of the motion of planar dislocations. In addition, on the other side of the crack tip, a subcrack initiates around the main crack as indicated by

the white arrow in Figure 4.9(b₁). The planar dislocation array is observed at the main-crack tip (Figure 4.9(b₂)), which is similar to the observed arrangement in Figure 4.9(a₂); therefore, the dislocation pile-up stress prevents the crack opening. It is also noteworthy that a subcrack propagates along the slip planes as shown in Figure 4.9(b₂), but the crack is deflected. The tip of the deflected crack directly meets the planar dislocation array, as indicated by the red arrow in Figure 4.9(b₃). The dislocations emitted from the tip of the deflected crack interact with pre-existing dislocations indicated by the yellow arrows in Figure 4.9(b₃). Hence, Lomer–Cottrell sessile dislocations are formed on account of the interaction between the planar dislocation arrays on two different slip planes (Figure 4.9(a₃)). Kubota et al. [31] reported that the main mechanism of work hardening in high-nitrogen steel is the occurrence of a Lomer–Cottrell reaction. The formation of Lomer–Cottrell sessile dislocations induces a pile-up back-stress on the fatigue crack tip (Figure 4.9(b₃)), which results in non-propagation of the fatigue crack in the Fe-25Cr-1N steel.

4.5 Section Conclusions

In this study, fatigue tests of the Fe-18Cr-14Ni, Fe-23Mn-0.5C, and Fe-25Cr-1N steels were performed to elucidate the effects of the Mn-C and Cr-N couples on the dislocation structure at the non-propagating fatigue crack tip via ECCI. The following conclusions were drawn from the study findings.

- (1) The Fe-18Cr-14Ni steel showed dislocation cells at the fatigue crack tip.
- (2) The Fe-23Mn-0.5C steel showed a high dislocation density at the fatigue crack tip. DSA due to the Mn-C interaction resulted in the high dislocation density at the crack tip, which stopped the motion of crack tip dislocations. Therefore, both the strain hardening at the crack tip due to the high dislocation density at this tip and the plasticity-induced crack closure led to non-propagation of the fatigue crack in the Fe-23Mn-0.5C steel.
- (3) The Fe-25Cr-1N steel showed planar dislocation arrays at the fatigue crack tip. The enhanced dislocation planarity resulting from the Cr-N interaction led to the formation of Lomer–Cottrell sessile dislocations via the interaction of the dislocations. This resulted in non-propagation of the fatigue crack owing to the pile-up stress on the crack tip.

4.6 References

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4.7 Figures and Tables

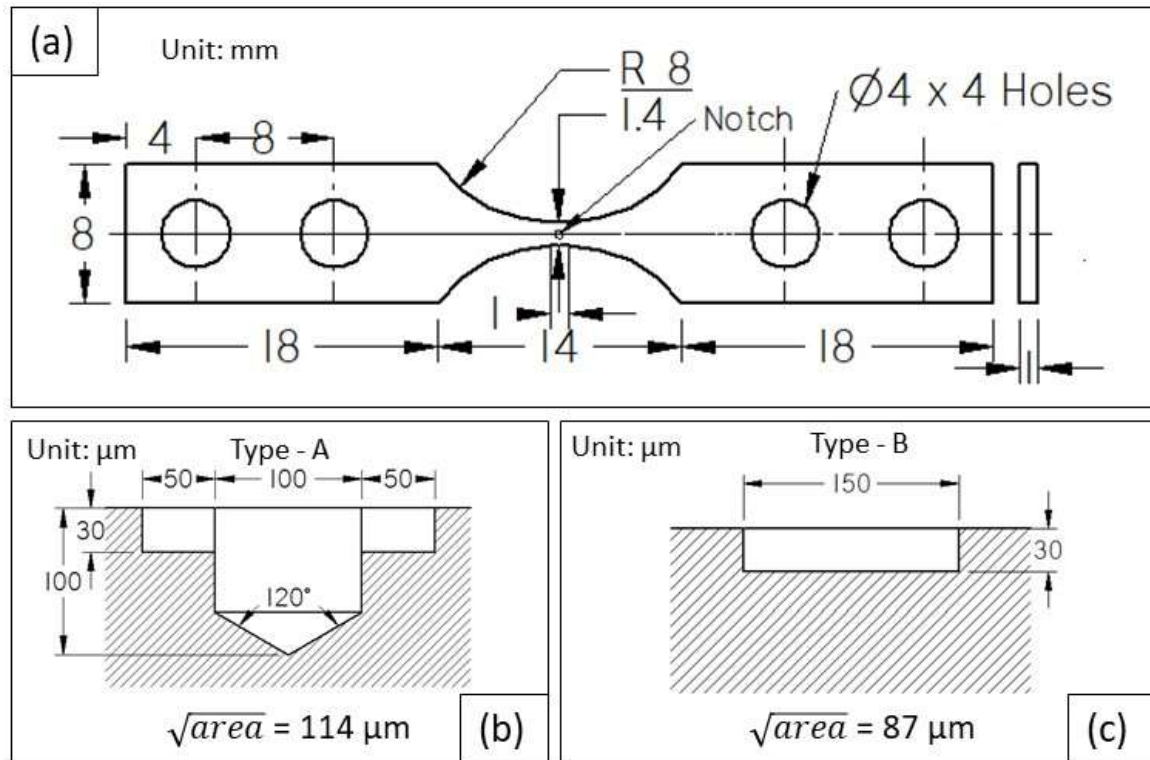


Fig. 4.1. (a) Specimen shape and dimensions. (b) Type-A notch. (c) Type-B notch.

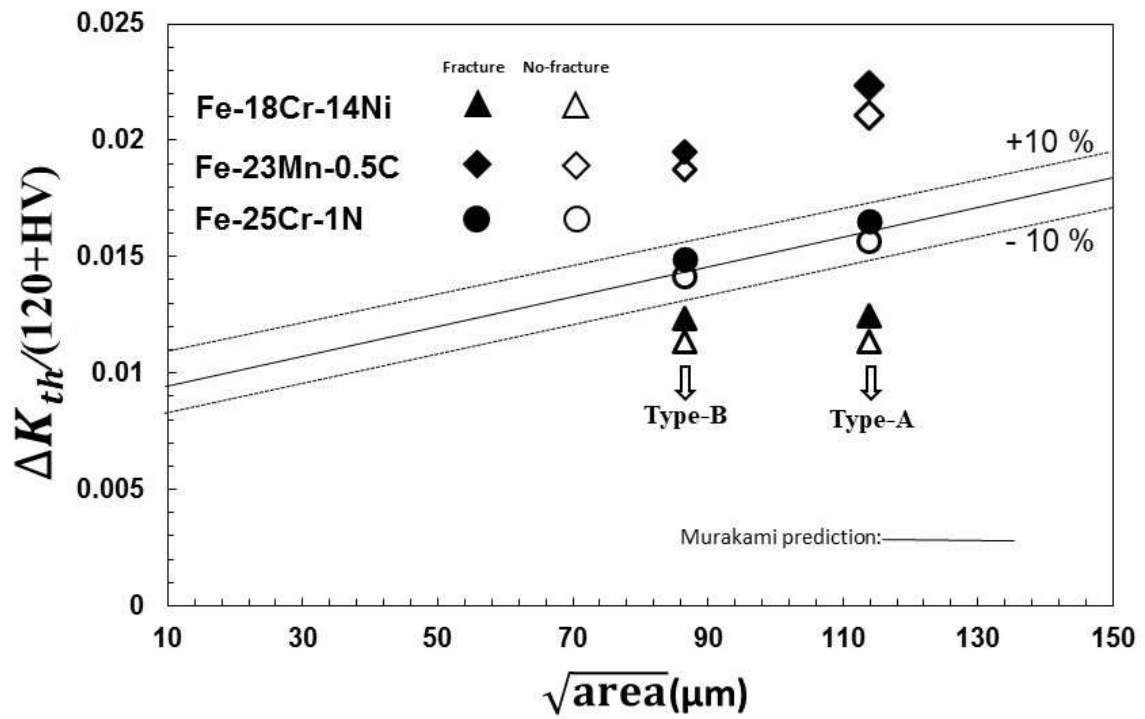


Fig. 4.2. Fatigue test results predicted using Murakami's equation. The dotted lines indicate $\pm 10\%$ error from the prediction curve.

