Materials Research Express

Fractography analysis into low-C steel undergone through various destructive mechanical tests

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Keywords: low carbon steel, tensile test, shear test, toughness test, fractographic analysis, FESEM, EDS

Abstract
The present work deals with a critical fractographic analysis into low carbon (0.18%-C) steel samples which were used for three different mechanical tests: tensile test; shear test; and toughness test. These mechanical tests were performed in standard sized specimens as recommended by ASTM. In each category of test, there were two different specimens with different physical states according to heat treated conditions. First specimen was in ‘as received’ condition and another was annealed. For annealing, sample was first heated up to austenitic temperature and inserted inside the sand for slow rate of cooling. As these two categories of samples were undergone through destructive tests, the variation in fracture behaviour of the samples was analysed by FESEM, XRD. A significant variation in fractographic images could be observed in different heat-treated samples. Micro-pores, dimples, cleavage facet, peaks, valleys, and cave formation were observed in the samples.

Nomenclature

C Carbon
ASTM American Society for Testing and Materials
FESEM Field emission scanning electron microscopy
XRD X-ray diffraction
BHN Brinell hardness number
VHCF Very high cycle fatigue
EBSD Electron Backscatter Diffraction Imaging
ECXI Electron Channeling Contrast Imaging
PP polypropylene
CP Carbon powder

1. Introduction
Low carbon steel is known for high strength, toughness, and flexural resistance. Due to which, it is widely used all over the world specially in construction, automobiles and numerous manufacturing industries which
manufacture machine parts. The typical mechanical properties and written in table 1. 0.18%-C steel is basically a ductile material as its carbon content is less than 0.25% [1]. The ductility is further affected by carbon content and other constituents. Some typical properties of low-carbon steel are given in table 1. Also, heat treatment plays an important role to decide the final mechanical properties of the low carbon steel. This paper is dealing with AISI 1020%-0.18% C steel. This steel mainly contains Ferrite (in majority) and a less pearlite in room temperature. During heat treatment and continuous cooling process, the nucleation and growth rate of ferrite and cementite are highly affected. Therefore, usually, coarse, and fine pearlite are the results of slow and fast cooling rates respectively. With the microstructural variation, a significant change in mechanical properties can be observed in the steel samples. Many of the mechanical tests are destructive in nature means the samples are broken or distorted during test and the properties are obtained.

The fracture attribute describes type of failure- ductile or brittle. Therefore, it is important to conduct fractographic analysis especially when the metals are taken into consideration. The high resolution fractographic images and elemental constituents present in the fractured surface are assessed along with microstructural study of the metal. The present work deals with analysis of fractographic images of heat treated low-carbon steel samples which were undergone through various mechanical tests such as shear test, tensile test, and toughness test.

| Mechanical properties       | Typical values |
|----------------------------|----------------|
| Hardness (BHN)             | 125            |
| Ultimate Tensile Strength  | 450 MPa        |
| Yield Strength              | 350 MPa        |
| Modulus of Elasticity       | 210 GPa        |
| Bulk Modulus               | 140 GPa        |
| Poisson’s Ratio             | 0.29           |
| Shear Modulus              | 85 GPa         |

2. Literature review

George Irwin was the pioneer to initiate fracture surface topography analysis (FRISTA) in 1980. He began to use microscopes to find topography in fractured surfaces of steels in which ductile-brittle transition phenomena happen. The detailed analysis on grain size variation, grain orientation, effects on grain boundaries, second phase particles inclusion in the fractured zone, etc can be easily done by FRISTA method [2, 3]. Kobayashi (1984) suggested a method to explain the topography in quantitatively manner by using an instrument called parallax bar. The innovative concept in this case was to take into account the conjugate fracture surfaces, which entails profiling the roughness along the same line on opposing crack faces, comparing the profile mismatches, and interpreting the overlap between the conjugate surfaces as denoting the amount of inelastic deformation that is still to take place prior to fracture [4, 5].

Many metal specially steels were analysed for fractography test thoroughly. Cotterell et al (1994) investigated that around the holes usually cracking and plastic deformation begin [6]. In addition, the pores also help in spreading the slip motion which usually results in the formation of slip steps on the free surface [7]. The higher the density of the pores, the more likely the rate of diffusion cracks to increase [8]. In the material matrix due to strong bonding at inclusion that is also the primary reason for the propagation of crack and as a result reduces life of the material. In the reverse case, if there is weak bond between them crack tip shielding takes place that reduces the driving force to crack [9].

