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Effect of different heat treatments on the fractural and microstructural behavior of pipeline steel

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ABSTRACT

APIX70 pipeline steel is classified under the American Petroleum Institute (API) specification as one of the high-strength low-alloy (HSLA) steels. The APIX70 has a very fine-grained microstructure so they have high strength and ductility properties. In this research work, an attempt has been made to compare the microstructural and mechanical behaviour of base and heat treated APIX70 pipeline steel. Heat treatment was performed at two different temperatures 900℃ and 800℃. Different phases such as ferrite, pearlite as well as blend of both were observed in base and heat treated APIX70 steel. Specimen heat-treated at 900°C shows an increase in hardness to 37.5 HRC as compared to the specimen heat treated at 800℃ which is 22.5 HRC. Base metal shows a 33.75 HRC hardness value. Microstructure of X70 base metal exhibit blend of ferritic and pearlitic phases. Heat treatment of X70 at 900℃ and 800℃ shows there is a significant grain growth takes place while lesser grain growth observed for specimen heat treated at 800℃.

1 Introduction

API X70 is a high-strength, low-alloy steel that is commonly used in pipeline applications for transporting oil and gas. It is a type of API (American Petroleum Institute) grade steel, which means it meets specific standards set by the API for use in the oil and gas industry. API X70 steel is known for its good mechanical properties, including high tensile strength, good toughness, and good weldability. Its low alloy content also makes it resistant to corrosion and other forms of degradation [1-3]. Due to its high strength and excellent properties, API X70 steel is widely used in the oil and gas industry for pipeline construction, as well as in other applications such as structural components in bridges and buildings [4]. API X70 steel is often used in harsh and demanding environments, including offshore drilling rigs, deep-sea pipelines, and high-pressure gas

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transmission pipelines. It is also used in the construction of bridges, buildings, and other structures where high strength and durability are required. Due to its excellent mechanical properties and corrosion resistance, API X70 steel has become a popular choice for pipeline construction in recent years. However, it is important to note that proper welding techniques and heat treatment processes must be followed to ensure the material's integrity and longevity. API X70 has a minimum yield strength of 70,000 psi (or 485 MPa) and a minimum tensile strength of 82,000 psi (or 565 MPa). It also has good weldability and is capable of being used in sour service applications (i.e., environments with high levels of hydrogen sulphide gas). API X70 is also suitable for use in sour service environments, which are characterized by the presence of hydrogen sulphide gas, as it can resist the corrosive effects of this gas [5-7]. Heat treatment processes such as annealing, normalizing, and tempering are crucial in modifying the microstructure by refining grain size, redistributing carbides, and relieving residual stresses. The fundamental principle governing these transformations lies in the thermodynamics and kinetics of phase transitions in steels, particularly the transformation of austenite into ferrite, pearlite, bainite, or martensite, depending on the cooling rate and temperature [8-9]. At elevated heat treatment temperatures (e.g., 800–900 °C), the austenitic phase becomes predominant, and upon controlled cooling, a refined ferrite–pearlite microstructure may form. Grain refinement is theoretically predicted to enhance yield strength and toughness according to the Hall–Petch relationship, which relates finer grains with higher resistance to crack initiation and propagation. Conversely, coarser grains formed at higher heat treatment temperatures can increase the likelihood of brittle fracture due to reduced grain boundary area available to hinder crack growth [10-12]. The fracture behavior of pipeline steel is also governed by the interplay between microstructural constituents. Ductile fracture typically initiates through void nucleation at inclusions and carbide particles, followed by their growth and coalescence. A finer and more uniform microstructure obtained through optimized heat treatment suppresses premature void formation, thereby enhancing ductility and toughness. On the other hand, inadequate heat treatment or excessive grain coarsening can result in brittle cleavage fracture, where crack propagation occurs along specific crystallographic planes with minimal plastic deformation [13-14]. In present work an attempt has been made to study the mechanical and microstructural behaviour of heat treated X70 pipeline steel.

