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Structure property correlation and cyclic behaviour of microalloyed high strength structural steel

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Abstract

Microalloyed structural steels are extensively used in load bearing applications that need superior weldability, as well as an optimum combination of strength, ductility, and fatigue resistance, according to carbon equivalent criteria. This study investigates the influence of microstructural characteristics, particularly grain size, microalloying, pearlite morphology, and phase fraction, on the tensile, and fatigue behaviour of three microalloyed steels. A combination of ferritic and pearlitic microstructure was observed in all the steels. Steel 1 exhibited the highest yield strength (582 ± 14 MPa) and ultimate tensile strength (662 ± 18 MPa), along with an impact energy of 61.51 ± 2.1 J, which was attributed due to combined effect of fine grain size, lower carbon content, lower crystallite size, higher microstrain and higher dislocation density. However, comparatively lower elongation ($18.35 \pm 2.5\%$) and microhardness (209 ± 5 HV_{0.2}). In contrast, steel 3 demonstrated the highest elongation ($26.89 \pm 2.23\%$) and microhardness (280 ± 6 HV_{0.2}), associated with its coarser grain structure, higher carbon content, reduced pearlitic interlamellar spacing, and phase fraction. Under cyclic loading, steel 2 exhibits the highest fatigue strength of 286 MPa at stress amplitude of 280 MPa for 10^6 cycles, followed by steels 1 and 3. Microstructural analysis revealed extensive dislocation structures and intergranular deformation in steel 1, promoting localised slip

accumulation and early crack initiation. Steel 2 exhibited more uniform cyclic deformation due to its fine pearlite spacing, enabling improved strain accommodation and superior fatigue performance. Conversely, steel 3 showed pronounced dislocation entanglement at pearlite ferrite interfaces, resulting in strain localisation and microcrack initiation.

Keywords: Structural steel, Hot rolling, Microstructure, X-ray diffraction, High cycle fatigue

1. Introduction

Structural steels are widely used in civil infrastructure, industrial machinery, transportation equipment and offshore apparatus. Among the various grades of structural steels, E450 grade structural steel has become a prevalent high-strength steel owing to its optimal balance of strength and ductility and weldability (Ref 1). The microstructure is often comprising of refined ferrite-pearlite structures with optimum strength, ductility, and toughness (Ref 2,3). E450 grade steel is commonly used in bridges, building frames, offshore platforms, towers, storage tanks, pressure vessels, and other critical infrastructure components subjected to fluctuating load (Ref 4,5). These features make E450 a preferred choice for structural application expected to sustain cyclic loading. Moreover, thermomechanical processing and micro alloying play a vital role in improving the mechanical properties, weldability and fatigue life (Ref 6,7). High strength, and fine grain martensite was achieved using hot rolling and quenching of metastable austenite (Ref 8). Increase of C content in hot rolled E410 steels exhibits a reduction of ~22% in the yield strength and an increase in ductility by ~4% (Ref 9). Increasing the carbon content from 0.01wt.% to 0.4wt.% in 5% Mn steels slowing down the cooling rate leads to the development of carbides and the improvement in the yield strength (Ref 10). Mn concentration between 2 to 10% contributed to the strengthening of the steels however Mn content between 11-14% acts as a strong austenite stabiliser (Ref 11). Synergic addition of Mn and C enhance the UTS and YS of the E450 hot rolled structural steel. Very low Nb content in pipeline steel exhibited a

stronger rotated cube texture and a bimodal surface grain structure, leading to pronounced surface centric differences in strain distribution and grain states, which resulted into reduction of strength by 10% (Ref 12). Although thermomechanical processing and microalloying improve the mechanical properties of the structural steels carbon equivalent (CE) is more critical parameter governing the weldability and fatigue response (Ref 13,14). The CE provides an empirical measure of steel's hardenability based on its alloying elements, enabling prediction of its susceptibility to welding-induced cracking, and formation of undesirable brittle phase (Ref 15). High carbon equivalent values promote the formation of hard, brittle microstructures such as martensite in the HAZ, increasing the risk of hydrogen-assisted cracking and reducing both toughness and fatigue resistance (Ref 16,17).

The fatigue resistance of structural steels is highly sensitive to microstructural characteristics including grain size distribution, dislocation density, and grain orientation (Ref 18,19). These microstructural characteristics determine the steel's response to cyclic stress, affecting dislocation movement, strain localisation, phase change, and eventually the initiation of fatigue cracks (Ref 7,18–22). Steels with ferritic and pearlitic microstructure exhibit the higher fatigue notch sensitivity in contrast to steel consists of quenched and tempered microstructure (Ref 23). In high-cycle regimes, characterised by limited macroscopic plastic deformation, small-scale cyclic slip mechanisms predominate (Ref 20). Crack initiation can arise from microstructural discontinuities, including inclusions, second-phase particles, or heterogeneities that locally intensify stress (Ref 24). Upon crack initiation, crack propagation is governed by the interplay of the cyclic stress intensity factor, microstructural impediments, and the developing crack-tip plastic zone (Ref 25). In addition, loading direction in hot rolled steels also affects fatigue life. Samples loaded parallel to the rolling direction demonstrate greater fatigue strength than those loaded perpendicular to the loading direction (Ref 26).

Despite structural steels is widely used in fatigue applications and ultra-low cycle fatigue behaviour of E250, E350 and 450 was investigated by Tamilselvan et al (Ref 24). High cycle fatigue behaviour of micro alloyed E450 steels is not studied. Therefore, a comprehensive examination of the cyclic behaviour and mechanical properties correlating with the microstructure is essential. This work considered the carbon equivalent parameters for the microalloying of hot-rolled structural steels. Microstructural characteristics were investigated using advanced characterisation techniques encompassing field emission scanning electron microscopy (FESEM), electron backscatter diffraction (EBSD), and transmission electron microscopy (TEM). The fatigue damage mechanism was investigated using TEM. Cyclic behaviour with mechanical properties and detailed microstructural analysis establish a comprehensive composition, processing, microstructure and property correlation with particular emphasis on high cycle fatigue behaviour in the three microalloyed E450 structural steels, providing insights into the factors controlling its mechanical and cyclic behaviour.

2. Materials and Methods

Three hot-rolled E450 structural steel samples mainly synergetic difference in Mn and C contented with the dimension of $300 \times 350 \times 12 \text{ mm}^3$ was used in the current study, hereafter referred as steel 1, steel 2 and steel 3. The all the steel is vacuum arc melted and casted at a temperature of 1550°C . The cast ingot is machined and followed by homogenisation at 950°C for 3 h. The hot rolling is performed at 1000°C temperature with 5 rolling passes followed by air cooling. The detailed processing parameters are given in our previous work (Ref 1). The impact, tensile and fatigue samples were cut from the plate using the wire cut electrical discharge machine (EDM). A schematic representation of specimen cutting scheme is provided in Fig. 1. The chemical composition of the all the steel specimen is given in Table 1. The CE of all the steels were estimated as per American Welding Society standard (AWS) as 0.395, 0.372 and 0.39 for steel 1, 2 and 3, respectively. The phase is measured using X-ray

diffractometer (XRD) with Cu source, Bragg Brentano method with a scanning step size of 0.001, wavelength of 1.5406 Å and scanning speed of 4°/min. Microstructure analysis is performed using the Optical microscope and Field emission scanning electron microscope (FESEM). Specimens were etched in a 2% Nital solution (2% nitric acid in 98% of ethanol) to reveal the microstructural constituents prior to imaging using optical and electron microscopy. Energy Dispersive Spectroscopy (EDS) was used to map the elemental distribution in the specimen. EBSD measurements were conducted using a 14 nm step size at a 70° stage tilt, ensuring high spatial resolution for quantifying grain morphology, crystallographic texture, and local misorientation. Before EBSD analysis, specimens were electropolished at a temperature of 25 °C with a constant voltage of 50 V for 20 seconds. High resolution transmission electron microscopy (HRTEM) JEOL, NEO-ARM 200F was conducted in bright field mode to analyse nano scale characteristics, including dislocation, morphology, and interlamellar spacing. The operating voltage was 120 kV. The wire EDM was used to cut 1 mm thick samples followed by manual grinding using SiC papers ranging 1500 to 2500 grade. After manually thinning the foil to a thickness of $60 \pm 5 \mu\text{m}$, a sample with a diameter of 3 mm was punched out of the foil. Thinning was carried out using the Gatan PIPS-II (Precision Ion Polishing System) MODEL-695, which was utilized for milling at a voltage of 5.00 keV under dual modulation with a chamber pressure of 8×10^{-5} torr in an argon atmosphere until the hole was made in the sample. changing the gun's angle from five to two degrees. Finally, Solarus II Plasma Cleaner MODEL-955 was used to clean the samples in the argon gas was environment. Tensile testing was conducted utilising a Walter + Bai universal testing machine in accordance with ASTM E8 standard with a constant displacement rate of 1 mm/min, and equivalent strain rate of $5.21 \times 10^{-4} \text{ s}^{-1}$ (Ref 27). Microhardness measurements were performed using an Innova Test microhardness tester. A load of 200 gf with a dwell time of 30 s was applied, and indentations were made at ten different locations and average value was reported. Impact

toughness was measured using Charpy impact tester in accordance with the ASTM E23 standard (Ref 28). A servo-hydraulic universal testing system Walter + Bai, 100 kN was used to conduct high-cycle fatigue (HCF) studies under fully reversed loading conditions ($R = -1$), frequency of 15 Hz and laboratory temperature was 22°C under load control environment ensuring accurate control of stress amplitude and cyclic loading parameters following ASTM E466 (Ref 29). FESEM was used to analyse the fracture surfaces of failed specimens in order to determine the crack nucleation, propagation, and sites of final fracture. The failed fatigue specimens post fatigue test was sectioned parallel to the loading axis for bright field TEM analysis to elucidate deformation mechanism.