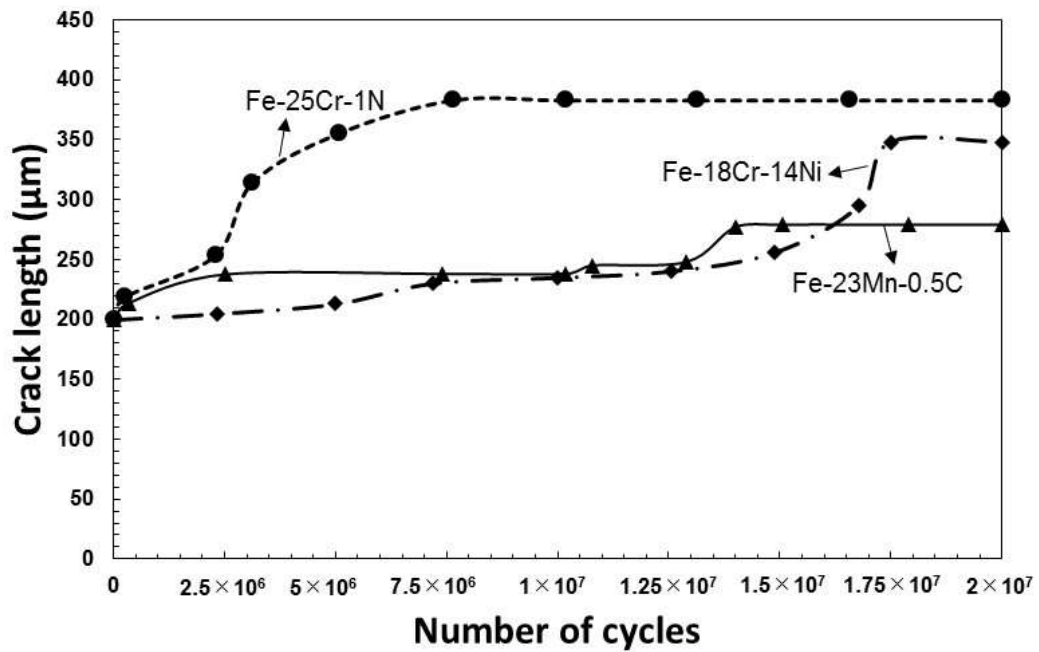


Fig. 4.3. Fatigue crack length plotted against number of cycles at fatigue limits of three steels with type-A notch.

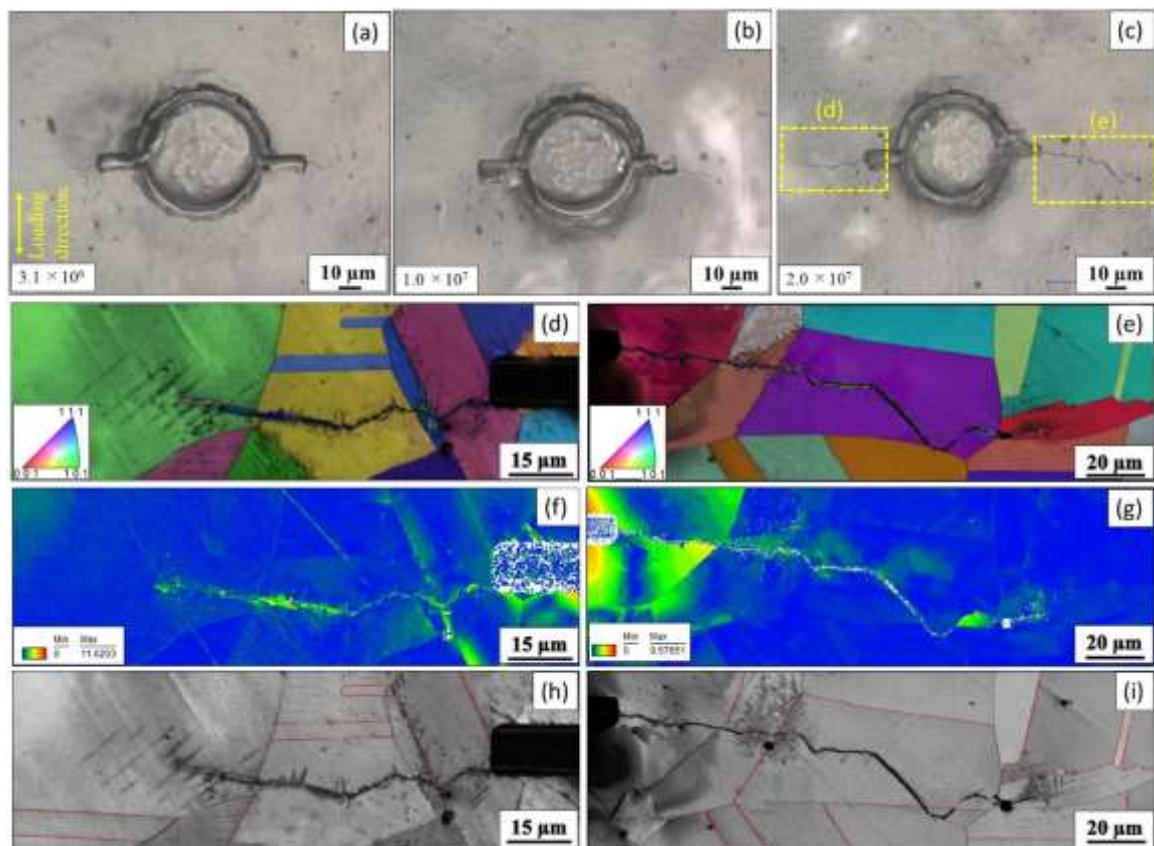


Fig. 4.4. (a)–(c) Replica images of Fe-18Cr-14Ni steel with type-A notch tested at 100 MPa: (a) 3.1×10^6 cycles, (b) 1.0×10^7 cycles, and (c) 2.0×10^6 cycles. Corresponding EBSD datasets: (d), (e) RD-IPF maps; (f), (g) GROD maps; and (h), (i) IQ maps. The red lines in (h) and (i) indicate the $\Sigma 3$ twin boundaries.

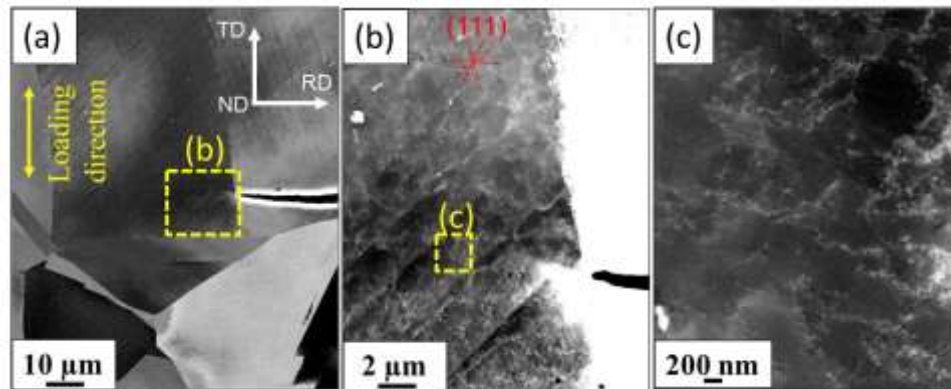


Fig. 4.5. ECC images of Fe-18Cr-14Ni steel: (a) image corresponding to crack shown in Figure 4.4(d). (b), (c) Magnified images of areas outlined by dashed lines in (a) and (b), respectively. The red lines in (b) indicate the (111) planes.