2 Experimental Procedure

The material was used to study the microstructural evolution of X70 tubular steel during heat treatment of API grade. Sample dimensions of L=25 × B=20 × T=22 mm was cut from the original size plate obtained. 290×290×22 mm. and then polished with 400,600, 800, 1000, 1500, 2000, 3000 paper, then velvet paper is used to finish the polishing process. Microstructural evaluation of all heat-treated samples and as-received samples. Heat treatment of X70 steel can improve the strength and toughness. Heat treatment can improve the mechanical properties of the steel, such as strength and toughness, by modifying the microstructure.

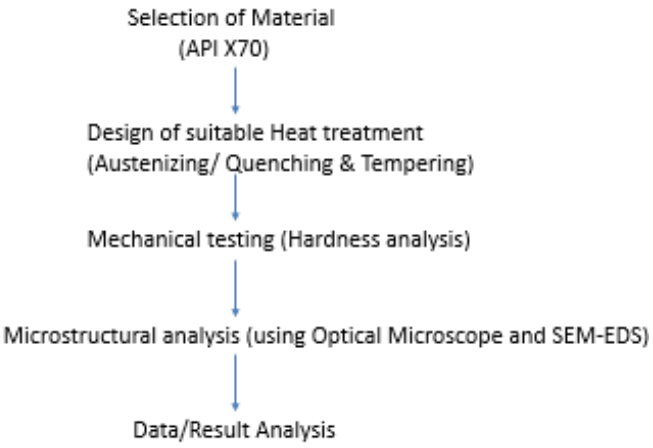


Fig. 1 - Experimental flow diagram

Quenching and tempering can improve the strength and toughness of API X70 steel, making it more suitable for use in high-stress applications. Annealing or normalizing, may be used to modify the microstructure of API X70 steel in different ways. For example, annealing involves heating the steel to a high temperature and then allowing it to cool slowly, which can help to relieve internal stresses and improve ductility. Scanning Electron Microscopy (SEM) is a powerful tool used

extensively in the analysis of microstructures of materials like API X70 pipeline steel. SEM can reveal the presence of different phases such as ferrite, pearlite, bainite, and martensite based on their morphology [7]. Fig.1 shows the experimental flow diagram of present study. Fig.2 (a-b) shows the real view of the specimen before and after heat treatment while fig.2c represents the actual dimension of the test specimen.

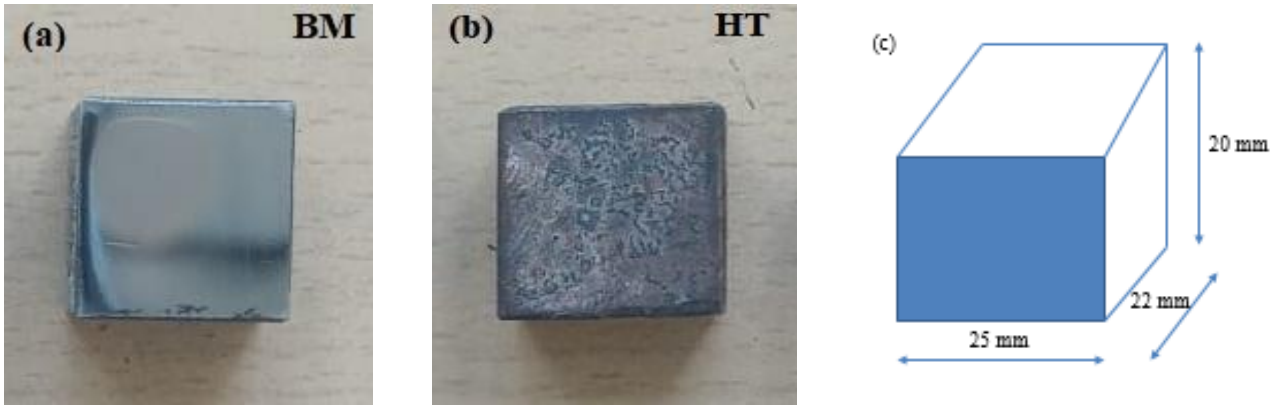


Fig.2 Real view of the specimen; (a) Base metal before heat treatment; (b) Base metal after heat treatment; (c) Actual dimension of the test specimen