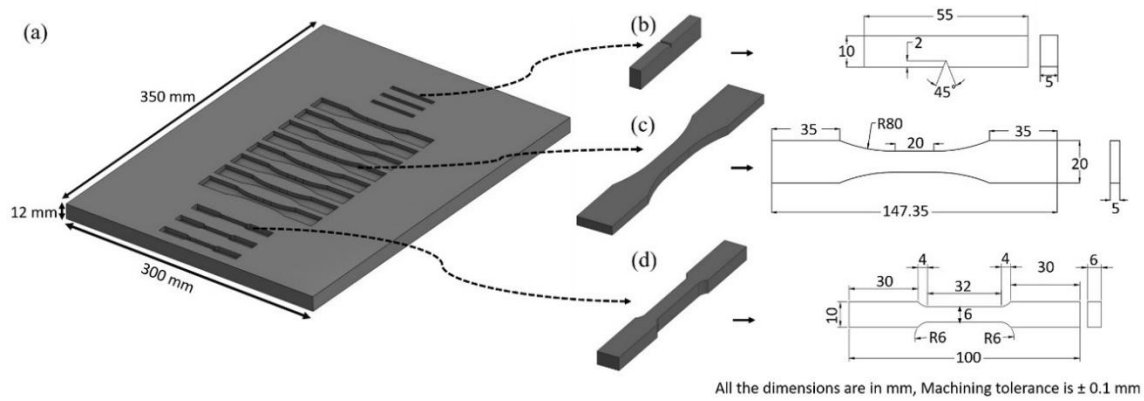


Fig. 1 Schematic representation of (a) E450 structural steel plates and (b) impact test specimen (c) fatigue test specimen and (d) tensile test specimen.

Table. 1 Chemical composition of steel 1, steel 2 and steel 3, respectively (Ref 1).

Material	Composition (wt.%)								
	Mn	Si	C	Nb	P	S	V	Al	Fe
Steel 1	1.58	0.19	0.10	0.05	0.04	0.03	0.03	0.04	Bal.
Steel 2	1.18	0.21	0.14	0.04	0.03	0.01	0.02	0.02	Bal.
Steel 3	0.97	0.23	0.19	0.04	0.04	0.03	0.03	0.02	Bal.

3. Results and Discussions

3.1 Phase

The X-ray diffraction results of all three steels are shown in the Fig. 2. The observed diffraction peaks were matched to standard reference patterns from the International Centre for Diffraction Data (ICDD), using JCPDS card 04-0140-0360. All three steels reveal the BCC structure with strong intensity along 110 planes at 2θ of 44.20° , 44.57° and 44.22° for steel 1, 2 and 3, respectively. The inter planner spacing (d) is calculated as 2.048, 2.032, and 2.047 Å for steel 1, 2 and 3, correspondingly. With prominent reflections corresponding to the α -Fe (110), (200), (211), (220) and (310) planes (Ref 30). The averaged BCC lattice parameters (a) from all the reflections are calculated as 0.2883 ± 0.0008 , 0.289 ± 0.0002 , and 0.2881 ± 0.0009 nm for steel 1, 2 and 3, correspondingly. The diffraction peaks were fitted using a gaussian function after background subtraction. Instrumental broadening was determined by measuring the standard reference material SRM 660c, consisting of LaB₆ powder, under the same experimental conditions used for samples. The instrumental broadening ($\beta_{\text{instrument}}$) is determined as 0.08° . The FWHM (β_{measured}) is estimated as $0.3887^\circ \pm 0.0006^\circ$, $0.2511^\circ \pm 0.001^\circ$ and $0.2584^\circ \pm 0.0006^\circ$ for the steel 1, 2, and 3, respectively. The crystallite size (D) is estimated using the Scherrer equation $D = \frac{K\lambda}{\beta \cos \theta}$ (Ref 31); where, K is shape factor taken as 0.94, λ is the wavelength and $\beta_{\text{corrected}} = \sqrt{\beta_{\text{measured}}^2 - \beta_{\text{instrument}}^2}$, and θ is the bragg angle. The average crystallite size is calculated from all the reflection as 16.68 ± 4.13 nm, 24.52 ± 7.64 nm and 25.59 ± 6.75 nm. Microstrain (ϵ) is calculated using the Williamson Hall equation $\beta \cos \theta = \frac{K\lambda}{D} + 4\epsilon \sin \theta$ (Ref 32) by plotting $Y = \beta \cos \theta$, Vs $X = 4 \sin \theta$ and linear regression was used with goodness of fit (R^2) as, 0.76, 0.86, and 0.82. The microstrain is estimated as, $2.20 \pm 0.72 \times 10^{-3}$, $1.91 \pm 0.043 \times 10^{-3}$ and $1.67 \pm 0.045 \times 10^{-3}$ for steel 1, steel 2, and steel 3 respectively. In addition,

the dislocation density (δ) is estimated using Williamson smallman relation $\delta = \frac{1}{d^2}$ (Ref 32) and found to be $2.55 \times 10^{15}/\text{m}^2$, $1.12 \times 10^{15}/\text{m}^2$, and $1.07 \times 10^{15}/\text{m}^2$ for steel 1, 2, and 3, respectively. Among the three constructional steels, steel 1 has the lowest crystallite size, and higher microstrain and higher dislocation density results in peak broadening as evident by FWHM results. The increased in the lattice microstrain suggested a larger density of crystal defects and residual lattice distortions, contributing to additional strengthening through dislocation interactions in steel 1.

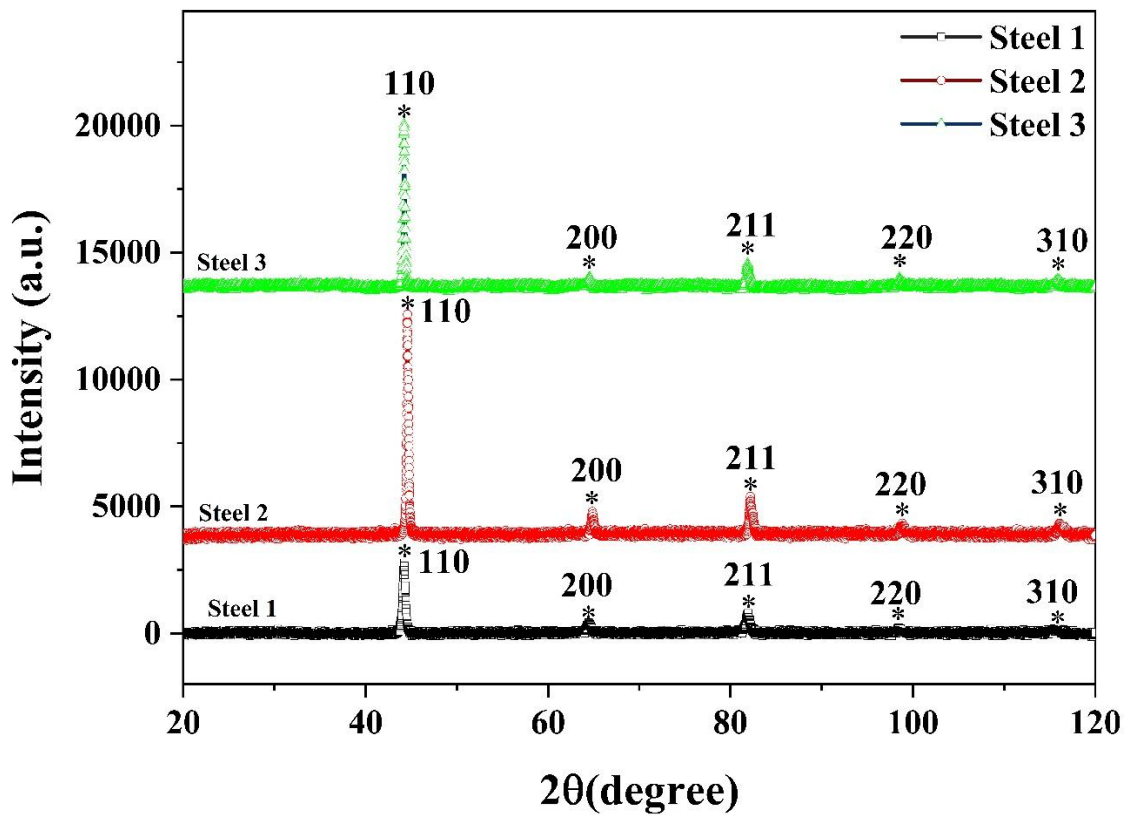


Fig. 2 X-ray diffraction graph of steel 1, steel 2 and steel 3.

3.2 Microstructure

Figures 3 (a), (b) and (c) shows the microstructure using optical microstructure and Figs. 3 (d), (e) and (f) shows the microstructure using FESEM of steel 1, 2 and 3, respectively. Steel 1 exhibits a predominantly fine ferrite microstructure, accompanied by discrete globular pearlite

located along the grain boundaries (Figs. 3 (a) and (d)). In contrast, steel 2 shows a microstructure composed of ferrite together with blocky regions of fine lamellar pearlite (Figs. 3 (b) and (e)). Steel 3 is characterised by a mixture of ferrite and comparatively coarse lamellar pearlite (Figs. 3 (c) and (f)). The pearlite phase consists of alternating lamellae of ferrite and cementite (Fe_3C) (Ref 33). Grain size measurements were conducted via the line-intercept method. The average grain size measured as $5.32 \pm 0.3 \mu\text{m}$, $8.57 \pm 0.2 \mu\text{m}$, and $10.98 \pm 0.8 \mu\text{m}$ for steels 1, 2, and 3, respectively. Grain size measurement results confirm that steel 1 features a fine grain structure, and steel 2 exhibits intermediate grain size, while steel 3 displays a coarse grain structure. The phase fraction quantification was performed with ImageJ software. Steel 1 consist of 92.67% ferrite and 7.33% globular pearlite; steel 2 contains 77.02% ferrite and 22.98% fine lamellar pearlite; steel 3 consists of 65.97% ferrite and 34.03% pearlite. The alternating lamellar structure of ferrite and cementite in steel 2 and steel 3 decides interlamellar spacing a crucial microstructural parameter influencing the mechanical properties of pearlitic steels (Ref 3). The interlamellar spacing of pearlite was calculated using the method adopted by Underwood (Ref 34). The interlamellar spacing was estimated as $132 \pm 18 \text{ nm}$, and $233 \pm 25 \text{ nm}$ for steels 2, and 3, respectively. Steel 2 exhibits the ~ 1.76 times lower pearlitic interlamellar spacing, while steel 3 displays higher pearlitic interlamellar spacing. The EDS elemental map for each of the three steels is displayed in Figure 4. The EDS elemental mapping verifies that all three steels contain Fe, Mn, C, Si, P, and S, which is in line with their designation as low-alloy carbon steels. While Mn and Si are evenly dispersed, indicating their formation of solid solution, Fe forms the dominant matrix.