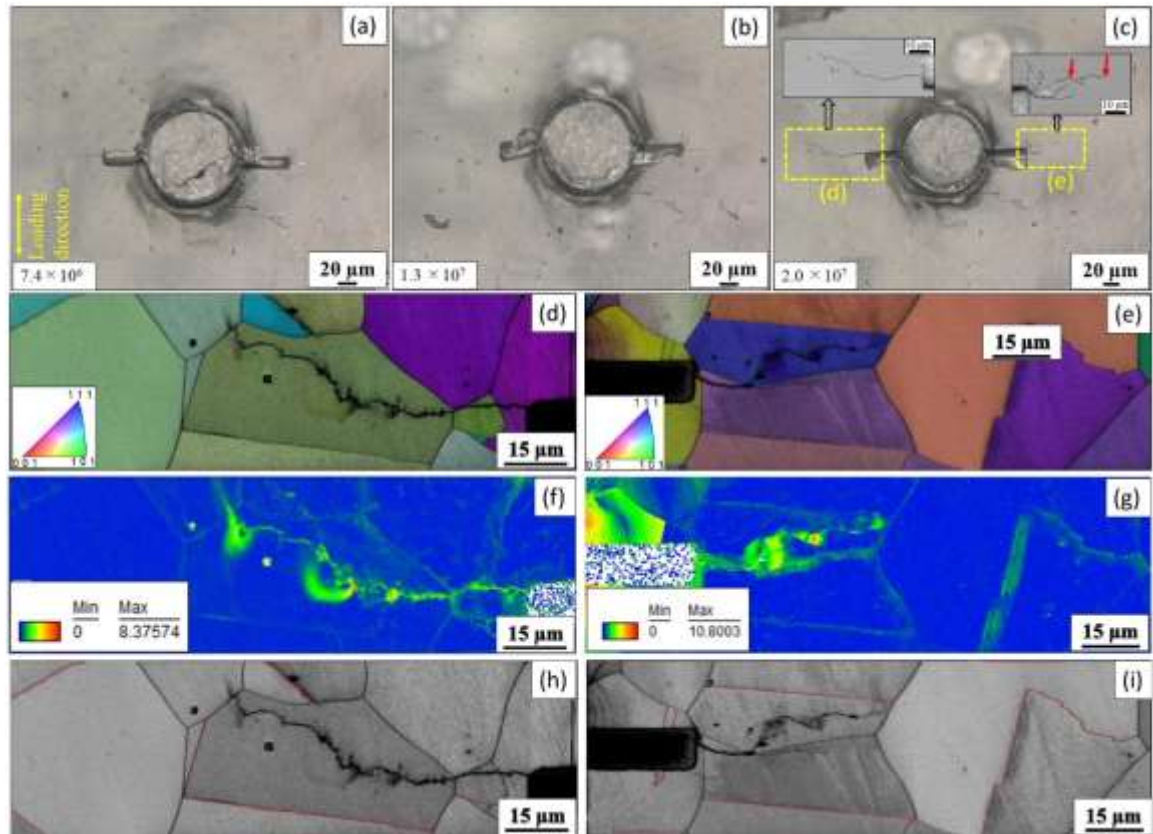


Fig. 4.6. (a)–(c) Replica images of Fe-23Mn-0.5C steel with type-A notch obtained during fatigue test at stress amplitude of 245 MPa. (d), (e) RD-IPF maps. (f), (g) GROD maps. (h), (i) IQ maps with identified $\Sigma 3$ twin boundaries. The red lines in (h) and (i) indicate the $\Sigma 3$ twin boundaries. The red arrows in (c) indicate the subcracks.

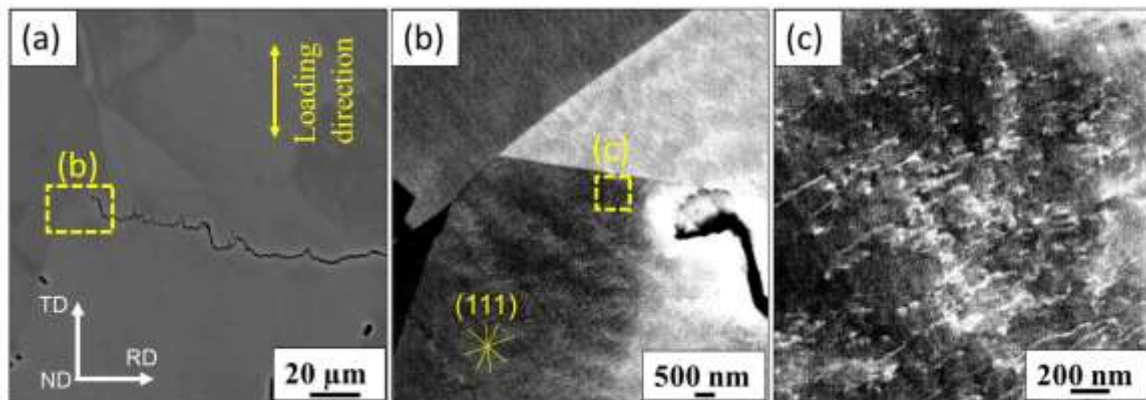


Fig. 4.7. ECC images of Fe-23Mn-0.5C steel. (a) Image corresponding to crack shown in Figure 4.6(d). (b), (c) Magnified images of areas outlined by dashed lines in (a) and (b), respectively. The yellow lines in (b) indicate the (111) planes.

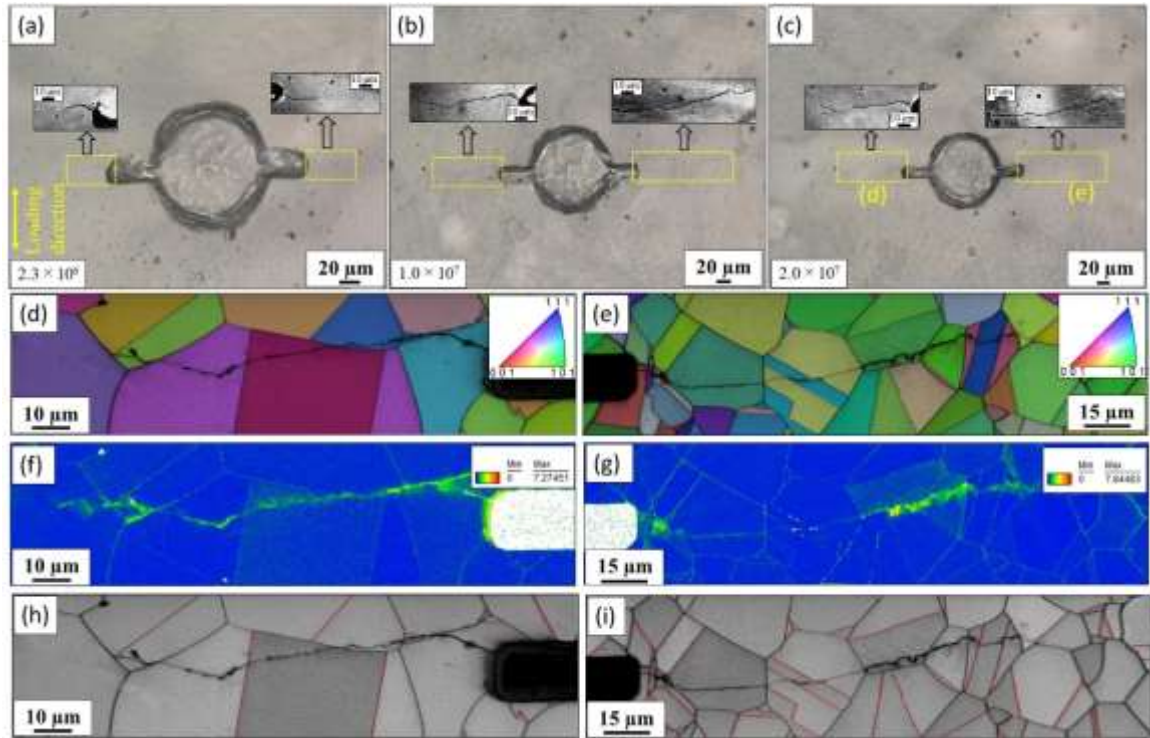


Fig. 4.8. (a)–(c) Replica images of Fe-25Cr-1N steel with type-A notch obtained during fatigue test at stress amplitude of 275 MPa. (d), (e) RD-IPF. (f), (g) GROD maps. (h), (i) IQ maps with identified $\Sigma 3$ twin boundaries. The red lines in (h) and (i) indicate the $\Sigma 3$ twin boundaries.