3 Result and Discussion

The varying hardness values obtained from the three API X70 steel specimens 'base metal, and those heat-treated at 900°C and 800°C indicate significant effects of the heat treatment temperatures on the steel's microstructure and resultant mechanical properties. The specimen heat-treated at 900°C shows an increase in hardness to 37.5 HRC. This increase suggests that the heat treatment led to changes in the microstructure that enhanced the steel's hardness. At 900°C, changes such as the dissolution of pearlite, the potential formation of bainite, or the partial transformation to austenite, followed by a transformation to a harder phase upon cooling (like martensite or lower bainite), could occur [8]. This temperature is likely within or close to the austenitizing range for API X70 steel, which can refine the grain structure or alter the phase balance toward a harder microstructure upon cooling. The significant decrease in hardness to 22.5 HRC for the specimen heat-treated at 800°C is indicative of substantial microstructural softening. Heating to 800°C could lead to over-tempering of pre-existing harder phases like martensite (if present in the base metal) or the growth and coarsening of ferrite grains, both of which would reduce the steel's hardness. This temperature might be below the austenitizing range but high enough to allow for significant tempering or relaxation of the steel's microstructure. The base metal hardness of 33.75 HRC indicates the inherent properties of the steel without any modification through heat treatment [9].

Table 1. Hardness analysis of heat treated steels.

Point	**Hardness After Quenching**		**Hardness After Tempering**	
Temperature	HT-1 900˚C	HT-2 800˚C	HT-1 900˚C	HT-2 800˚C
1	25	37	35	25
2	29	34	38	22
3	26	38	40	19
4	28	40	37	24
Average	27	37.25	37.5	22.5

The hardness is directly related to strength; higher hardness usually means higher strength. The base and the 900°C treated specimens would be expected to exhibit higher strength, suitable for conditions requiring resistance to mechanical

deformation. However, higher hardness often comes with reduced ductility, which is critical to consider in applications where flexibility and impact resistance are important.

The impact of hardness due to heat treatment of API X70 steel can have a significant effect on its mechanical properties, particularly its strength, toughness, and ductility. During the heat treatment process, the steel undergoes a series of microstructural changes that affect its hardness. For example, the rapid cooling during quenching causes the austenite phase to transform into martensite, which is a hard and brittle phase with a high hardness level [10]. Table 1 shows the hardness of heat treated specimens.