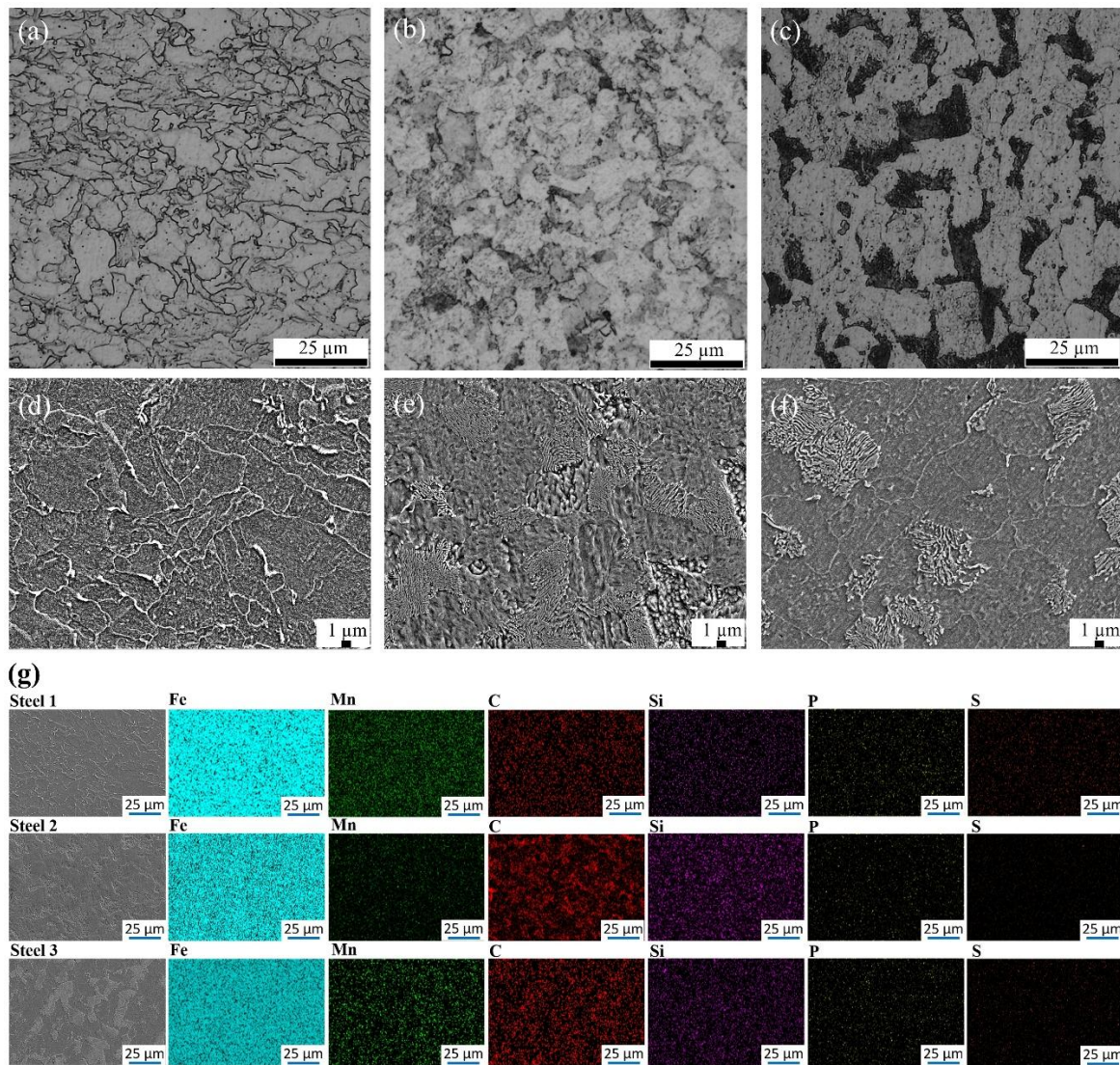
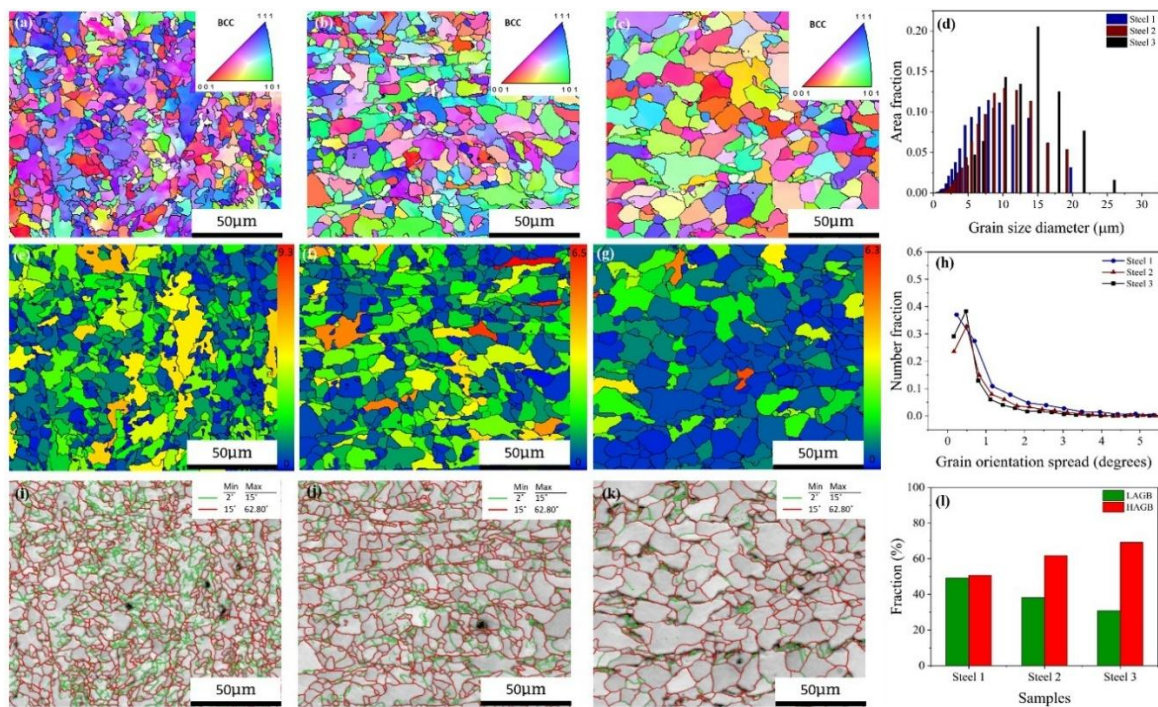


Fig. 3 Microstructure examination (a), (b) and (c) are the optical microscopy images and (d), (e) and (f) are the FESEM images and (g) EDS mapping of steel 1, steel 2 and steel 3, respectively

The EBSD analysis is performed using the OIM V8 software with a scan dimension of $150 \times 125 \mu\text{m}^2$, scanning step size of 14 nm and 3 distinct field analysed per steel. The accelerating voltage was 20 Kv and probe current was 7 nA. During the cleanup operation CI tolerance of 5.0 was applied, with a minimum grain size of 2 pixels and Multi Row set to 0. Subsequently, neighbour confidence index (CI) Correlation was performed to improve the spatial consistency of the CI

values between neighbouring measurement points. A minimum CI threshold of 0.15 was used with a single iteration. Kernel Average Misorientation (KAM) maps were calculated using the first-neighbor kernel with a maximum misorientation angle threshold of 5° . Phase indexing was performed assuming a body centered cubic (BCC), $Im-3m$ crystal structure. The EBSD analysis provides detailed insight into the crystallographic texture and microstructural refinement of steels 1, 2, and 3, as shown in the inverse pole figure (IPF) maps in Figs. 4(a), (b) and (c), respectively. The grain size distributions illustrated in Fig. 4(d) indicating that steel 1 exhibits the finest grain distribution, followed by steels 2 and 3. Steel 1 reveals a finer grain size than steels 2 and 3 owing to its manganese content and lower carbon level. The grain orientation spread (GOS) maps of steel 1, 2 and 3 is shown in Figs. 4(e), (f) and (g), respectively. Steel 1 exhibits the higher GOS values followed by steel 2 and steel 3 as shown in Fig. 4 (d). GOS indicate significant variations in intragranular deformation and stored energy across the steels. Steel 1 demonstrates elevated GOS values, signifying more strained grains, which aligns with its predominant ferrite and globular pearlite structure as shown in Fig. 4 (d). Moreover, steels 2 exhibits intermediate GOS values comprising of fine lamellar pearlite with ferrite grains, indicative of decreased local misorientation and dislocation density in contrast to steel 1. Steel 3 reveals the lower GOS among all three steels consist of coarse lamellar pearlite and ferrite correlated to lower local misorientation and dislocation density in contrast to steel 1 and 2. The grain boundary maps of steel 1, steel 2 and steel 3 are illustrated in Figs. 4 (i), (j) and (k), respectively. The angular resolution threshold was 0.05° . Figure. 4 (l) shows a low angle grain boundary (LAGB ranges $2^\circ - 15^\circ$) and high angle grain boundary (HAGB ranges $15^\circ - 62.80^\circ$) distribution map. Steel 1 shows 46.6% LAGB and 53.4% HAGB, while steel 2 shows 37.1% LAGB and 62.9% HAGB, and steel 3 shows 29.4% LAGB and 70.6% HAGB. Steel 1 shows the highest fraction of LAGB among all the steels, indicating the existence of a larger number of subgrains and a higher dislocation density. This observation is

in close agreement with the XRD measurements, which likewise demonstrated the maximum dislocation density for steel 1. The increased dislocation density and subgrain structure effectively impede motion of dislocation, therefore leading to greater yield strength and higher microhardness through dislocation and subgrain strengthening mechanisms. However, steel 3 exhibits the lowest LAGB proportion, which suggests lower subgrains formation by deformation and a lower dislocation density attributed to a comparatively coarser grain and less strained microstructure, which is expected to reduce dislocation strengthening and promote plastic deformation.