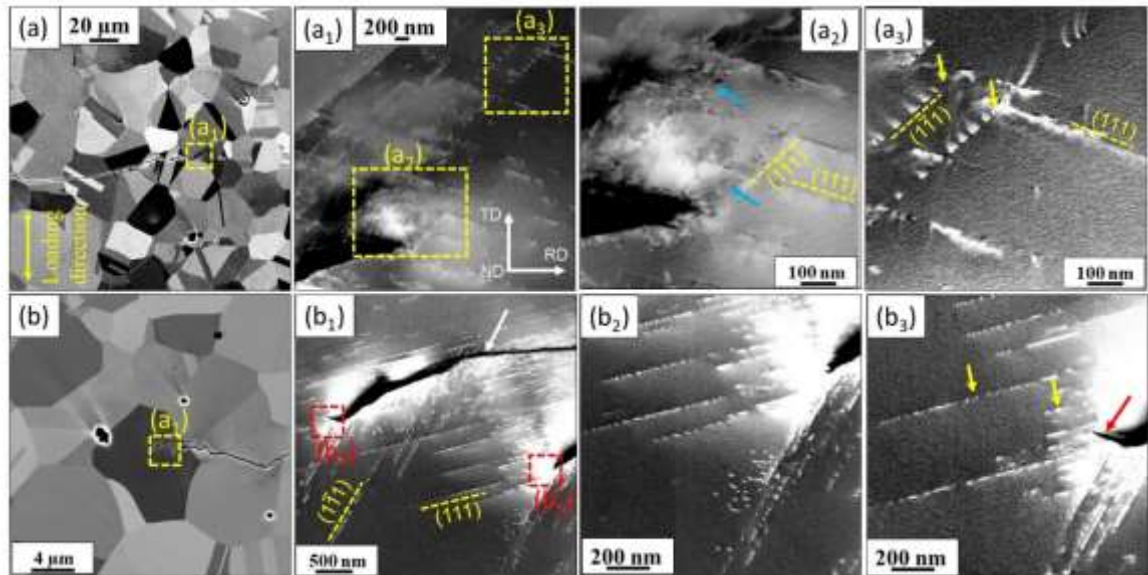


Fig. 4.9. ECC images of Fe-25Cr-1N steel. (a), (b) Images corresponding to cracks shown in Figures 4.8(e) and (d), respectively. (a₁), (b₁) Magnified images of areas outlined by dashed lines in (a) and (b), respectively. (a₂), (a₃) Magnified images of areas outlined by dashed lines in (a₁). (b₂), (b₃) Magnified images of areas outlined by dashed lines in (b₁). The blue and yellow arrows indicate the retarded dislocation motion at the intersection of two slip systems for each location shown in (a₂) and (a₃), respectively. The white and red arrows in (b₁) and (b₃) indicate the subcrack and the change in the propagation direction of the subcrack tip, respectively.

Table 4.1 Fatigue limits and predicted and experimental values of ΔK_{th} for three steels.

Material	Notch type	Fatigue limit (MPa)	ΔK_{th_pre} (MPa $\cdot\sqrt{m}$)	ΔK_{th} (MPa $\cdot\sqrt{m}$)	Vickers hardness Hv
Fe-18Cr-14Ni	Type-A	100	3.46	2.45	96
	Type-B	110	3.16	2.46	
Fe-23Mn-0.5C	Type-A	245	4.57	6.02	165
	Type-B	250	4.17	5.36	
Fe-25Cr-1N	Type-A	275	6.90	6.76	310
	Type-B	290	6.29	6.21	

Chapter 5

Fatigue crack initiation and its associated small fatigue crack propagation in Fe–N fully ferritic steel

5.1 Introduction

In terms of austenitic steels, the Interstitial solute atom interaction had a positive effect on the fatigue crack growth behavior and the fatigue limit is enhanced as described in Chapter 3, and Chapter 4. Hence solute atoms such as carbon and nitrogen would also have a similar effect as austenitic steels on enhancing the fatigue limit of ferritic steels. Actually in previous studies [1-4] with carbon as solute atoms in an Fe–C binary alloy ferritic steels, certain points were reported in terms of small crack growth behavior. First, transgranular cracks stopped at the grain interior, even at high stress levels, because of strain-age hardening at the crack tips within the grain interiors. Second, the failure mechanism in smooth specimens showed intergranular crack initiation and propagation, this perhaps may be because of lack of carbon segregation to the grain boundaries. Third, when the carbon content was increased from 0.006 mass% to 0.017 mass%, the Fe–C alloy showed a high coxing effect because of suppression of the intergranular fatigue crack initiation by strain aging; it also showed a higher fatigue limit because of strain-age hardening at the crack tip. However, the correlation between the diffusion/segregation of nitrogen as solute atoms and microstructurally small fatigue crack initiation and propagation has not been investigated. Therefore, in this chapter, study was performed on a single-ferrite-phase Fe–N binary steel, to clarify the underlying effects of solute nitrogen on the fatigue properties and its correlation to small fatigue crack, and we compared the effects with the solute carbon in an Fe–C binary system, and IF ferritic steels.

5.2 Experimental Procedure

In this study, Fe–0.0113N (mass %) ferritic steel that did not contain a cementite or pearlite phase was used. The detailed chemical composition is given in Chapter 2 Table 2.2. The fatigue test specimens were cut on a lathe machine, as shown in Fig. 5.1. The specimens were mechanically polished and subsequently etched with a mixture of 3% nitric acid and 97% ethanol (vol%). Tension–compression fatigue tests were performed at room temperature at a frequency of 30 Hz. The stress ratio R was -1 , and a sinusoidal waveform was employed. The fatigue limit was assumed to be the highest stress amplitude at which the specimen did not fracture at 1×10^7 cycles. The coxing effect was also studied for the

Fe–0.0113N steel. The fatigue test was stopped when the specimen was run until the fatigue limit up to 10^7 cycles, then the stress amplitude was increased by 5 MPa after a constant number of 10^7 cycles, to examine the degree of coxing effect. The replica technique was used to observe the fatigue cracks with the help of an optical microscope. The replica was immersed in methyl acetate before being placed on the specimen surface. The fracture surfaces were observed with a scanning electron microscope (SEM) at an accelerating voltage of 15 kV.

5.3 Experimental Results

5.3.1 Stress amplitude versus number of cycles to failure curves, and coxing effect plots

Figure 5.2(a) shows the diagram of stress amplitude–number of cycles to failure (S–N) for the Fe–0.0113N steel. For comparison, the Fe–0.017C, Fe–0.006C, and IF steels results are also plotted from our previous paper [2]. The fatigue limit of the nitrogen steel was determined to be 150 MPa. Normally, the differences in fatigue limit and fatigue life are derived from the tensile strength or hardness, therefore the number of cycles to failure was plotted against stress amplitude normalized by the Vickers hardness value, as shown in Fig. 5.2(b). It is clear that the fatigue limit of the Fe–0.0113N steel is lower than that of the Fe–0.017C, Fe–0.006C and IF steels. The stepwise lines indicate the results of the coxing effects test after 10^7 cycles. The Fe–0.0113N steel did not show a high coxing effect like the Fe–0.017C, it was similar to the Fe–0.006C steel and higher than the IF steel.