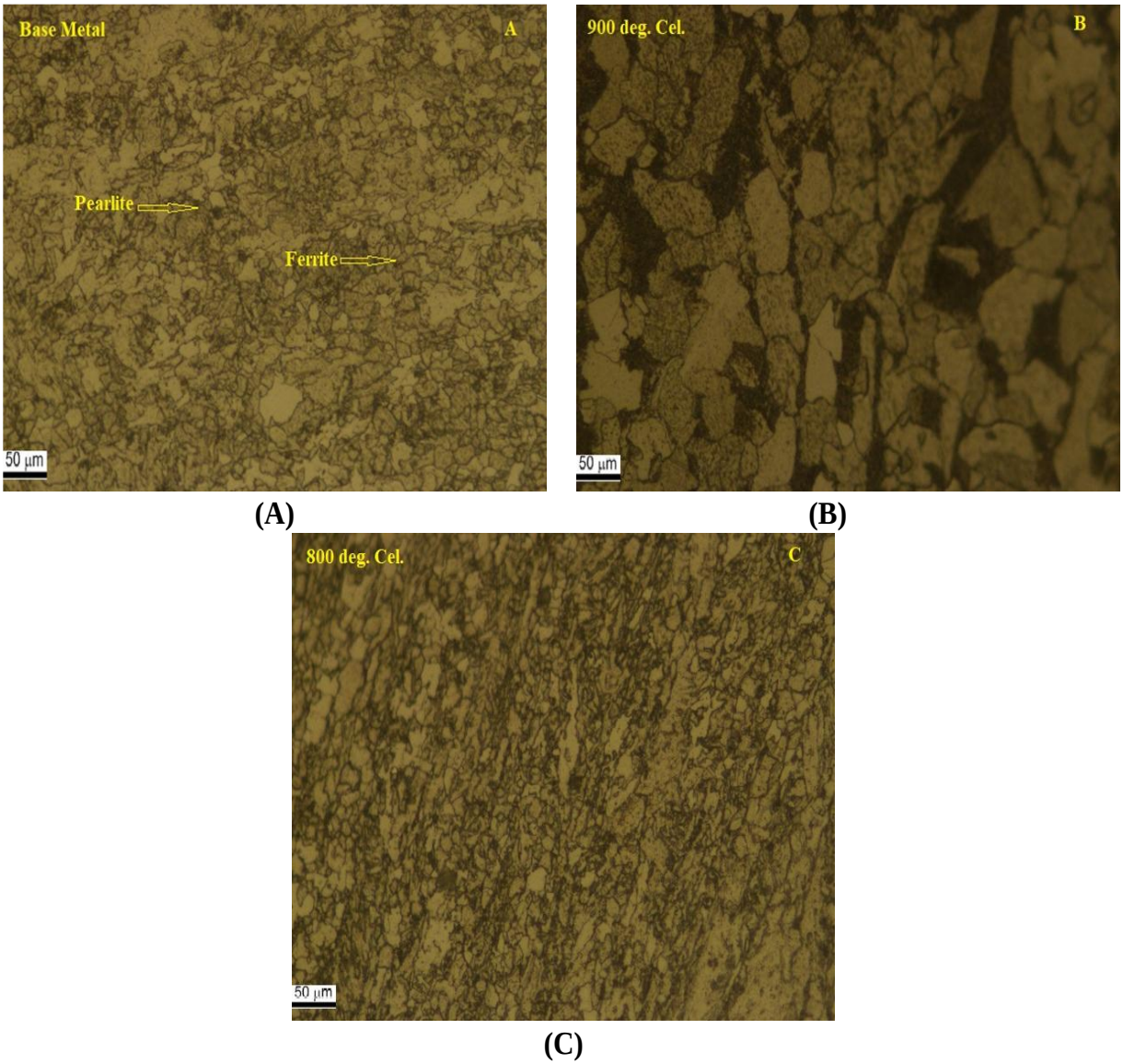


Fig. 3: Microstructure analysis of base and heat treated API X70 steel (A) Base metal; (B) Heat treatment at 900°C; (C) Heat treatment at 800°C.

There are different phases developed during the heat treatment such as at HT-1 at 900°C and HT-2 at 800°C. Fig.3 shows the microstructure behaviour of base and heat treated API X70 steel. API X70 steel microstructure most consist of the ferritic and pearlitic phases. Depending on the heat treatment and processing conditions, the volume fraction of pearlite differs in the microstructure of the API X70 steel. Ferrite is a soft, ductile phase, and appears lighter in the microstructural image as observed in Fig. 3. Ferrite has BCC crystal structure and is characterized with better ductility and toughness. Pearlite is a lamellar structure composed of ferrite and cementite and that represented by the dark regions of the microstructure. The mixture of ferrite and pearlite phases, imparts a blend of strength and toughness [11-16]. On observing Fig. 1, it could be noticed that at 900ºC (Fig. 3(b) microstructure experience a significant grain growth in which smaller grains combine to form