Figs. 4 EBSD analysis results (a), (b) and (c) are crystallographic orientation map; (d) grain size diameter distribution; (e), (f) and (g) are grain orientation spread (GOS) map; and (h) is the GOS graph (i), (j) and (k) are the LAGB and HAGB map; (l) is the LAGB and HAGB distribution of steel 1, steel 2 and steel 3, respectively.

The grain average misorientation (GAM), kernel average misorientation (KAM), and Taylor factor was investigated to understand of the deformation substructure and local lattice resistance in steels 1, 2, and 3 using EBSD. Figures 5 (a), (e), and (i) show the GAM map,

while Figures 5 (b), (f), and (j) show the KAM map. Figures 5 (d) and (h) represent the GAM and KAM distribution graphs for all three steels. Figures. 5 (m), (n) and (o) shows the pole figure of steel 1, 2 and 3, respectively. Steel 1 display the highest average GAM value ($0.61 \pm 0.33^\circ$) and average KAM values ($0.62 \pm 0.44^\circ$) among all the steels, indicating significant intragranular misorientation. This finding consistent with the observation that ferrite dominated microstructures exhibit a high density of LAGB leading to larger local misorientation (Ref 1). Steel 2 exhibits intermediate average GAM value (0.52 ± 0.34) and average KAM values ($0.44 \pm 0.37^\circ$) revealing of its mixed ferrite and fine lamellar pearlite microstructure. Steel 3 exhibits the lowest average GAM value ($0.45 \pm 0.34^\circ$) and KAM values ($0.32 \pm 0.29^\circ$) among all the steels. Figures 5 (c), (g), and (k) show the Taylor factor maps for steels 1, 2, and 3, respectively, whereas Figure 5 (l) show the grain average image quality graph for all the steels. Taylor factor is a measure of a polycrystalline BCC material to facilitate slip (Ref 9). The BCC slip systems, namely the $\{110\}\langle 111 \rangle$, $\{112\}\langle 111 \rangle$, and $\{123\}\langle 111 \rangle$ families are assumed for the Taylor factor calculations. The possible slip directions were assumed along the $\langle 111 \rangle$ directions. A lower Taylor factor signifies easier plastic deformation, however an increase in Taylor factor hinders the activation of slip mechanisms (Ref 9). Steel 1 has a higher Taylor factor value (~ 3.6) followed by steels 2 and 3 exhibits a Taylor factor of ~ 3.2 and ~ 2.9 , respectively indicating prone to slip activation. The findings reveal that Steel 1 is the higher resistant to deformation while steel 3 poses lower resistant. The pole figures for the crystallographic planes $\{001\}$, $\{111\}$, and $\{110\}$ show that the crystallographic texture of all three steels is randomly oriented, and no clear preferred grain orientation is observed. However, there were minor differences in texture intensity between the samples is found. Steel 1 exhibits the poor texture intensity (1.873), while steel 2 poses the highest maximum texture intensity (2.509), closely followed by steel 3 (2.504). Steel 2 higher maximum texture intensity attributed to improved alignment of grains along preferred orientation in contrast to steel 1.

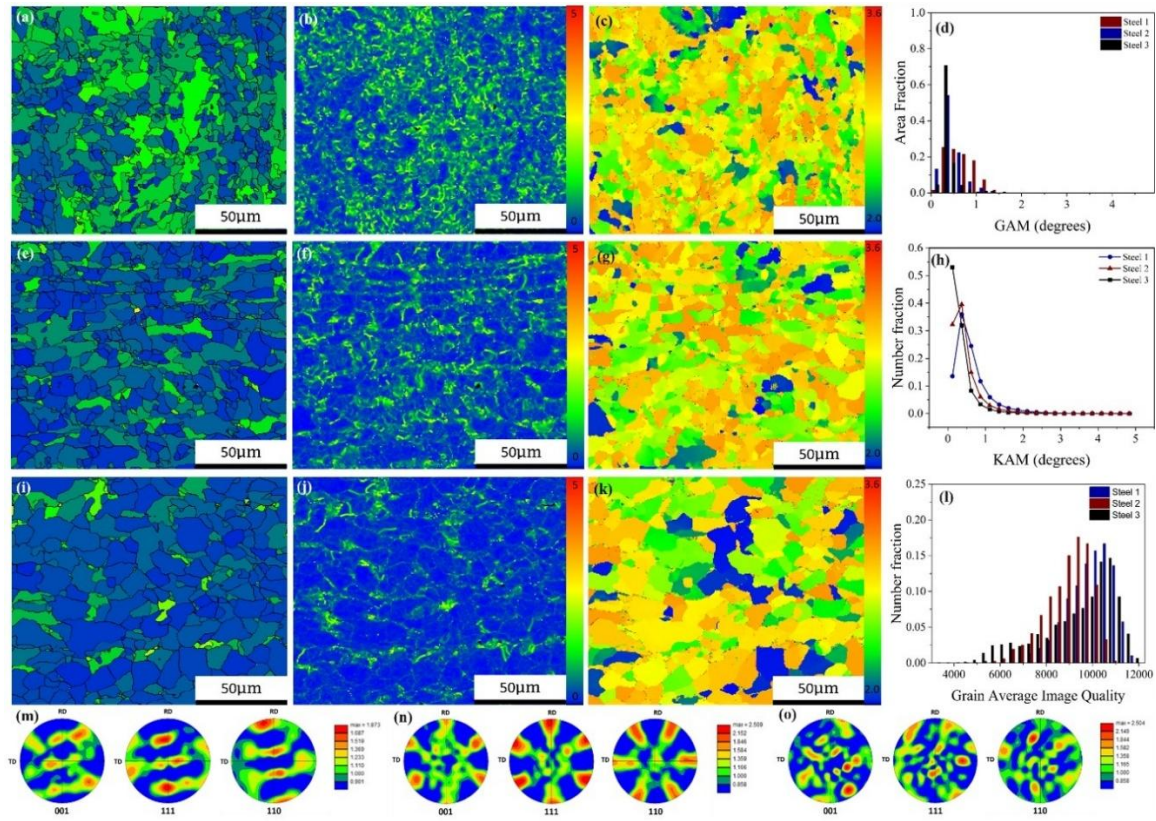


Fig. 5 EBSD analysis results (a), (e) and (i) are GAM map; (b), (f), (j) are KAM map; (c), (g) and (k) are Taylor factor; (m), (n) and (o) are the pole figures of steel 1, steel 2 and steel 3, respectively; (d), (h) and (l) is the GAM, KAM, and GAIQ distribution graph of all steels together.

Figures 6 (a), (b), and (c) show bright-field TEM images of steels 1, 2, and 3, respectively. The TEM foils of dimensions $6 \times 6 \times 1 \text{ mm}^3$ are extracted 2 mm away parallel to the rolling direction. In addition, Figs. 6 (d), (e) and (f) show the magnified microstructural features. Steel 1 exhibits a refined ferritic matrix with globular pearlite distributed along the grain boundaries as shown in Fig. 6 (a) and (d). This globular morphology, combined with the fine ferrite grains, enhances strengthening by Hall–Petch effect and therefore contributes to the higher YS and UTS of steel 1. The relatively fine scale of these lamellae pearlite is promoted by steel 2's synergetic combination of carbon and slightly higher manganese concentration, which retards lamellar coarsening during cooling and hot working by reducing diffusion rates and stabilising a finer

pearlite morphology as shown in Figs. 6 (b) and (e) (Ref 35). The coarsening of pearlite in steel 3 aligns with its reduced manganese content and increased carbon concentration, both of which accelerate the formation of thicker pearlite lamellae and diminish nucleation density during phase transformation as shown in Figs 6 (c) and (f) (Ref 36). The selected area diffraction (SAED) pattern of steel 1, steel 2 and steel 3 is shown in Figs. 6 (g), (h) and (i), respectively. The SAED pattern of steel 1 exhibits innermost shell with a hexagonal array of six spots at 60° intervals, with the second shell positioned at a radius ratio of $\sqrt{3}$ relative to the first and rotated 30° exhibits the characteristic of BCC phase. The experimentally measured interplanar spacings are 0.202, 0.117, 0.101 and 0.0765 nm, respectively, which are in close agreement with the theoretical spacings of BCC ferrite 0.2027, 0.1170, 0.0906 and 0.0766 corresponding to (110), (211), (220) and (321) planes of BCC α -ferrite phase with zone axis [001]. The error in the measured values lies within 0.1 to 0.5% of the theoretical value. However, no clear diffraction reflections attributable exclusively to orthorhombic cementite (Fe_3C) are identified in steel 1. The SAED pattern of steel 2 exhibits distinct diffraction spots corresponding mainly to the orthorhombic cementite phase with zone axis [113] as shown in Fig. 6 (b). The interplanar spacing is determined as 0.239, 0.141 and 0.144 nm are in close agreement with the theoretical interplanar spacings of Fe_3C are 0.244, 0.139 and 0.140 nm are indexed to (110), ($2\bar{1}1$) and ($3\bar{0}1$) planes of Fe_3C along with [113] zone axis. Furthermore, the SAED pattern of steel 3 reveals a spotty ring pattern rather than a single crystal zone axis pattern. It exhibits orthorhombic cementite phase with measured interplanar spacing of 0.495, 0.280, 0.262, 0.245, 0.236, 0.226, and 0.224 nm indexed as (100), (120), (200), (121), (210), (002), and (030) planes, with a mean deviation of 1.4%. However, ferrite (BCC) reflection is not observed in steel 3.