Drar had worked in the fracture behaviour of twelve steel samples (Powder metallurgy) in the year 2000. He had divided the fractures into three segments R1 for growth initiation R2 for stable crack growth and R3 for unstable crack growth. In the result it was found that entire cracks were at right angle to the direction of principle stress. Such types of alloys which contain pores, the initiation of cracks started near the pores. But such alloys which do not have the pores two types of cracks were reported-one due to hard inclusion and second from irregularity of the surface. The second was due to machining or it was already present in the material. Initiation sites associated with MnS additives were not observed. More than one crack origins were not found in R1 type growth, it was found for high periodic stress [10].

Greenhalgh (2009) analysed that the study and analysis of fractography are very complex. The stress generated during the loading of thermoplastic composite, as a result tension creates in the interface. Due to different elastic properties of polymeric matrix and fiber, the generation of stress takes place [11].
To examine the nature of steel very high frequency is required and need almost same frequency to conduct the VHCF test in compression with old methods of fatigue test for life cycle more than $10^6 – 10^7$. 15 – 30 kHz range ultrasonic frequency was applied in the machine to reduce the test duration. Generally, in the region of VHCF the crack starts internally or may be in the surface. There are many stages of crack evaluation, first is initiation of crack second growth of crack in the region of fisheye third growth of crack outside region of fisheye and finally get fractured. Apart from that a new phenomenon takes place in the fish-eye region i.e., FGA (Fine granular area). 20 kHz frequency and −1 loading ratio are required to conduct the VHCF test for crank [12].

A work gives the total information of crack initiation nature and the reason of fracture of machine parts. From the striation spacing we can get the crack initiation rate with $\Delta K$ values. But the non-conventional fractography methods need existence of striations for calculating crack initiation rate and $\Delta K$ values. This is the main reason for the limitation for the quantitative analysis of the fracture material. So, for the development in the analysis of Fractography quantitative existence of fatigue crack is required. Hence in this work we suggest the fractography associated with analysis of fracture surface. EBSD and ECCI process were used to analyse the microstructural variation. Fe-3Al bcc single crystalline alloy has been used in this work. Apart from that Vein- like and labyrinth types of microstructures was also present in the fracture surface. These discussions are very informative for analysis of microstructure of crack propagation [13].

An experiment was done to analyse the impact of micro-organism on the corrosion nature of plane carbon steel when subjected to normal stress. It was found that environments play a vital role on the topography of tensile sample. But after the tensile test it was not possible to identify any clear trends of stress corrosion cracking [14].

In a work, an electrical resistance of thermoplastic composite with PPs and CP process was used to analyse the fracture surface of welded specimens. Shear loading is used for fracture analysis. In this test the same process was, used in mechanical test to generate the fracture surface. After the fracture test FESEM was used to capture the images. It helps to identify the interfacial adhesion of the material. It was clearly shown that it contains fibre recovered from very fine layer, cusps, and river lines. In addition to that it also indicates the strong interfacial adhesion was there with pores present. In addition to that, for high lap shear strength value laminates the heating adhesion is very useful. This heating element shows the excellent adhesion property. It was also clear that from the lap shear strength test the existence of matrix region with globular aspects shows unexpected effort [15].

Two different grades of steel, such as, 10HNAP and S355J2 were considered for combined bending-torsion fatigue test and the fractured samples were analysed under fractal dimension measurements. An optical profilometer was used under study. Proper levelling was done to get an extra-fine resolution. Author has suggested a proper improvement in this area [16].

The work of Dewangan et al (2022; 2022) focus on fractography analysis of welded steel samples which were fractured after tensile test. Steel samples with two different physical conditions i.e., ‘as received’ and ‘heat treated’ showed two different fracture characteristics. The quenched sample mainly showed zig-zag path and cleavage facets in the centre part of the fracture zone whereas normalized and as received samples showed higher degree of peak and valley as well as numerous dimples [17, 18].