larger grains as compared to that at 800ºC (Fig. 3(c). The microstructural grain growth can modify the mechanical properties of the material, such as tensile strength and ductility. Such transformations are typically enabled by high-temperature exposure, resulting in a coarsening of the grain size, as observed. Mouadji et al (2018) have studied the influence of heat treatment on microstructure and tribological properties of flame spraying Fe-Ni-Al alloy coating. Post heat treatment effects on the mechanical and microstructure were observed by him. The results showed that with the increase of annealing temperature, the microstructure of coating treated at 823 K and 1023 K had several changes as follows: the reduction of porosity, formation of carbides and oxides [17]. Younes et al (2022) have studied the microsturure and tribological behaviour of thermally sprayed molybdenum coating. Results show clearly that good metallurgical bonding was achieved between the molybdenum coating and the substrate. The coatings consist of a lamellar structure along with some globular pore. X-ray spectroscopy shows that the coating is essentially constituted of a molybdenum matrix and carbides (Mo_2C) which improve hardness [18]. Sadki et al (2023) have studied the effect of NiAl bond layer on the wear resistance of an austenitic stainless steel coating obtained by arc spray process. He studied the tribological and adhesion properties of X10CrNi18-8 austenitic stainless steel coatings deposited on aluminium alloys such as AU_4G by using the arc-spraying process [19].

The SEM-EDX examination also makes it possible to observe the arrangement of the microstructural constituents including carbides, inclusions and other alloying constituents illustrated in fig.4. The main component of ferrite is Fe_2O_3 (Iron oxide) while other metals such as magnesium, aluminium, manganese, copper and nickel can combine with it to form ferrite and shown by EDX results in fig.4A. Pearlite having silica (SiO2) as the main component while it can combine with other oxides such as aluminium oxide, sodium oxide, iron oxide, and magnesium oxide in very small amount and shown by EDX results in fig 4B. As seen in fig 3A, the general polycrystalline microstructure seen in the image has a large number of grains. During austenization heat treatment at 900° C (Fig. 4A), the grain size showed a gradual increase over that of the samples austenized at 800° C (Fig.3C). This phenomenon was expected due to annealing temperature and time in the furnace.

In fig.4, the surface seems to have a rough, or irregular features with several crevices or boundary protrusions. This may indicate some extent of plastic deformation or exposure to high temperature, which in turn creates microstructural changes, including but not limited to seam development or crack propagation. In selected previous studies it has been noticed that some inclusions such as MnS, MgS and NaCl in the microsturure gives preferential sites for crack formation and facilitate appropriate path for crack propagation.[13]. To mitigate the formation of such inclusions in the microstructure autenization above the non-recrystallization temperature followed by air cooling process was adopted which develop the formation of upper bainite and polygonal ferrite in the microstructure. Bainite sheaves were nucleated at the prior austenitic grain boundaries and surrounded by iron carbide particles. The ferrite plates in the bainitic microstructure are nucleated at the austenitic grain boundaries and form packets consisting of parallel plates with similar crystallographic orientations. In pipeline steels, austenite first transforms into polygonal ferrite during continuous cooling, and carbon concentrates in the remaining austenite.

During the cooling, the remaining austenite with high-carbon content transforms into granular discrete island constituents that are martensite lath-like when the temperature reaches the martensitic transformation point [14]. It has been observed in the previous literature that how structural hardening heat treatments affect the mechanical and microstructural properties of the B_2O_6 aluminum-copper (Al-Cu) alloy, particularly when an iron (Fe) additive is included. Various experimental methods, including hardening heat treatments and hardness measurements, were conducted to assess the changes in mechanical and structural characteristics. β-phase which is a specific microstructural phase, dominated the alloy's response, influencing its mechanical behavior more than any hardening effect [20]. Abdelhek et al (2023) has observed the correlation of tensile properties of arc-sprayed coatings and easy testing methods. He observed the relationship between mechanical and metallurgical behaviour by depositing two metallic coatings through the arc-spray technique. He observed that both coatings (Mo and stainless steel) have increased the total elongation of the substrate, improving ductility. Also the yield strength and hardness both increased when compared to the uncoated substrate [21]. Rassim et al (2016) has studied the effect of TiO_2 and ZrO_2 reinforcements on properties of Al_2O_3 coatings fabricated by thermal flame spraying. He observed the microstructure, phase composition, microhardness and tribological properties of the coatings. The XRD results of the coatings reinforced by TiO_2 (AT-3) revealed the presence of α-Al_2O_3 phase as matrix and new metastable phases of γ-Al_2O_3 and κ-Al_2O_3. However, the coatings reinforced by ZrO_2 (AZ-25) consist of α-Al_2O_3 as matrix, q-ZrO_2 and m-ZrO_2 [22]. Rassim et al (2019) has evaluated that how post-spray heat treatments (PHTs) alter the microstructure, hardness, and friction/wear of Ni-Cr-Al-Mo coatings. Coatings were deposited on tool steel by arc spray and subjected to PHT at 600–1,100 °C for 1–4 h. Microstructure (XRD/SEM-EDS), porosity, and microhardness (HV0.3) were measured; tribological tests used a pin-on-disk configuration at room temperature and 400 – 600 ° C. Heat treatment densified the lamellar structure, reduced porosity, and promoted β