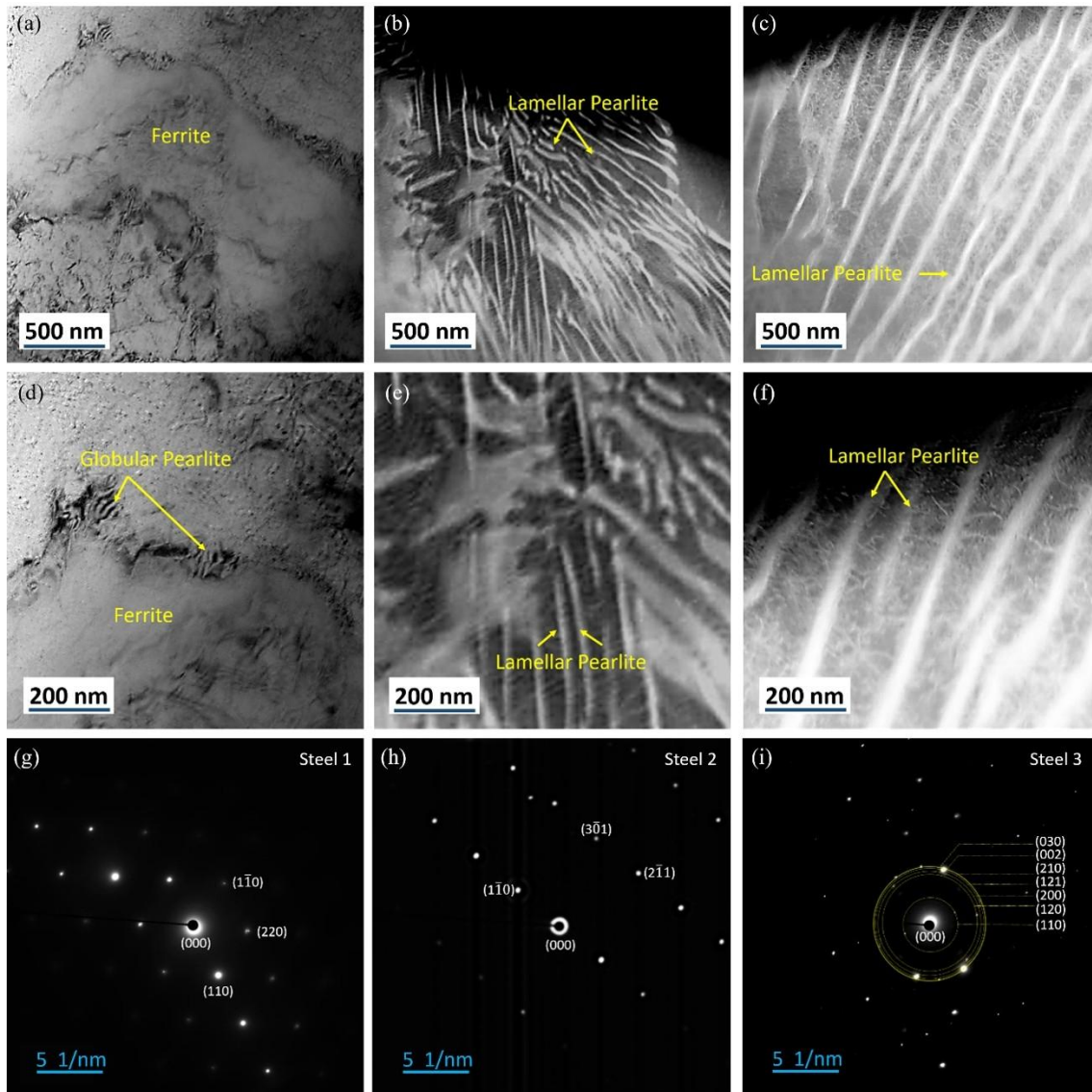


Fig. 6 TEM bright field images (a), (b) and (c); (d), (e), (f) are magnified images and (g), (h) and (i) are the selected area electron diffraction (SAED) pattern of steel 1, steel 2 and steel 3, respectively.

3.3 Mechanical properties

The tensile stress strain plateau and impact energy graph for all steel samples are presented in Figs. 7 (a) and (b), respectively. Tensile and impact tests were conducted on three specimens for each steel composition, and the average values are reported. The gauge length of the tensile specimen was 32 ± 0.1 mm with a 10 ± 0.1 mm width and 5 ± 0.1 mm thickness. Steel 1 shows a higher YS of 582 ± 14 MPa, and UTS of 662 ± 18 MPa along with $18.35 \pm 2.5\%$ elongation

among all the steels attributed due to fine ferrite microstructure with globular pearlite along grain boundaries, refined grains, a higher GAM, Taylor factor, and elevated KAM values as shown in Fig. 7 (a). The comparatively reduced elongation of $18.35 \pm 2.5\%$ in steel 1 line up with the EBSD results. The higher GAM and KAM values revealed by EBSD indicate substantial intragranular strain accumulation, increasing strength while reducing ductility (Ref 37). Conversely, steel 3 shows YS of 458 ± 16 MPa and an UTS of 520 ± 20 MPa due to coarse lamellar pearlite within ferrite with lower GAM and KAM values and higher interlamellar spacing revealed by FESEM, EBSD and TEM analysis. Nevertheless, the coarser lamellar pearlite and larger interlamellar spacing favours interlamellar slip leading to the highest elongation of $26.89 \pm 2.23\%$ among the three steels. Besides, steel 2 exhibit fine pearlite needles with 1.97 times lower interlamellar spacing than steel 3 attributed to higher YS of 500 ± 14 MPa and an UTS of 590 ± 16 MPa, with an elongation of $23.20 \pm 2.0\%$. Intermediate grain size, GAM and KAM values with lower interlamellar spacing promotes strengthening without significantly decrease in ductility.

Impact toughness test was performed on the 3 samples of each steel, and the average value was reported. The impact specimen has a length of 55 ± 0.1 mm with 10 ± 0.1 mm width and 5 ± 0.1 mm thickness. A 45° V shaped notch-oriented transverse to the rolling direction with a depth of 2 mm was cut at the centre of each specimen using wire EDM. The impact test was performed at the laboratory temperature of 22°C . The impact test results are illustrated in Fig. 7 (b). Steel 1 demonstrates the highest impact energy of 61.51 ± 2.1 J, due to the synergetic effect of refined ferritic grains and globular pearlite. Steel 2 demonstrates a slightly decreased impact energy of 58.58 ± 1.52 J attributed due to fine lamellar pearlite and lower interlamellar spacing. Steel 3 exhibits lower impact energy of 38.06 ± 1.19 J among all the steels due to its coarse pearlitic lamellae which serve as preferential fracture paths and weaken capacity to absorb impact energy. The microhardness test was performed at the 10 distinct points, and the

average value was reported. Steel 3 demonstrates the highest hardness value of $280 \pm 6 \text{ HV}_{0.2}$ attributed due to the higher carbon content, while steel 1 presents the lowest hardness value of $209 \pm 5 \text{ HV}_{0.2}$ owing lower carbon content. Steel 2 demonstrate an intermediate hardness value of $265 \pm 6 \text{ HV}_{0.2}$ due to synergetic combination of carbon and manganese.

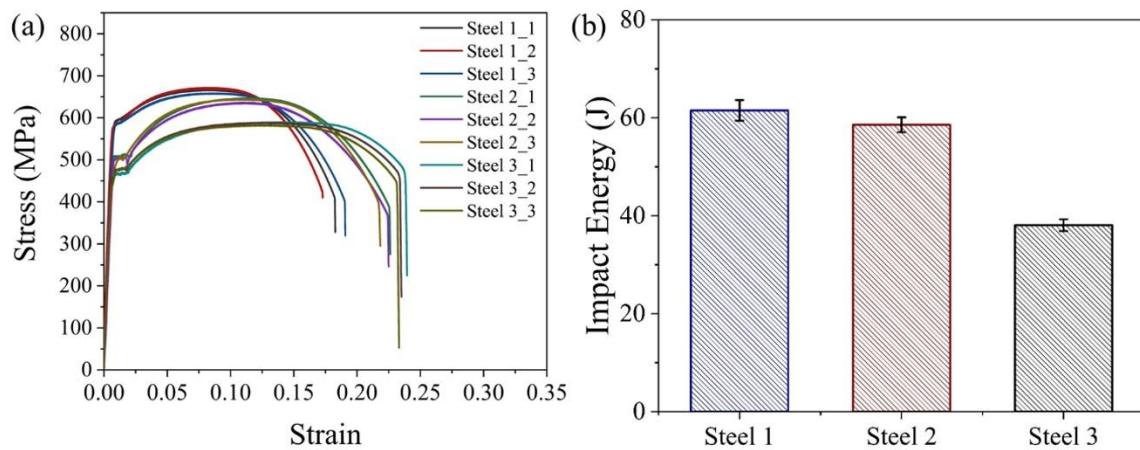


Fig. 7 shows the graph of (a) stress Vs strain plateau and (b) impact energy graph of steel 1, steel 2 and steel 3, respectively.