### 3. Materials and methodology

The low-carbon steel with 0.18% carbon content was taken as work material in this experiment. Total 6 samples were prepared - 2 for tensile test; 2 for toughness test; 2 for shar test. Means two samples in each category. First sample was kept as original one means no changes were done in it. Second sample of each category was heat treated. The sample was heated at a temperature level of 900 °C for a period of 1 h then cooled inside building sand. As sand provides a slow cooling rate, the samples got cooled after 3 h. In other words, second samples in each category were annealed. The samples and their microstructures are shown in figure 1. For tensile testing, two rods with 300 mm length and 20 mm diameter were taken initially. Both the rods, including the heat treated one, were turned in lathe machine to shape them into tensile test specimens as per ASTM standard. The universal testing machine of Model: MT-001; Make: India was used to conduct tensile test. The capacity of the machine is 600 kN. The tension test was carried out by selecting a strain rate of 0.001 s$^{-1}$. The low carbon steel rods, tensile test specimens and broken specimens are shown in figures 1(a)–(c). The toughness test specimens were also prepared as per standard- a cuboid with $55 \times 10 \times 10$ mm with V notch of 30° angle at the mid of the length. For toughness analysis, Charpy impact test apparatus was utilized in accordance with ASTM. The toughness test specimens (pre- and post-testing) are shown in figures 1(d), (f). For shear test, two small rods of 8 mm diameter and 100 mm length were taken under study. The double shear test was performed on the same UTM which was used for tension test. The shear test specimens (pre- and post- testing) are shown in figures 1(e), (g).

Heat treatment is the technique to modify the grain structure and thereby to alter the mechanical properties of the steel as per the requirement. In annealing method, the steel, heated in the austenitic range, are slowly...
cooled to allow the $\gamma$-austenite into 100% pearlite. As the cooling time in annealing is slow, the coarse pearlite becomes final microstructure of the steel. In this study also, coarse pearlite was observed in the annealed specimen. The microstructure of both types of steels, original and annealed, was observed by using an optical microscope. The micrographs are shown in figures 1(i), (h). The micrographs were captured at a common magnification and scale of 100 $\times$ and 200 $\mu$m. In addition, XRD analysis was conducted to analyse the phases of steel which was taken under study. The XRD image is shown in figure 2.

**Figure 1.** Experimental procedure: (a), (b), (c) Rods for tensile test and broken specimens after tensile test; (d) Specimens for toughness test; (f) Broken specimens after toughness test; (e) Specimens for shear test; (g) Broken specimens after shear test; (h) Microstructure of original sample (i) Microstructure of annealed sample.
After conducting destructive tests, the small portion of the fractured parts from broken test samples were cut-off for further microscopic analysis. The fractographic analysis was conducted by using field emission scanning electron microscopy (FESEM). The samples for fractography analysis are shown in figure 3.

4. Analysis of mechanical test

The analysis of mechanical test results is shown in figure 4. Figures 4(a), (b) are the graphs between load and extension values measured from tensile test of original and annealed specimens. The ultimate tensile strength (UTS) of Original and Annealed specimens are 740 MPa and 511 MPa respectively. By heat treatment, the UTS value of annealed specimen got reduced with a remarkable increment in extension. The extension values of original and annealed specimens are 43 mm and 67 mm respectively. An increment of 58% in extension value was noticed in annealed specimen which is a clear indication of improved ductility. Figures 4(c), (d) is showing the load-displacement graphs in both the samples after conducting shear test. The shear stress values of original
and annealed specimens are 439 MPa and 381 MPa respectively. Again, a reduction in ultimate shear stress was observed in annealed specimen with an increment in extension prior to failure. An enhancement of 8% in elongation was observed in annealed specimen as compared with original one. Therefore, annealing has again proved to increase the ductile nature of the specimen. Figure 4(e) is showing a comparative bar chart with toughness values of original (88 J) and annealed (116 J) specimens. After heat treatment, the specimen showed an improvement of 32% in toughness value which is a drastic change in property.

5. Fractography analysis into tensile test specimens

Both types of fractured specimens, original and annealed, are shown in figure 3. The aim behind this work is to analyse the fracture behaviour which can be obviously either ductile or brittle fracture. The central part of the fractured zone was focused in FESEM (samples are shown in figure 3). Low-C steel is basically a ductile material which generally shows a cup-cone fracture on macroscopic observation. Therefore, on microscopic level also, availability of pores, dimples and valley formation are expected- which are the indication of ductile failure. The FESEM images of original and annealed specimens are shown in figure 5.
Figure 5(a) belongs to original sample which includes dimples, valleys, and facets. Dimples are not too small but more like shallow holes. Large dimples can be more specifically termed as valley formation. Valleys were predominantly found in two places. Both, dimples, and valleys are the indication of ductile failure. Besides these, cleavage facets are also clearly visible in the sample. Facets are formed when breaking parts are hard and brittle in nature. So, some parts in Low-carbon steel might be having brittleness which needs to further investigate.