-NiAl/ γ -Ni_3Al precipitation and (in Mo-bearing systems) $MoSi_2$ formation, increasing hardness and adhesion while lowering friction and wear via formation of compact tribo-oxides/glaze films at higher PHT temperatures [23].

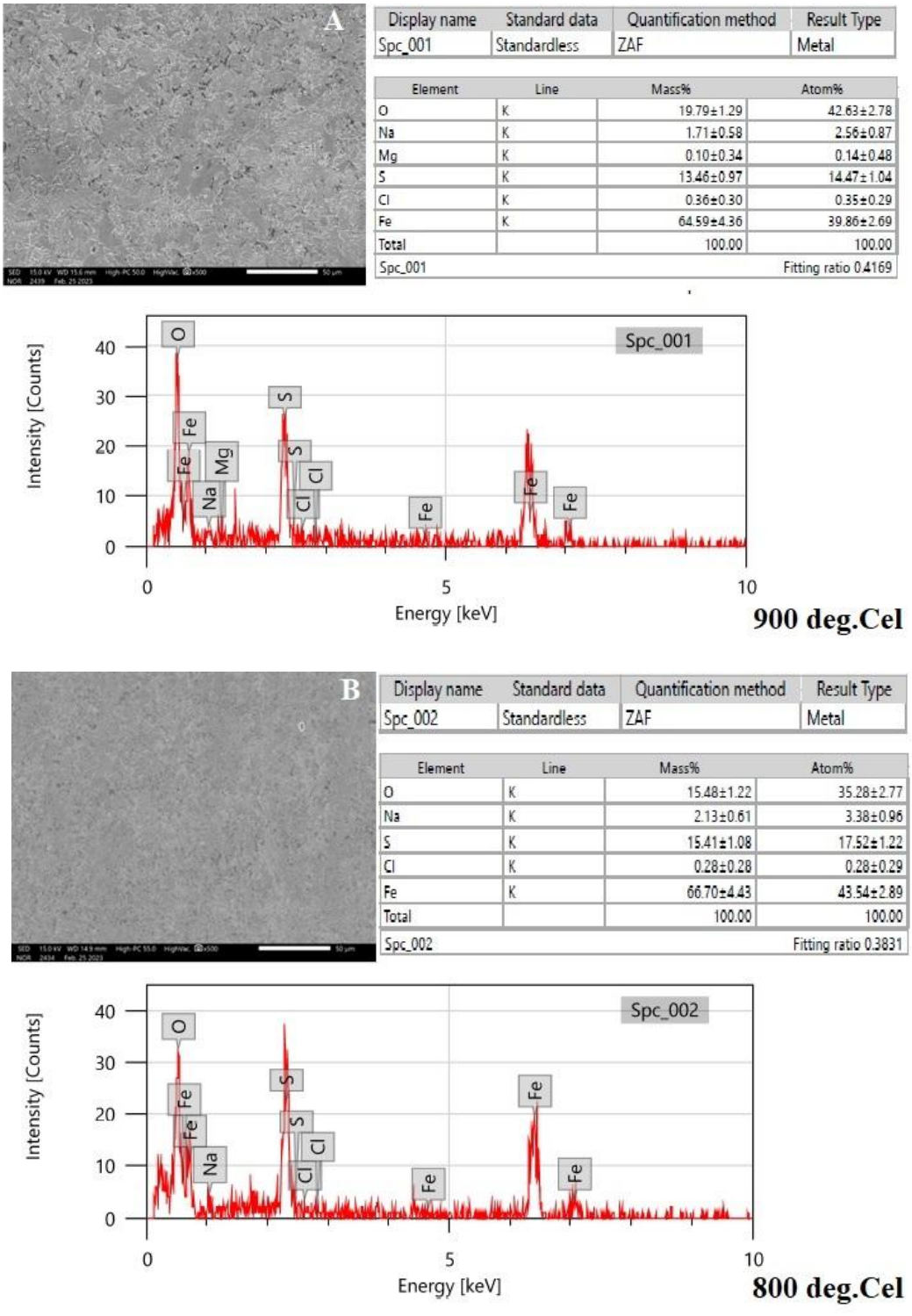


Fig.4: SEM-EDS analysis of heat treated API X70 steel. (A-B) Microstructure and EDS of specimen (900 ℃ and 800 ℃) at 500X magnification.

3.1 Fractography Analysis

The features observed are extremely important with regard to the failure as they can be used to obtain information on the material and the conditions that led to its failure. In a similar way, it can be noted that the presence of both ductile and brittle

features points towards a mixed mode of fracture occurring on the material, that is, plastic and cleavage fracture occurring at the same time. Fig.5 is a fractography of specimens impacted at base metal and heat treated (900 ℃ and 800 ℃) respectively. Impact fractographic tests were done to fix materials towards their conditions at 500X magnification electron microscope as per ASTM C1322 criterion. The analysis of the fractures has been performed Graham, 2005. From fig.5a, it has been seen that base metal contains dimple type dense morphology, while heat treated samples showed presence of deep dimple and sharp facets beside irregular facets. This is because of the tempering of heat treated specimens takes place during heat treatment which causes grain coarsening and small voids occur. In addition, the cause for ductile tearing mode and the origin of sharp features and irregular ridges is due to the merging of the small pores during the heating. It was noted that “deep dimples” which are considered typical features of ductile failure appeared in Fig. 5 (a and b).