The fractography of the tensile tested specimens is presented in Figs. 8 (a), (b), and (c) for steel 1, 2, and 3, respectively. Figures. 8 (d), (e) and (f) represent the enlarged view of steels 1, 2, and 3, respectively. Fractography image reveals predominantly ductile fracture behaviour in all the steels. The presence of equiaxed dimples in each steel is a feature of ductile failure as the main fracture mechanism. Steel 1 has a substantially finer and more dispersed dimple distribution, in accordance with its finer ferritic grains, globular pearlite and greater dislocation density which enhance homogenous plastic deformation before fracture. On the other hand, steel 2 shows moderate dimple size correlated to its finer pearlitic structure. The comparatively bigger dimples in steel 3 compared to steels 1 and 2 indicate a higher degree of local plastic deformation and are correlated with higher elongation observed.

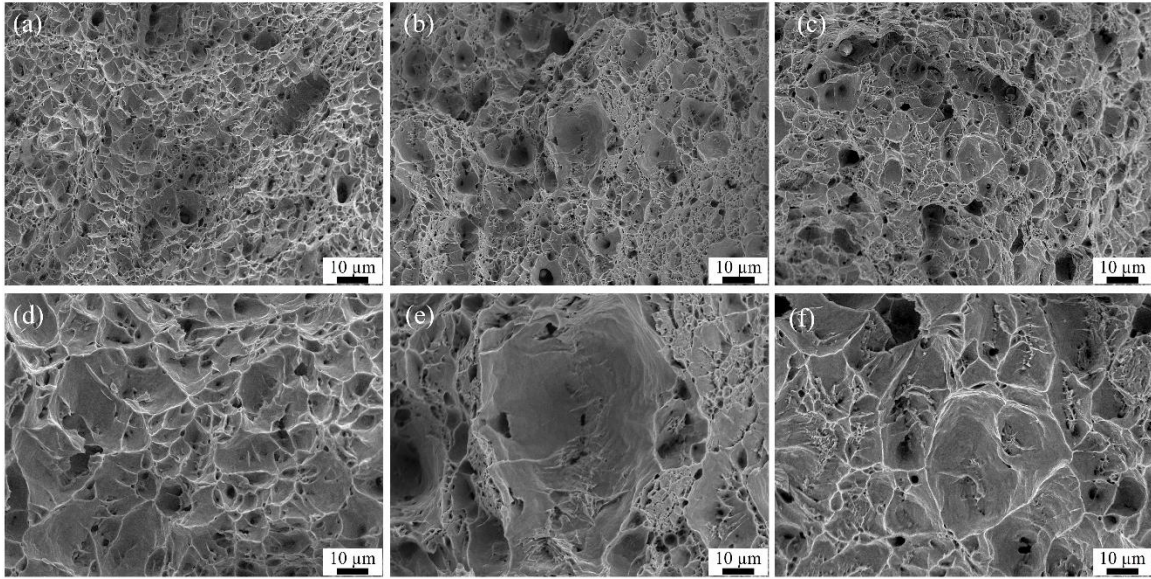


Fig. 8 shows the tensile test fractography of (a) steel 1, (b) steel 2 and (c) steel 3 and enlarged view (d) steel 1, (e) steel 2 and (f) steel 3.

3.4 Fatigue behaviour

The fatigue specimens had an overall length of 147.35 ± 0.1 mm, a width of 20 ± 0.1 mm, and a thickness of 5 ± 0.1 mm. The initial surface roughness of the machined fatigue specimen was $R_a = 0.3 \pm 0.02$ μm . No additional surface finishing was performed. Fatigue test is carried out up to 10^6 cycles or until complete fracture of the specimen, whichever transpired first. Samples that survived 10^6 cycles without failure were designated as runout specimens. The criterion used to define failure is complete fracture of the test specimen. Three samples were tested at each stress amplitude under the stress ratio ($R = -1$). The fatigue samples longitudinal axis was parallel to the rolling direction. Steel 1 was tested at nine stress amplitude levels: 464, 435, 406, 348, 290, 275.5, 261, 246.5, and 232 MPa. Steel 2 was tested at ten stress amplitude levels: 400, 375, 350, 325, 312.5, 300, 286, 275, 262.5, and 250 MPa steel 3 was tested at eight stress amplitude levels: 354.6, 325.05, 295.5, 265.95, 236.4, 221.62, 212.76, and 206.85 MPa. A sinusoidal waveform is used for the all the steels samples. The fatigue life of steels 1, 2 and 3

was estimated using stress-life (SN) curve, while experimental data fitted using Basquin's law $\sigma_a = \sigma'_f (2N_f)^b$, where, b is the fatigue strength exponent, σ'_f is fatigue strength coefficient and N_f is number of cycles to failure as shown in Figs. 9 (a), (b) and (c) accordingly. As shown in Fig. 9 (d), the Basquin's prediction fatigue strengths of steel 1, steel 2 and steel 3 at 10^6 cycles are 252 MPa, 286 MPa and 226 MPa at 10^6 cycles, respectively. The reported fatigue strength values are extrapolated. The goodness of fit R^2 is determined as 0.83, 0.99 and 0.99 for steel 1, 2 and 3, respectively. The 95% confidence intervals were calculated for the fitted parameters and for the predicted fatigue strength at 10^6 cycles. The lower limit of the 95% of CI is 225, 285, and 223 MPa, and the upper limit of the 95% CI is 282, 288, and 228 MPa for steel 1, 2, and 3, respectively. The Basquin exponent b was -0.1388 ± 0.031 , -0.059 ± 0.002 and -0.067 ± 0.002 for steel 1, 2 and 3, respectively. Predicted fatigue strength (MPa) at 10^6 cycles of all three steels and 95% confidence intervals (CI) of fatigue strength (MPa) and goodness of fit is given in Table. 2. Furthermore, the normalised fatigue strength is evaluated by taking the ratio of fatigue strength to ultimate tensile strength of the respective steel. The normalized fatigue strengths of steel 1, 2, and 3 is calculated as 0.381, 0.485, and 0.434, corresponding to 38.07%, 48.52%, and 43.39% of their UTS values. Although Steel 1 shows the highest UTS of 662 MPa, its normalized fatigue strength was the lowest as 38.07 % of its UTS value. In contrast, Steel 2 exhibited the highest normalized fatigue strength of 48.52% of its UTS value, indicating superior fatigue resistance. Steel 3 showed an intermediate normalized fatigue strength of 43.39% of its UTS value. Steel 1 exhibits a longer fatigue life at a stress amplitude of 350 MPa. Steel 1 and steel 2 survived 1.49×10^5 cycles and 2.48×10^4 cycles, respectively at a stress amplitude of 350 MPa, whereas steel 3 withstood until 1.6×10^3 cycles. The maximum fatigue life of 1×10^6 cycles are found for Steel 2 at a stress amplitude of 286 MPa, then Steel 1 at 4.41×10^5 cycles and steel 3 at 2.73×10^3 cycles. Steel 2 demonstrates the superior fatigue strength, attributable to its fine pearlitic microstructure and intermediate

grain size. The refined lamellar spacing improves resistance to crack formation by strengthening barriers to dislocation motion and delaying strain localisation. Moreover, Steel 2's fatigue performance is improved by its moderate grain refinement and intermediate Kernel Average Misorientation, which promote uniform plastic deformation under cyclic loading (Ref 38).

Table. 2 Predicted fatigue strength (MPa) at 10^6 cycles of all three steels and 95% confidence intervals (CI) of fatigue strength (MPa) and goodness of fit.

Samples	Fatigue strength (MPa)	95% of CI lower limit (MPa)	95% of CI Upper limit (MPa)	Goodness of fit (R^2)
Steel 1	252	225	282	0.83
Steel 2	286	285	288	0.99
Steel 3	226	223	228	0.99

Although, Steel 1 has highest KAM values and higher proportion of GAM and Taylor factor, demonstrates a lower fatigue strength compared to Steel 2. Presence of globular pearlite at grain boundaries in steel 1 is beneficial for strengthening via Hall-Petch effect, however, is considered as a preferred site for fatigue crack initiation (Ref 39). The increased intragranular strain remaining inside grains, as shown by higher KAM and GOS values, increases the risk of early microcrack generation, despite the rise in monotonic strength (Ref 40). Steel 2 shows higher fatigue life because of lower interlamellar spacing indicated to dislocation slippage (Ref 41,42). Steel 3 exhibits the lowest fatigue strength due to its coarse interlamellar pearlite spacing and coarse grain size. The coarse pearlite, characterised by broader interlamellar spacing, presents less obstacles to dislocation movement, hence facilitating fracture initiation and propagation under cyclic stress [39]. Furthermore, decreased KAM values and decreasing local misorientation signify a reduced potential for strain hardening during cyclic loading [40].