5.3.2 Fatigue crack initiation and propagation behavior

Figure 5.3 shows the replica images of the Fe–0.0113N steel at the fatigue limit (150 MPa). A fatigue crack initiated at the grain boundary (Fig. 5.3(b)) and propagated to the grain interior in both of the top and down grains (Fig. 5.3(c)). A subcrack also initiated along the grain boundary indicated by blue arrow in inset Fig. 5.3(c), and slip bands (SB) were observed along the grain boundaries, shown as an inset in Fig. 5.3(d). The subcrack coalesced with the main crack, as shown in Fig. 5.3(e). Due to subcrack formation near the main crack, stress release occurs especially when the subcrack is aligned parallel to the main crack [5]. Hence, the crack stopped propagating after 7×10^6 until 1×10^7 cycles, as shown in Figs. 5.3(e–f), indicating that non-propagating fatigue crack occurred at the fatigue limit.

Figures 5.4(a–b) show the replica images of Fe-0.0113N steel with intergranular and transgranular fatigue crack initiation at a stress amplitude of 220 MPa. The crack first initiated at the grain boundary at 3×10^4 cycles, as shown by the red arrows indicating the tips of one crack in Fig. 5.4(a). As the number of cycles increased to 6×10^4 cycles, the crack initiated at the grain interior, as shown by the blue arrow in Fig. 5.4(b). In addition, slip bands (SB) were formed around the grain boundary, shown by the magnified image in the inset of Fig. 5.4(b). The number of fatigue cracks increased with increasing number of cycles, as shown in Fig. 5.5, observed at a different area from Fig. 5.4. Primary crack initiation site is at the grain boundaries as shown by red arrow in Fig. 5.5(a), and propagated along grain boundary and grain interior as shown in Figs. 5.5(b–d). Slip bands were formed at the vicinity of the fatigue crack, as shown by blue arrow in Fig. 5.5(b). Crack propagation rate is increased due to crack coalescence, indicated by brown arrow in Fig. 5.5(c).

Figure 5.6 shows the fatigue crack growth rate of the Fe–0.0113N steel at a stress amplitude of 180 MPa. The fatigue crack propagation rate increased at some stages, marked by arrows in Fig. 5.6(b). This sudden increase in crack propagation was caused by crack coalescence of the fatigue cracks, as shown by red arrow in Fig. 5.7(e). Even at higher stress amplitudes above the fatigue limit, the fatigue crack initiated along the grain boundaries, as shown by green arrow in Fig. 5.7(a), and propagated along the grain boundaries and grain interior, as shown in Figs. 5.7(b–e). Slip bands were also observed at the vicinity of the crack, as shown by the blue arrows in Fig. 5.7(d).

5.3.3 Fractographs

Figure 5.8 shows the SEM fractographs of the Fe-0.0113N steel. Figures. 5.8(a–b) shows an overview of the fracture surface. Fig. 5.8(c) shows the crack initiation site from the top surface, an intergranular fracture surface was observed, indicated by the blue arrows. Striation-like features were observed at the mid-surface, indicated by small red arrows in Fig. 5.8(d).

5.4 Discussion

5.4.1 Fatigue crack initiation and propagation behavior

Crack initiation occurred at the grain boundaries in the Fe-0.0113N steel as shown in Fig. 5.4(a) and later in the grain interior (Fig. 5.4(b)). This result of the Fe–N steel is similar to the Fe-0.017C steel [1]. In the fatigue process, once the critical resolved shear stress is reached the dislocations start to multiply at the grain boundaries, and start to glide on the

slip system in preferentially oriented grains. Dislocations continues to slip until the dislocations reach an obstacle such as grain boundary. The cyclic loading increases the dislocation density at the grain boundaries, resulting in the formation of ledges, intrusions and extrusions at the grain boundaries and free surfaces. The increase in defects pile-up at the grain boundaries, resulting in stress concentration that activates slip in the neighboring grain (Fig.5.3(c)). As the accumulation of plastic strain increases within a grain or across several grains the strain is localized into a small volume within persistent slip bands (PSB's) in the material [6]. Therefore, the dislocation patterning forces the material to crack at the grain boundary. The intergranular feature on the fracture surface as marked by blue arrows in the fractograph of Fig. 5.8(c) also indicates grain boundary crack initiation. In addition, as the number of cycles increased, the crack propagated along the grain boundaries as shown in Fig. 5.5(b-d) with slip bands in the vicinity of the fatigue crack, as shown by blue arrow in Fig. 5.5(b). It indicates that the fatigue crack propagation is closely associated with local plastic deformations. The crack propagation along the grain boundaries occurs through repeated crack initiation and their coalescence as shown in Fig. 5.7(e) by red arrow. The striations observed at the mid surface shown by small red arrows Fig. 5.8(d) proves that the crack propagation is due to crack blunting and resharpening [7, 8]. Thus conventional mechanism of crack blunting and resharpening is responsible for crack propagation in the Fe-0.0113N steel similar to the Fe-0.017C steel.

5.4.2 Effect of nitrogen on grain boundary crack initiation

As discussed above, the major mode of crack initiation in the Fe-0.0113N steel was at the grain boundaries, similar to the results of the Fe-0.017C steel [1]. The major reason for the ease of cracking along grain boundaries in the Fe-0.0113N steel is perhaps because of the lower grain boundary strength than the Fe-C steel. The addition of nitrogen seemed to harden the grain interior because of the presence of nitrogen atoms within the grain, but nitrogen has less of a tendency to cause grain boundary segregation in comparison with carbon [9-12]. Therefore, the ease of intergranular crack initiation would be attributed to the low amount of solute nitrogen in the grain boundaries in the water-quenched steels. The effect of reducing the grain boundary strength is considered to support the crack initiation in grain boundaries that are damaged significantly by localization of plastic strain.

Furthermore, because of frequent crack initiation at the grain boundaries and subsequent coalescence, the main crack becomes longer and the fatigue crack propagation no longer stopped when the load was increased for the coaxing test. Hence, the Fe-0.0113N

steel did not show high coxing effect and fractured at the third increment stage of stress amplitude (Fig. 5.2(a)). In contrast, the Fe-0.017C steel showed a high coxing effect: nine increments were required for fracture (Fig. 5.2(a)). It is because strain-age hardening suppressed the intergranular fatigue crack initiation during the coxing test of the Fe-C steel [2]. The strain-age hardening effect of nitrogen on intergranular crack initiation resistance had less of an effect than that of carbon. In this context, the nitrogen effect on transgranular crack resistance associated with DSA would also be different from that of solute carbon, which showed a dramatic improvement of the threshold-stress intensity factor range for small crack growth [3, 4]. Therefore, to study the behavior of nitrogen on transgranular crack propagation resistance, the threshold-stress intensity factor for micro-notched specimens should be investigated in a future work.

5.4.3 Effect of nitrogen on the fatigue limit

In terms of the fatigue limit when normalized with the Vickers hardness value, the Fe-0.0113N steel showed a lower fatigue limit than the Fe-C steels and IF steel (Fig. 5.2(b)). In all the steels, crack initiation occurred at the grain boundaries. Therefore, the lower fatigue limit of the Fe-0.0113N steel is perhaps due to a lower grain boundary strength against fatigue. In comparison with the IF steel, the Fe-0.0113N steel showed a lower fatigue life because the relative crack initiation resistance was poor in the Fe-0.0113N steel. As discussed above in the Fe-0.0113N steel, many cracks initiated at the grain boundary (Figs. 5.5 & 5.7). It has positive and negative effects on fatigue crack growth. In terms of positive effect subcrack formation (Fig. 5.3(d)) near the main crack tip causes stress re-distribution near a crack tip [5], and roughness induced crack closure [13]. Hence the Fe-0.0113N steel showed a non-propagating fatigue crack at the fatigue limit (Fig. 5.3) due to the formation of a subcrack (Fig. 5.3(d)). In terms of negative effect, the subcrack coalesces with main crack (Fig. 5.7(e)) and increased the fatigue crack growth at higher stress above the fatigue limit, hence reducing the fatigue limit of the Fe-0.0113N steel. In addition, the Fe-0.0113N steel showed a lower fatigue limit than the Fe-C steels because of the absence of a DSA effect (Fig. 2.5(b)) in the Fe-0.0113N steel as discussed in Chapter 2.