Figure 5(b) is an image of annealed tensile test sample. It includes multiple micro-and macro-dimples, valleys, pores, and less-facets. The appearance of micro-dimples in a large area are an indication of increased ductility due to annealing process. Peaks and valley formation is like original samples. Unlike figure 5(a), a little part of facet in figure 5(b) proves that brittleness and hardness of the specimen has got reduced due to annealing.

6. Fractography analysis into toughness test specimens

As explained above, Charpy impact test was conducted to measure the toughness of the samples. From the experimental work, the toughness of annealed specimen was found better than that of original sample. The fractured parts of toughness test specimens are shown in figure 3. Similar to tensile specimens, these specimens were also observed at the central zone. To get the FESEM images, the scale and magnification of the set-up was fixed at a common value, i.e., 100 μm and 100×.

The FESEM image shown in figure 6(a) belongs to original sample of toughness test. It mainly includes holes, shallow valley, and dimples. Hole is so named as it is not having the characteristics of dimples. These types of
holes are less in numbers and looks like larger in appearance. The original sample does not include a significant number of peaks and valleys formed, although shallow valleys are always formed whenever a ductile material like low-carbon steel is fractured. The presence of dimples is different from usual. Generally, dimples are formed like a micro-pore which is having circular appearance in macro- and micro-level. But in this case, the dimples are not looking like pores but like small cracks with definite depth. In addition, a cleavage facet was also reported in this sample. Facets are the indication of brittle fracture.

Unlike original sample, the annealed sample is possessing a significant peaks and valleys formation in the middle part of the broken sample (figure 6(b)). In the entire region of study, a multiple number of dimples are available with a clear circular opening. Holes are visible in the valley itself means a pure ductile fracture happened in the annealed sample.

7. Fractography analysis into shear test specimens

The double shear test was conducted in this work means two cross sectional areas of a rod were sheared off simultaneously. Hence, shear stress will be measured as Load per unit $2 \times$ area. After shearing, a significant plastic deformation was noticed at the end of loading. Hence, the end portion of the sample was considered for FESEM analysis (as shown in figure 3). Figure 7 is showing the FESEM images of fractured surfaces done by shear test. Figures 7(a) and (b) belong to original sample and annealed sample respectively. Both the samples possessed similar kind of characteristics. Mainly, micro-dimples and macro-holes were observed in the samples with no facets, peaks, and valley formation. As in shearing action, the valley formation is not expected. The presence of
Dimples and holes are the proofs of ductile fracture. No facets mean there is no sign of brittleness in the fractured regions.

8. Conclusion

Fractography is the study of topography of fractured surface in order to establish the relation between microstructure and fracture behaviour. It may include a detailed study on microscopic observation, crack initiation and growth, mechanisms of fracture, mechanical characteristics of fracture, etc. Particularly for steel, this study becomes important as steel is mainly used in construction work. The broken steel samples, after performing destructive mechanical tests, are usually undergone through fractography test so that the exact failure behaviour could be determined.

An attempt has been made to analyse the fracture behaviour of specimens undergone through tensile test, toughness test and shear test. For the purpose, 0.18%-C steel workpieces have been taken under study. For all the three tests, the specimens were prepared as per ASTM standard. The fractured parts were analysed by FESEM. Steel samples were divided into two categories: Original and Annealed. Original samples mean there were no physical changes done in this sample. As the name suggests, Annealed samples were heat treated and cooled inside the building sand to provide slow cooling and thereby reforming the microstructure and mechanical properties of them.

A significant variation in fracture behaviour was noticed between original and annealed samples. In tensile test specimens, both types of specimens possessed mainly dimples and holes. Dimples are the key indication of ductile fracture. Annealed sample had significant peaks and valleys, but original sample did not show this. Also, the appearance of large facets in originals sample proved that it contained a little bit brittleness. In the toughness test, original specimen had a different kind of dimples that is not appeared like circular holes but like a crack. The annealed specimen contained higher degree of valley formation and greater numbers of micro-dimples than these of original sample. No significant variation in fractography test was observed after shear test because both the specimens showed a similar kind of appearance—dimples and holes. But the appearance of annealed specimen is quite different (with higher number of holes and dimples) resembling high ductility. It is concluded that annealing process has remarkably altered the properties of the steel. In other words, the annealing process has made the sample ductile. Ductility is an important characteristic of structural steel which is required to withstand shocks until elastic deformation.

Although the comparative results between original and annealed specimens were properly established in this work, there may be an issue with repeatability of experimentation. Taking only one sample in each test imposes a justifiable limitation in this work. Based on the result of only one sample in each category, throughout explanation of fractography was carried out. A large number of samples may provide more accurate results.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).
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