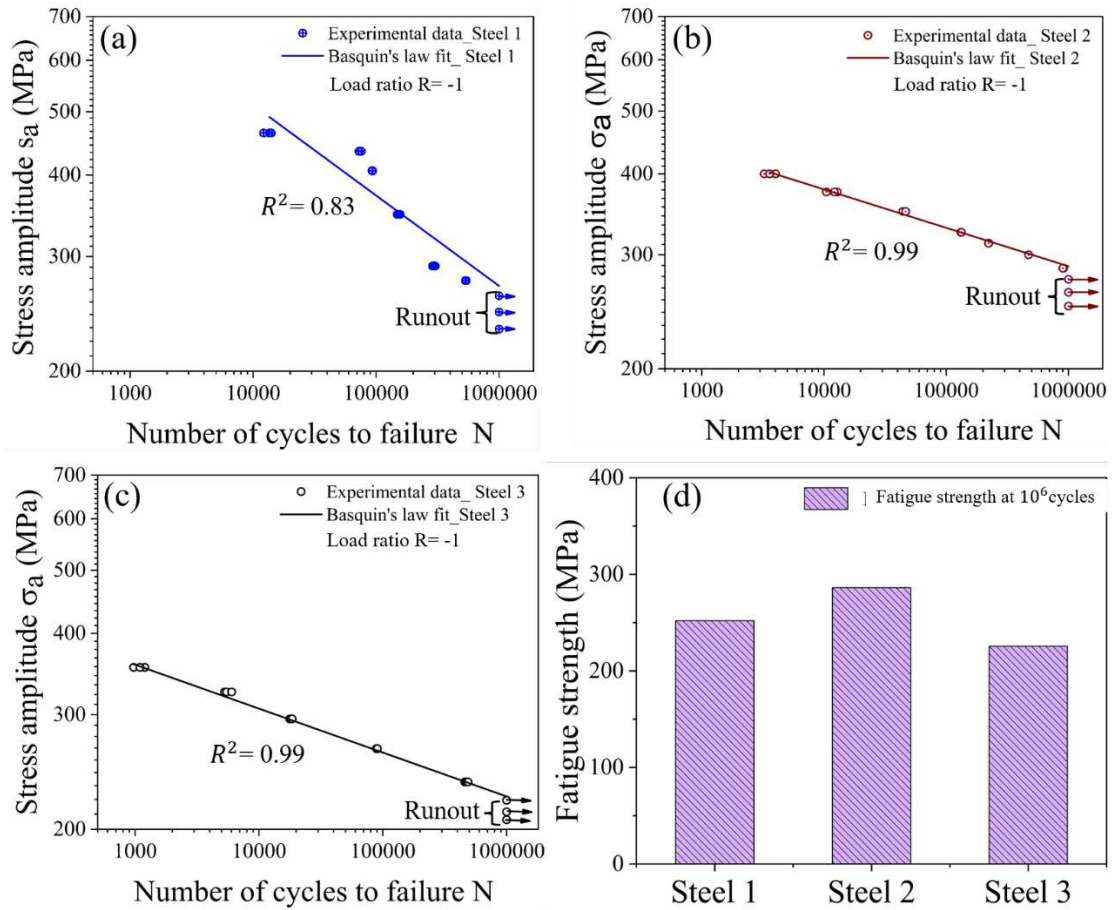


Fig. 9 Graph of stress amplitude versus number of cycle graph for (a) steel 1, (b) steel 2 (c) steel 3, and (d) fatigue strength of steel 1, steel 2 and steel 3 corresponding to 10^6 cycles.

Steel 1 sample subjected to a stress amplitude of 275.5 MPa failed after 5.32×10^5 cycles, and the analysis using FESEM is depicted in Fig. 11. Figure 10 (a) illustrates the fractography of the complete fracture surface, distinctly showing the fatigue crack start, crack propagation, and the instantaneous fracture region. Figure 10 (b) reveals several initiations of surface fatigue cracks, indicating that the nucleation of these cracks is caused by surface phenomena associated with localised stress concentrations. The existence of distinct fatigue striations in the fracture propagation area confirms consistent, incremental crack progression under high cycle loading as depicted in Fig. 10 (c). In proximity to the instantaneous fracture zone, many secondary microcracks are seen in advance of the primary crack front (Fig. 10 d), indicating localised

plastic deformation and crack coalescence. The instantaneous fracture zone (Fig. 10 e) reveals a dimple morphology, indicating ductile failure driven by microvoid nucleation, expansion, and coalescence.

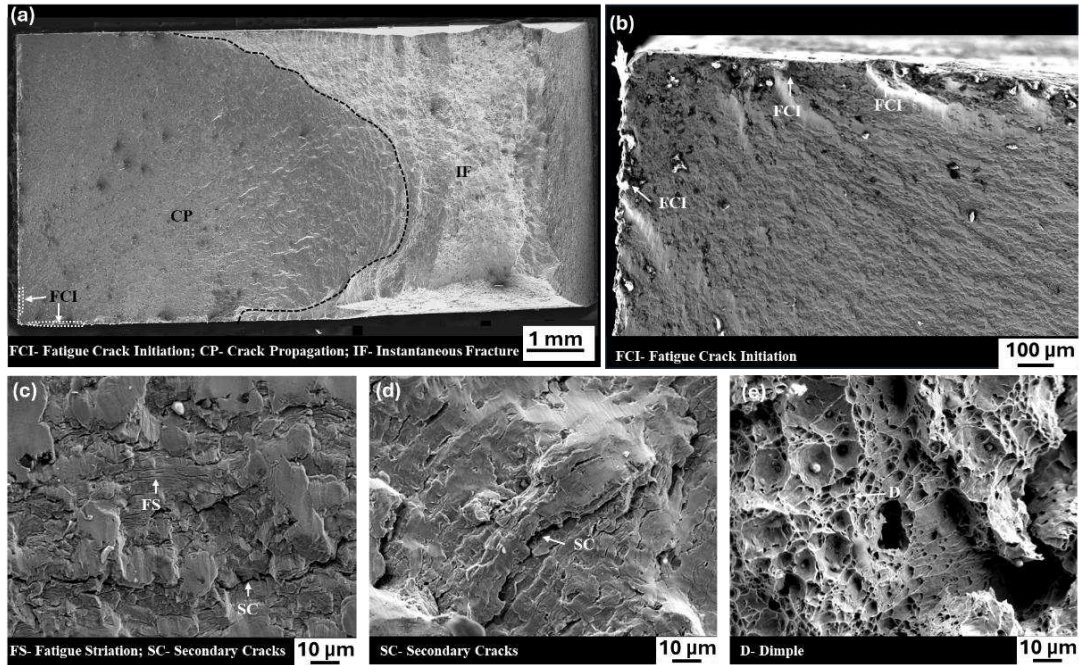


Fig. 10 Fractography of steel 1 post fatigue fracture (a) shows the image of entire fracture surface (b) shows the fatigue initiation (c) shows fatigue striation (d) shows the propagation stage with secondary cracks and (e) is instantaneous fracture region with dimples.

Similarly to steel 1, FESEM fractography of steel 2 specimen tested at a stress amplitude of 300 MPa and failed after 4.7×10^5 cycles is illustrated in Fig. 11. The overall fracture surface shown in Fig. 11 (a) reveals the fatigue crack initiation, crack propagation and instantaneous fracture zone. Figure 11 (b) reveals multiple crack-initiation sites along the surface, indicative of stress concentration and surface driven fatigue crack initiation. The presence of fatigue striations in the mid region of the crack propagation region as illustrated in Fig. 11 (c) confirms progressive, cycle by cycle crack propagation. As the crack approaches the instantaneous fracture region, numerous microcracks are observed as shown in Fig. 11 (d), reflecting localised plastic deformation and coalescence processes. Fig. 11 (e) exhibits the instantaneous

fracture region with pronounced dimple morphology, demonstrating that the final fracture occurred by ductile micro-void coalescence similar as steel 1.

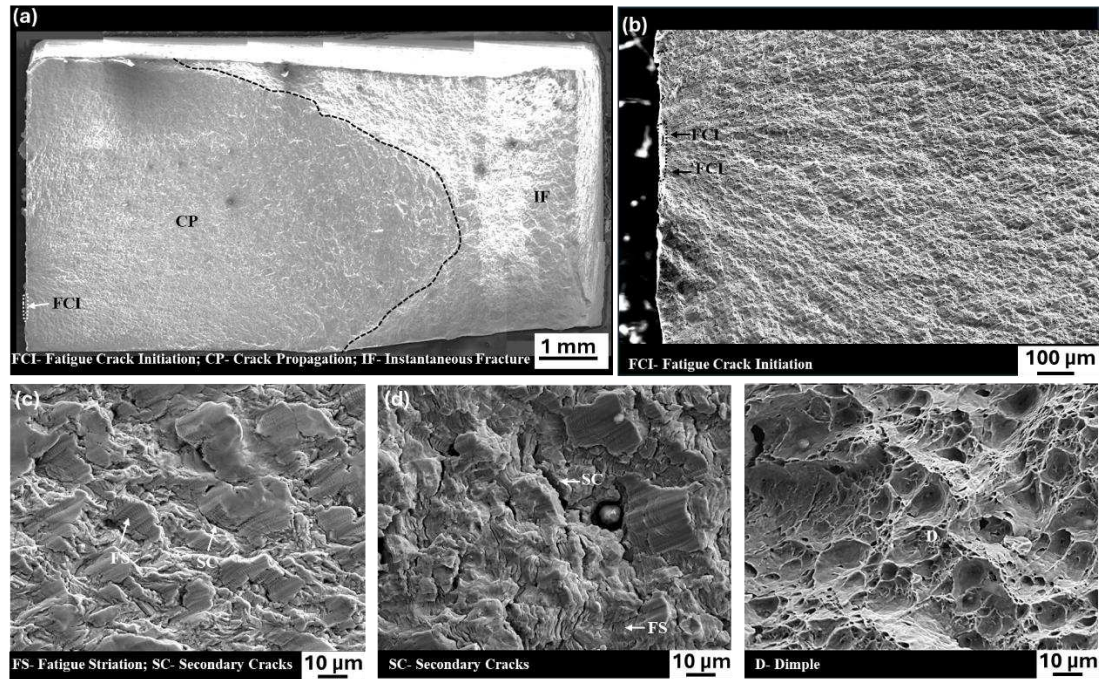


Fig. 11 Fractography of steel 2 post fatigue fracture (a) shows the image of entire fracture surface (b) shows the fatigue initiation (c) shows fatigue striation (d) shows the propagation stage with secondary cracks and (e) is instantaneous fracture region with dimples.