5.5 Section conclusions

In this study, the effect of solute nitrogen on the fatigue crack initiation and small fatigue crack propagation was studied in a Fe–0.0113N single-phase ferritic steel, and the results were compared with those from our previous papers for Fe–0.017C, Fe–0.006C, and IF steels [1, 2]. The following conclusions are made from the results:

- (1) The fatigue limit of the Fe–0.0113N steel was determined to be 150 MPa, and it showed non-propagating fatigue crack phenomena at the fatigue limit.
- (2) The Fe-0.0113N steel did not show high coaxing effect, because of frequent crack initiation at the grain boundaries and subsequent coalescence, the main crack becomes longer and the fatigue crack propagation no longer stopped when the load was increased for the coaxing test.
- (3) The major mode of fatigue crack initiation in the Fe-0.0113N steel is at the grain boundaries because of the low amount of solute nitrogen at the grain boundaries in the water-quenched steels.

5.6 References

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5.7 Figures

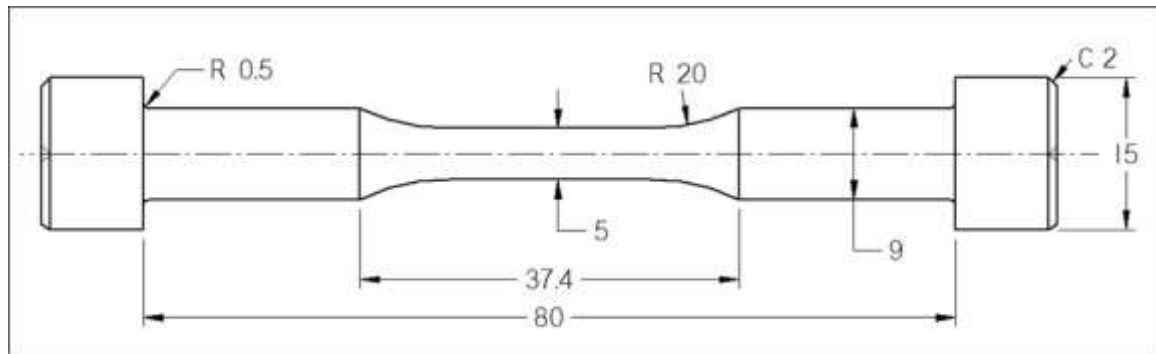


Fig. 6.1 Fatigue test specimen geometry in millimeters (mm).

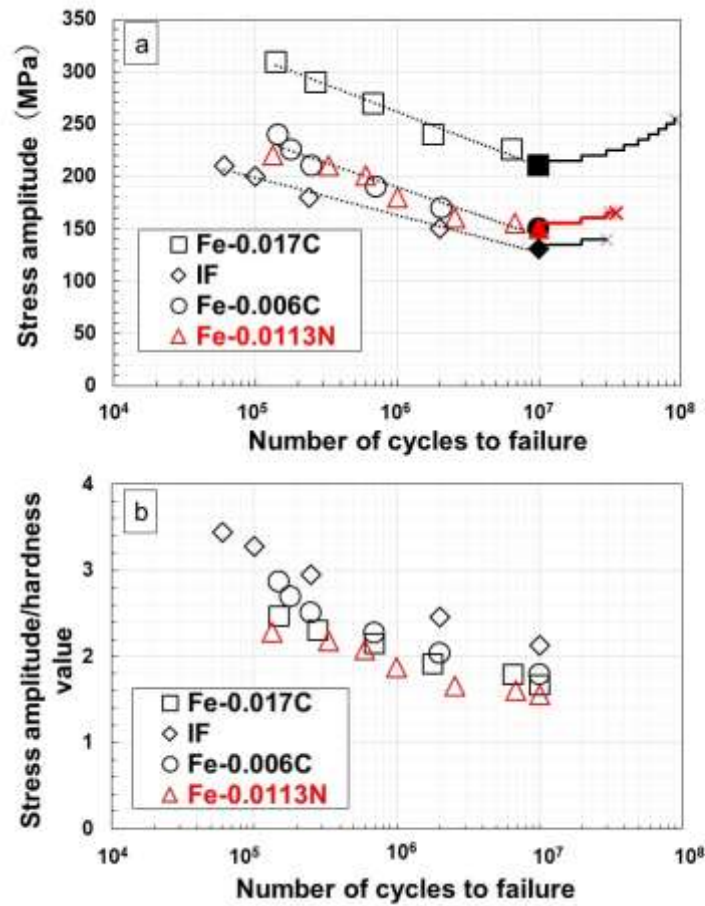


Fig. 6.2. (a) Stress amplitude vs. number of cycles curves (S–N). The open marks show the fatigue lives of broken specimens and the solid marks show the unbroken specimens. (b) Number of cycles to failure plotted against stress amplitude normalized by Vickers hardness value. S–N curve data of the Fe–0.17C, Fe–0.006C, and IF steels are from previous paper [2].

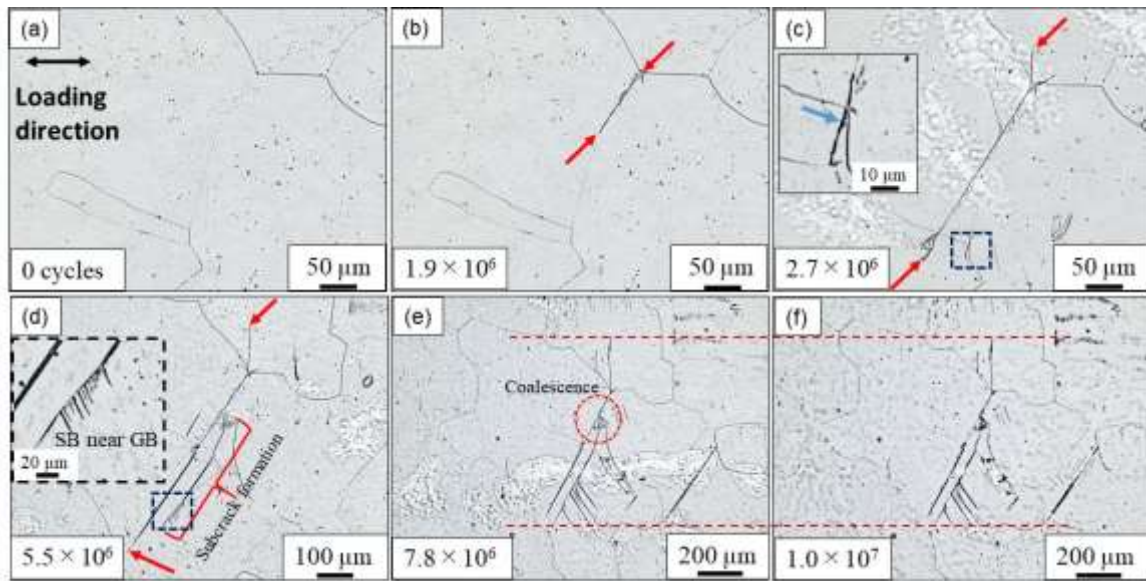


Fig. 6.3 Replica images showing non-propagating fatigue cracks of Fe-0.0113N steel at the fatigue limit (150 MPa): (a) 0, (b) 1.9×10^6 , (c) 2.7×10^6 , (d) 5.5×10^6 , (e) 7.8×10^6 , and (f) 1×10^7 cycles. Note that the magnification is different in (d-f).

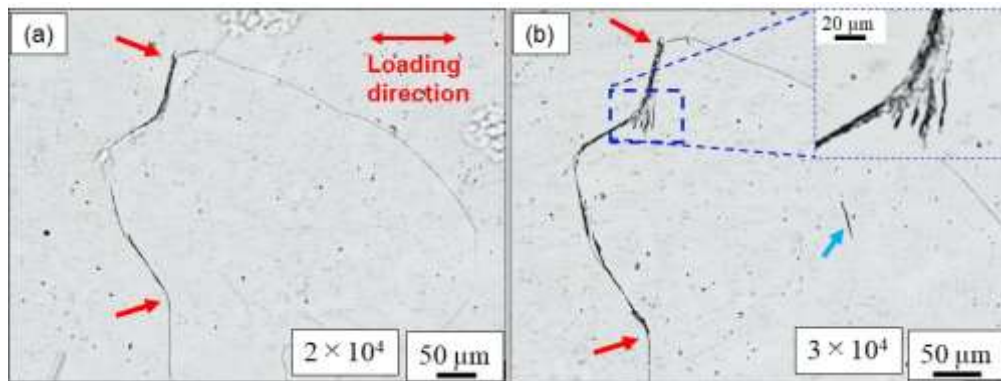


Fig. 6.4 Replica images showing (a) intergranular ($N = 2 \times 10^4$) and (b) transgranular ($N = 3 \times 10^4$) fatigue crack initiation at a stress amplitude of 220 MPa.