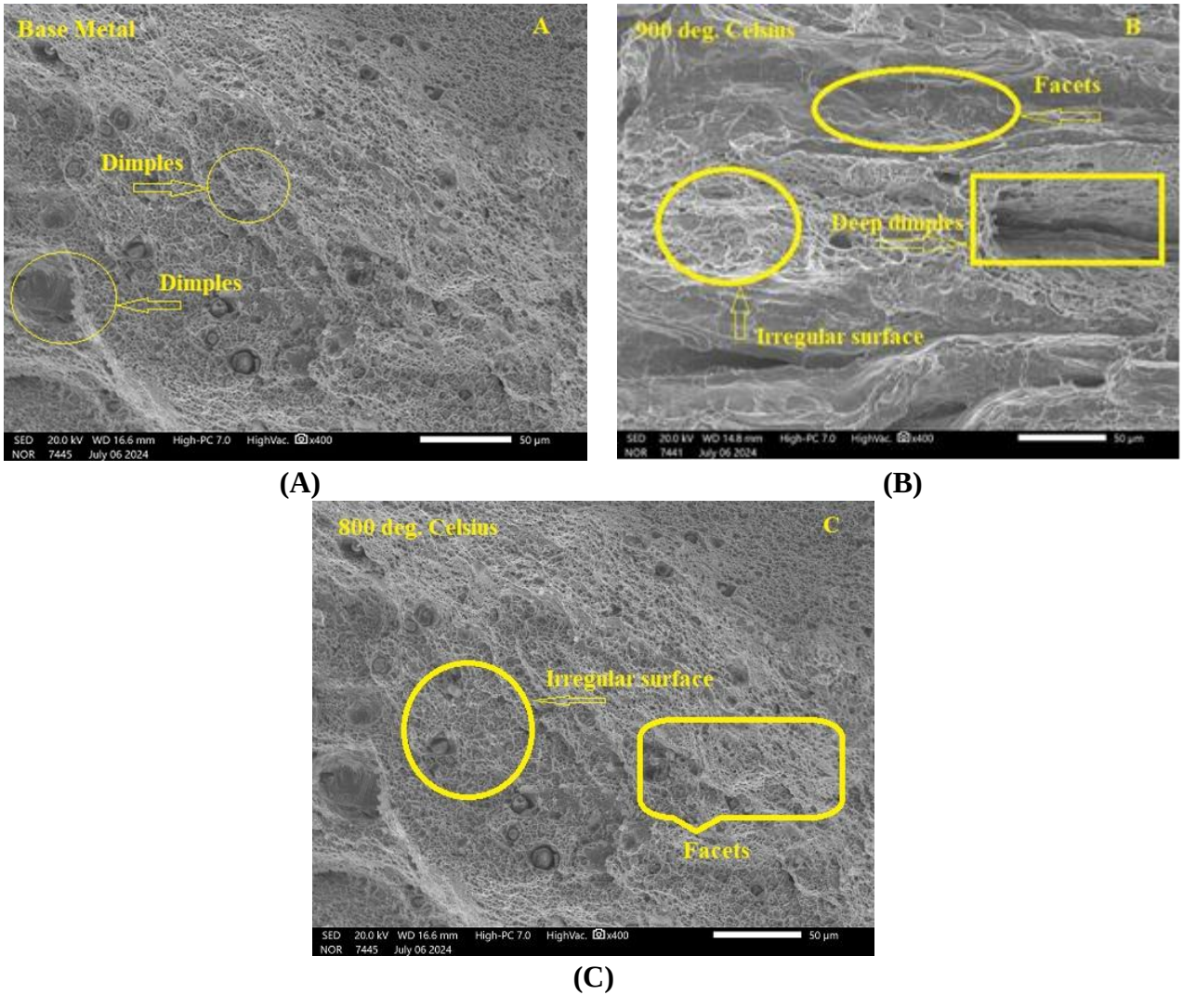


Fig.5 Fractography analysis of impact specimen; (A) Base metal; (B) Heat treated at 900 ℃; (C) Heat treated at 800 ℃.

Future research work could be carried out by using advanced characterization techniques like EBSD, TEM, and XRD to understand grain refinement, phase transformations, and dislocation structures. Additionally, fracture mechanics testing under varying service conditions such as temperature, pressure, and corrosive environments will provide insights into long-term reliability. Investigating novel heat treatment cycles and thermo-mechanical processes may help optimize strength–toughness balance, reduce susceptibility to brittle fracture, and improve pipeline steel performance in demanding applications.

4 Conclusion

The hardness of HT-1 (900°C) specimen is increased as compared to the HT-2 (800°C) specimen. Microstructure analysis of both HT-1 and HT-2 specimens, there is formation of different phases such as pearlite and ferrite with some small inclusions of carbides. The microstructural grain growth can modify the mechanical properties of the material, such as tensile strength and ductility. Such transformations are typically enabled by high-temperature exposure, resulting in a coarsening of the grain size, as observed. Microstructure of X70 base metal exhibit blend of ferritic and pearlitic phases. Heat treatment of X70 at 900℃ and 800℃ shows there is a significant grain growth takes place while lesser grain growth observed for specimen heat treated at 800℃. Specimen heat-treated at 900°C shows an increase in hardness to 37.5 HRC as compared to the specimen heat treated at 800℃ which is 22.5 HRC. Base metal shows a 33.75 HRC hardness value. The decrease in hardness due to the microstructural softening at 800℃ as compared to the specimen heat treated at 900°C. Higher hardness often comes with reduced ductility, which is critical to consider in applications where flexibility and impact resistance are important. Ductile tearing mode of failure and origin of sharp facets and irregular ridges were observed in heat treated sample as compared to the base metal. Dimpled and faceted features have been identified hence suggesting mixed mode failure which is characterised by both plastic deformation and cleavage. The temperature higher than 900°C leads to changes in grain size and phase structures, the formation of voids in the material and its embrittlement.

Conflicts of interest statement:

It is certified on behalf of corresponding author (Lochan Sharma) that present research is not funded by any external agency and authors declared that there is no conflicts of interest in the present research.

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