Likewise, FESEM fractographic examination of the steel 3 specimen, subjected to a stress amplitude of 236.4 MPa and failing after 4.8×10^5 cycles is shown in Fig. 12. Figure 12 (a) displays the overall fracture surface, which exhibits fatigue failure steps as fatigue crack initiation, crack propagation and instantaneous fracture zones. Figure 12 (b) uncovers many crack-initiation sites along the surface, signifying stress concentration and surface-induced fatigue crack initiation. The existence of pronounced fatigue striations in the crack propagation zone of the fracture surface, as seen in Fig. 12 (c), validates incremental crack propagation. As the crack approaches the instantaneous fracture zone, several microcracks are observed in front of the primary crack front in Fig. 12 (d), indicating localised plastic deformation and coalescence phenomena. The magnified depiction of the instantaneous fracture region shown

in Fig. 12 (e) reveals distinct dimple morphology, indicating that the final fracture occurred by ductile micro-void coalescence, akin to steel 1 and 2. Mostly, all three types of micro alloyed steels exhibit the multiple surface fatigue crack initiation and fatigue striation and ductile instantaneous fracture.

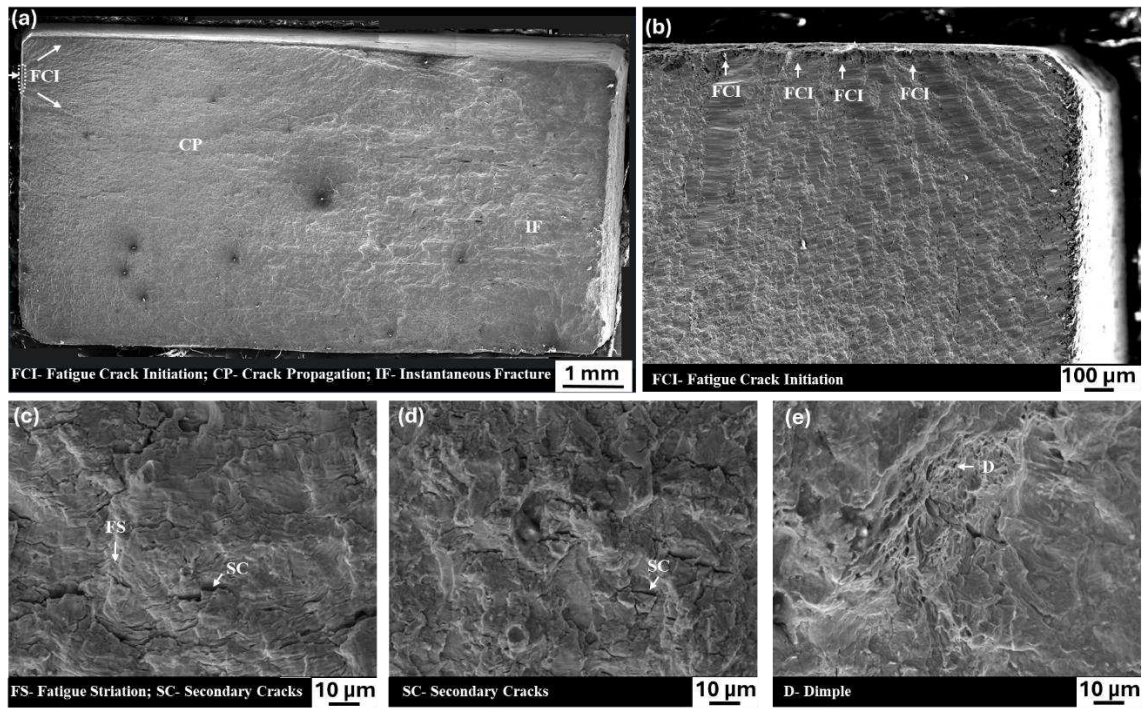


Fig. 12 Fractography of steel 3 post fatigue fracture (a) shows the image of entire fracture surface (b) shows the fatigue initiation (c) and (d) shows the propagation stage with fatigue striation and secondary cracks and (e) is final fracture region with dimples.

Bright field TEM investigation was performed on steel 1 sample subjected to a stress amplitude of 275.5 MPa failed after 5.32×10^5 cycles, steel 2 specimen tested at a stress amplitude of 300 MPa and failed after 4.7×10^5 cycles and steel 3 specimen, subjected to a stress amplitude of 236.4 MPa and failing after 4.8×10^5 cycles. The TEM foils of dimensions $6 \times 6 \times 1 \text{ mm}^3$ are extracted 2 mm away along the rolling direction from the fractured surface from all three steels specimens to understand the nanoscale deformation and damage mechanisms influencing high cycle fatigue behaviour. The sample preparation method is discussed in detail in the material and method section. The bright field pictures of steels 1, 2, and 3 shown in Figs. 13 (a), (b),

and (c), respectively exhibit distinct microstructural responses to cyclic loading, while the associated high-magnification images in Figs. 13 (d), (e), and (f) emphasised the localised deformation characteristics. Steel 1 exhibits significant grain deformation characterised by extensive dislocation at grain boundaries, indicating that cyclic loading may promote interlamellar slip buildup and localised stress concentration, hence promoting early fatigue crack initiation. In steel 2, the microstructure exhibits finer pearlitic lamellae, and the bright field TEM images show deformation in the ferrite areas with pearlitic needles, indicating that cyclic stress may be accommodated by interlamellar slip, as seen in Figs. 13 (b) and (e). Additionally, steel 3 exhibits thicker pearlite lamellae with considerable dislocation entanglement at grain boundaries, indicating limited slip transfer and increased strain localisation at hard pearlite interface.

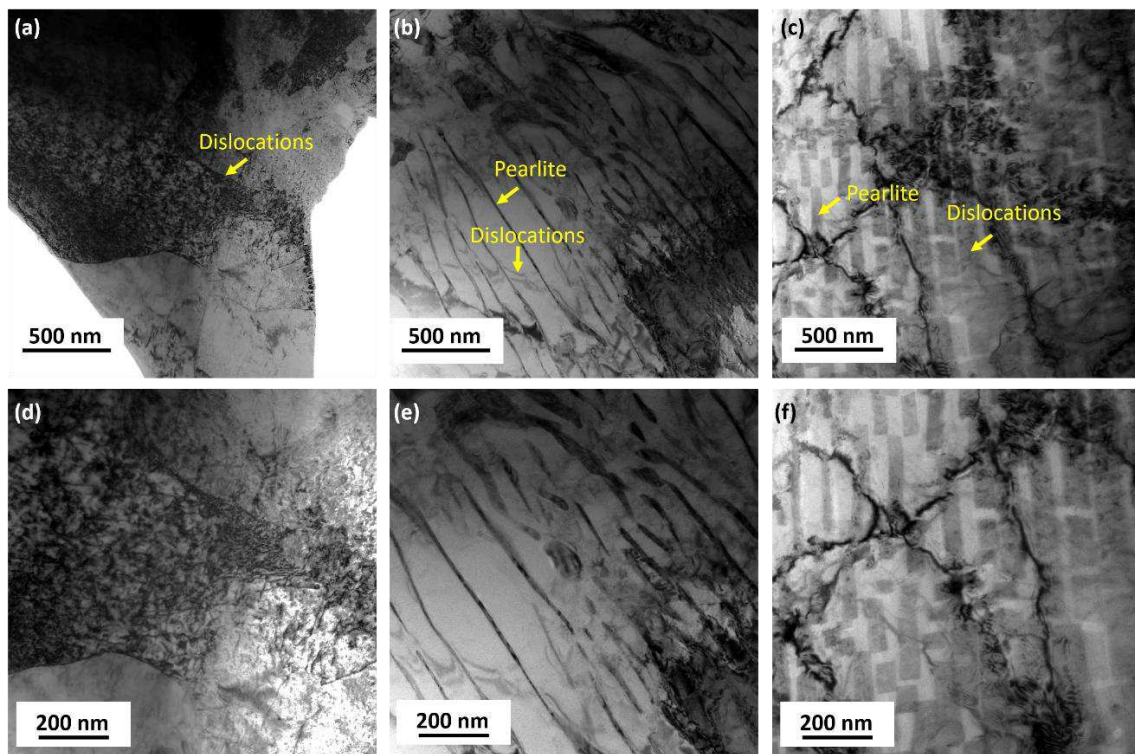


Fig. 13 (a), (c) and (e) are the bright field TEM image post fatigue and (b), (d) and (f) are the enlarged view of steel 1, 2 and 3, respectively.

4. Conclusions

Microstructure and cyclic behaviour of microalloyed E450 structural steels is studied, highlighting the influence of microstructure on fatigue life. X-ray diffraction revealed a lower crystallite size, higher microstrain and higher dislocation density in steel 1 associated with combined effect in improving yield strength and hardness value. All three steels comprise of combination of ferritic and pearlitic microstructure with globular and lamellar pearlite. Steel 1 shows higher yield strength of 582 ± 14 MPa, UTS of 662 ± 18 MPa, impact energy of 61.51 ± 2.1 J and lower elongation of $18.35 \pm 2.5\%$ and lower microhardness of 209 ± 5 HV_{0.2} attributed due to combined effect of fine grain size and lower carbon content among all the steels. However, steel 3 exhibits the higher elongation of $26.89 \pm 2.23\%$ and higher hardness of 280 ± 6 HV_{0.2} is attributed due to combined effect of coarse grain and higher carbon content, lower pearlitic interlamellar spacing and phase fraction. Steel 2 shows the intermediate yield strength of 500 ± 14 MPa, UTS of 590 ± 16 MPa with an elongation of $23.20 \pm 2\%$ and an average microhardness value of 265 ± 6 HV_{0.2}, however a superior fatigue strength of 286 MPa at a stress amplitude of 280 MPa for 10^6 cycles followed by steel 1 and steel 3. Steel 1 exhibited extensive dislocation structures and intergranular deformation at grain boundaries, attributing to the potential early crack initiation by localised slip buildup. Additionally, steel 2 accommodates cyclic strain attributed to fine pearlite spacing, resulting in more uniform deformation. Likewise, steel 3 demonstrated significant dislocation entanglement at pearlite ferrite interfaces due to thicker pearlite lamellae, culminating in strain localisation and microcrack initiation.

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Declarations

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Competing Interests

The authors declare no conflict of interest.

Data Availability

Data will be made available upon request.

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