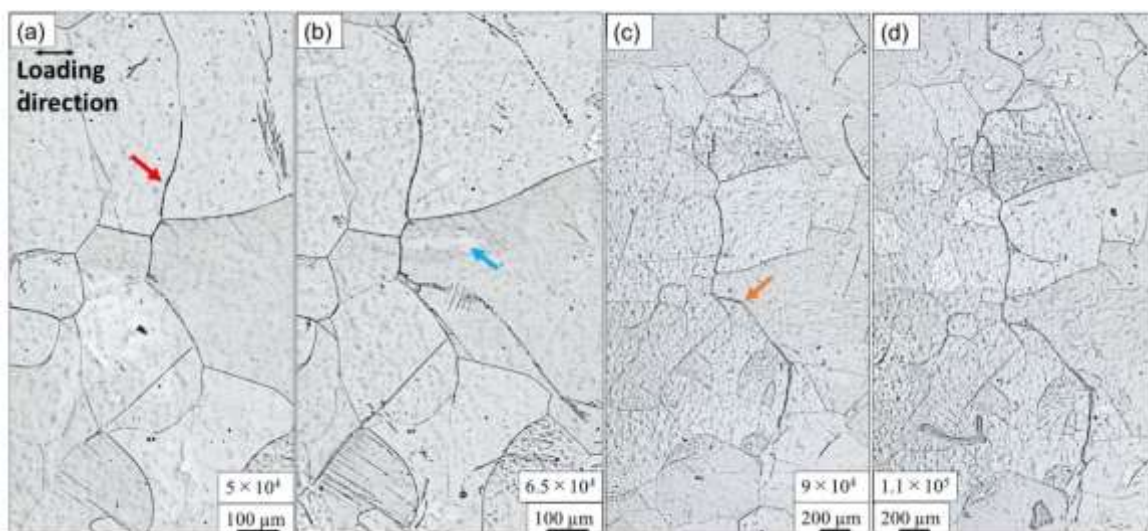


Fig. 6.5 Replica images of the Fe-0.0113N steel showing intergranular and transgranular fatigue crack propagation at a stress amplitude of 220 MPa: (a) 5×10^4 , (b) 6.5×10^4 , (c) 9×10^4 , and (d) 1.1×10^5 cycles.

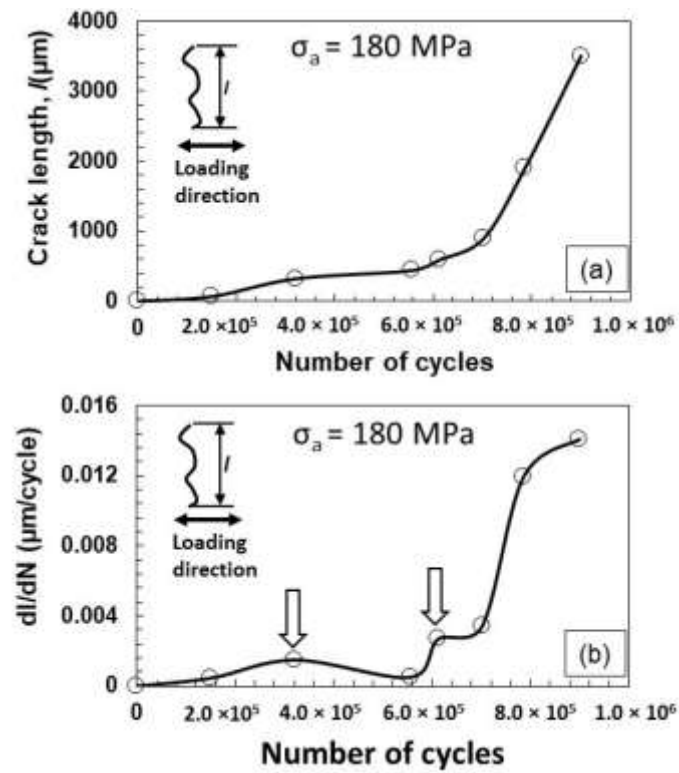


Fig. 6.6 (a) Fatigue crack growth rates of the Fe-0.0113N steel at 180 MPa stress amplitude. (a) Crack length versus number of cycles and (b) fatigue crack growth rate versus crack length.

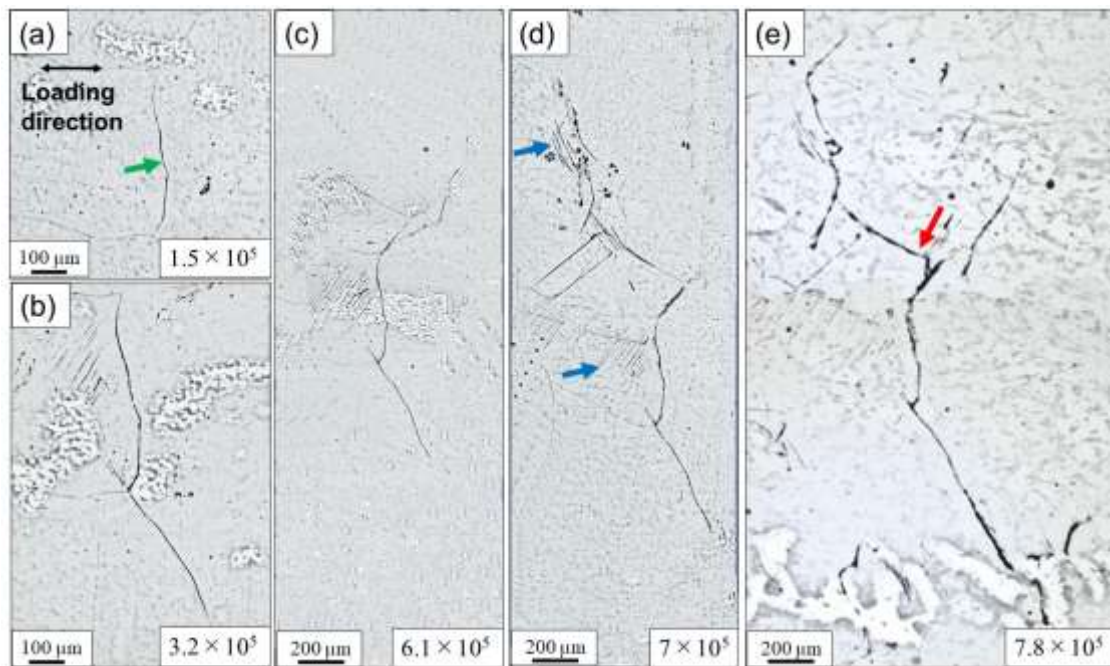


Fig. 6.7 Replica images of the Fe-0.0113N steel showing fatigue crack propagation and its associated coalescence at a stress amplitude of 180 MPa: (a) 1.5×10^5 , (b) 3.2×10^5 , (c) 6.1×10^5 , (d) 7×10^5 , (e) 7.8×10^5 cycles.

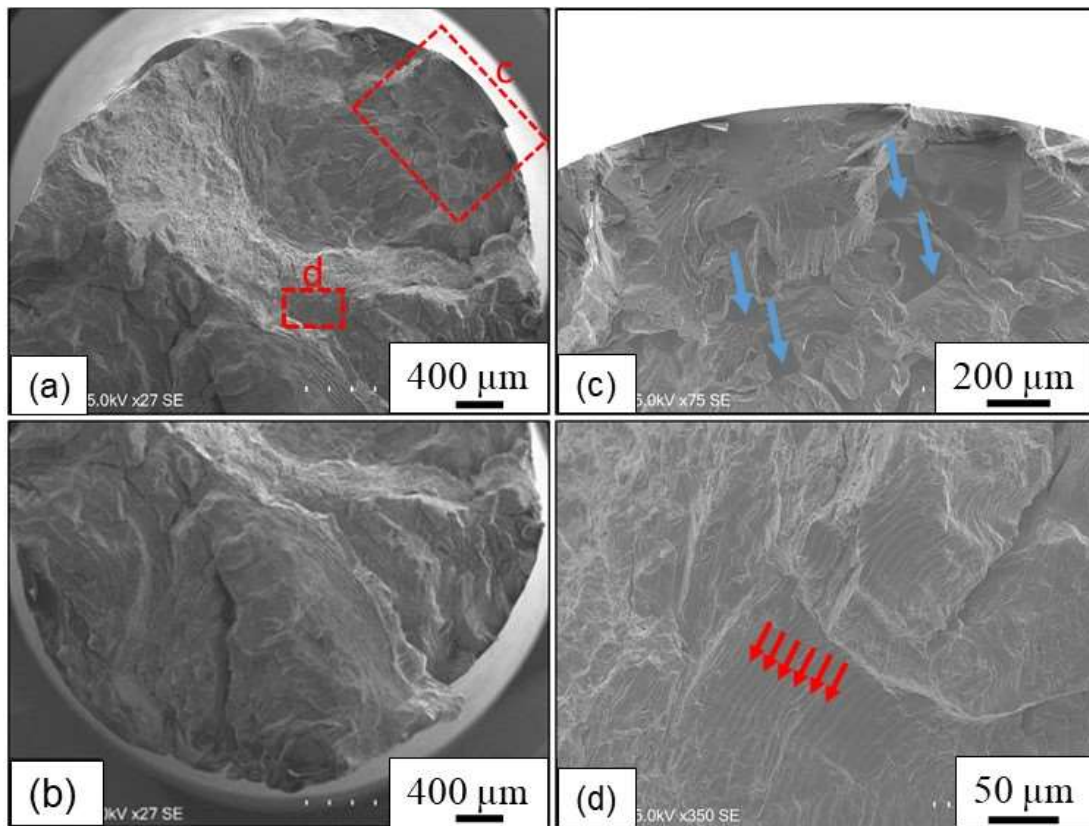


Fig. 6.8 SEM fracture surface images of the Fe-0.0113N steel tested at 220 MPa. (a and b) Overview of the fracture surface, (c and d) magnified images of the regions outlined by dashed lines in Fig. (a). Blue arrows in Fig. (c) indicates the intergranular fracture surface feature, and small red arrows in Fig. (d) indicate striations like feature.

Chapter 6

General Conclusion

In this research work, to analyze the influence of Interstitial solute atom interaction on the fatigue crack growth resistance fatigue tests were conducted. In terms of austenitic steels, the effect of DSA due to Mn-C couples, and enhanced dislocation planarity due to Cr-N couples on the fatigue crack growth behavior and the corresponding threshold stress intensity factors were examined in comparison with the normal steel without any I-S interaction. In terms of ferritic steels, effect of solute nitrogen on the fatigue crack initiation and propagation behavior was examined and compared with the previous results of Fe-C, and IF ferritic steels. The detailed conclusion and information were shown as followed.

Rotating bending fatigue tests were conducted on three types of austenitic steels, i.e. Fe-23Mn-0.5C, Fe-25Cr-1N, and Fe-18Cr-14Ni steels. Fatigue limit of the Fe-23Mn-0.5C, Fe-25Cr-1N, and Fe-18Cr-14Ni steels are determined to be 260 MPa, 110 MPa, and 310 MPa. The ratio of the fatigue limit/tensile strength of the Fe-23Mn-0.5C and Fe-25Cr-1N steels is higher than the Fe-18Cr-14Ni steel.

The Fe-23Mn-0.5C steel showed a lower fatigue crack propagation rate and higher fatigue limit compared to the Fe-18Cr-14Ni steel because of an attractive Mn-C atomic interaction. The Mn-C interaction has the following effects on the fatigue crack growth behavior: As a negative effect, deformation-induced decomposition of Mn-C couples results in local softening, causing strain localization that cause crack initiation along $\{111\}_{\gamma}$ slip planes. On the other hand, the ease of crack initiation along the slip planes assists crack branching, zigzag crack growth, and subcrack formation. These factors enhance roughness-induced crack closure. In addition, the formation of subcracks brings about stress release at a crack tip. Furthermore, as previously reported, the occurrence of dynamic strain aging arising from Mn-C couples assists fatigue crack non-propagation. These positive effects contributed to enhanced fatigue crack propagation resistance, which results in the lower fatigue crack propagation rate of the Fe-23Mn-0.5C steel compared to that of the Fe-18Cr-14Ni alloy.

The Fe-25Cr-1N steel exhibited a lower fatigue crack growth rate than the Fe-18Cr-14Ni steel because of the enhanced dislocation planarity produced by the Cr-N interaction. The Cr-N interaction affected the fatigue crack growth behavior as follows. The short crack region exhibited a planar glide dislocation pattern, but multiple slip systems were activated as the crack lengthened. As the dislocation pattern remained planar on each slip plane, the

crack propagation occurred along the $\{111\}_\gamma$ slip planes, even in the long crack. Moreover, the dislocation pile-up at the grain boundaries caused grain boundary subcracks, which can induce crack toughening through mechanisms such as stress redistribution. These positive effects contributed to the lower fatigue crack growth rate in the Fe-25Cr-1N steel than the Fe-18Cr-14Ni steel.

Threshold stress intensity factor range was investigated in the Fe-25Cr-1N, Fe-23Mn-0.5C, and Fe-18Cr-14Ni steels with two different size of notches. Murakami's equation is used to determine the ΔK_{th} from the stress amplitude showing non-propagating fatigue cracks. The Fe-25Cr-1N, and Fe-23Mn-0.5C steels showed higher ΔK_{th} than the Fe-18Cr-14Ni steel for both the notches. To study the effect of Mn-C and Cr-N on ΔK_{th} we applied electron channeling contrast imaging near tips of non-propagating fatigue cracks to observe the dislocation structure. Higher ΔK_{th} in the Fe-23Mn-0.5C steel is attributed to local hardening at the crack tip owing to dislocation density increment by dynamic strain aging. In case of the Fe-25Cr-1N steel, the enhanced dislocation planarity stemming from Cr-N interaction acts as significant factor increasing crack resistance. The enhanced dislocation planarity resulted in the dislocation pile-up stress at the crack tip, preventing dislocation emission from the crack tip.

To study the solute nitrogen on the fatigue crack initiation and propagation effect in the ferritic steels, Fe-0.0133N binary ferritic steels was used. Tension compression and tension tests were conducted at room temperature and the results were compared with the Fe-0.017C, Fe-0.006C, and IF steels. The Fe-0.0113 mass% N steel showed a fatigue limit at 150 MPa and a non-propagating fatigue crack at the fatigue limit. Intergranular and transgranular crack initiation were observed, and cracks propagated along the grain boundaries and grain interiors at stresses above the fatigue limit. The Fe-N steel did not show a high coaxing effect because of repeated crack initiation and coalescence with the main crack. The fatigue limit of the Fe-N steel was lower than that of Fe-C and IF steels when normalized with the Vickers hardness value.

The strain-age hardening effect of nitrogen on intergranular crack initiation resistance had less of an effect than that of carbon. In this context, the nitrogen effect on transgranular crack resistance associated with DSA would also be different from that of solute carbon, which showed a dramatic improvement of the threshold-stress intensity factor range for small crack growth. Therefore, to study the behavior of nitrogen on transgranular crack propagation resistance, the threshold-stress intensity factor for micro-notched specimens should be investigated in a future work.

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