Strength characterization and microstructure of untreated and cryo treated CrMoV low alloy steel

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Abstract. This study examines the impact of various cryogenic treatments compared to traditional vacuum heat treatment on the development of mechanical characteristics and microstructure of CrMoV Steel. The mechanical characteristics of the samples were analysed to determine the impact of untreated and cryogenically treated materials on hardness, toughness, tensile strength, and microstructure. Experimental evidence showed that the cryogenically treated samples exhibited improved mechanical characteristics compared to the traditionally treated ones. The findings indicated that specimens subjected to cryogenic treatment at a temperature of -100°C for a soaking period of 2 hours, followed by furnace tempering at 200°C for 2 hours, exhibited an 81% increase in tensile strength, an 18% increase in hardness, and a 123% increase in toughness. The confirmation of numerous phases and complicated carbides was achieved using X-ray diffraction, while the shape of microstructure and cracked surfaces was examined using field emission scanning electron microscopy methods.

1 Introduction

Low-alloy CrMoV steels, specifically engineered for high-temperature applications, are classified as fundamental building materials used in the production of power equipment, including pipes for live steam and reheat steam. The CrMoV steels exhibited good strength and plasticity qualities throughout the temperature range of steam up to 560°C [1]. Deep Subzero treatment (DST) enhances the mechanical characteristics of metalic materials by facilitating the conversion of remaining austenite (RA) into martensite (M), augmenting the development of scattered carbides, and expediting the elimination of residuary strain. This procedure was primarily used for the treatment of tool steels. Cryogenic treatment of high-speed steel has been explored for its effect on hardness performance more recently by Molinari et al. [2-4]. The findings showed that cryogenic treatment significantly increased toughness and hardness. The researchers in the follow-up study demonstrated that the white coating and the precipitation of secondary carbides significantly boosted the steel's wear resistance using conventional heat treatment and CT [2,3]. Material characteristics and

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microstructure have a major role in hardness and wear resistance. It is usual practice to temper martensitic steels to increase their resistance to impact. Their findings reveal that surface hardness is the most important component in wear resistance [5-7] and that increasing the steels' impact toughness won't help much if the steels don't have a sufficiently hard surface. An increase in hardness increases the material's resistance to wear because abrasive particles on the worn surface cut at a shallower depth. However, wear resistance in martensitic steels that are rich in primary carbides is dependent on both the cohesive force between the primary carbides and matrix and the hardness of the martensitic matrix. It is important to consider the probability of selective matrix wear as well as carbide spalling and cracking. High-chromium white cast irons have higher wear resistance due to Kim et al.'s heat treatment's [8] reduction of the hardness difference between the carbides and the matrix. Zheng et al. [9] found that the interaction between the cementite and pearlite in chromium white cast iron decreased the wear resistance as the hardness of the pearlite matrix increased. A low-alloy ferritic steel made of vanadium (V), molybdenum (Mo), and chromium (Cr) is called CrMoV. Applications requiring great strength and stability at high temperatures are the focus of its engineering, particularly in the petrochemical and power generation sectors. CrMoV is appropriate for high-stress applications such as steam turbines, pressure vessels, valve casings, and similar settings because it retains its mechanical integrity at temperatures up to about 580 °C. It improves the following: weldability, strength, toughness, and yield ratio; secondary hardening effects, superior low-temperature toughness after normalizing and grain refinement. Even with a lot of research, there are still a few areas that need more study. Segregation of phosphorus and persistent embrittlement in weld regions, Ultrahigh-strength variations and their wider uses, composition optimization for improved creep qualities, and heat treatment to strike a balance between strength and toughness. The primary objective of this study is to examine the mechanical strength, and microstructure alteration of CrMoV steel of untreated and DCT treatment and tempering. It has been verified that this innovative process is an efficient technology for enhancing the mechanical characteristics of low alloy steel.

2 Material and experimental methods

2.1 Materials and processing

The experimental material chemical composition CrMoV novel low alloy steel used for the study were depicted in table 1.

Table 1. Chemical composition of CrMoV alloy steel (weight percentage).

W%	C	Mn	Si	P	S	Ni	Cr	Mo	V	Cu	Fe
CrMoV Alloy Steel	0.32	0.89	0.34	0.011	0.009	0.54	1.34	1.49	0.28	0.33	Balance

The present investigation uses Low Alloy CrMoV Steel forging material. Rectangular Shape Specimens of Size 45 mmx20 mmx6 mm were prepared from Low Alloy CrMoV Steel forgings by using wire cutting machine then Surface grinding operation was performed to all prepared samples by surface grinding machine and at last samples were diamond polished by using diamond polishing machine.

2.2 Heat and cryogenic process

Figure 1 shows the three stage process steps were followed in this study. Stage-1 was performed in Vacuum hardening furnace which are displayed in Figure. 2.a. CrMoV steel samples are heated to 535°C for one hour, then soaked there for an additional hour. They were then heated to 835°C for an additional hour, soaked there for an additional hour, and then heated to 1060°C for another hour, soaked there for an additional hour, and then cooled to room temperature. Following this, samples were stabilized at 180°C for two hours. Stage-2 was performed in sub-zero chamber which is shown in Figure 2. b. Samples were taken out from the vacuum hardening furnace and kept in the sub-zero chamber at -100ºC for two hours. Stage-3 was performed in tempering furnace which is shown in Figure 2. c. Samples were stored in a tempering furnace after being removed from the Sub-zero chamber and tempering process was performed twice at the temperature of 200ºC for two hours. Figure 3 shows the untreated and treated samples used for the characterization.

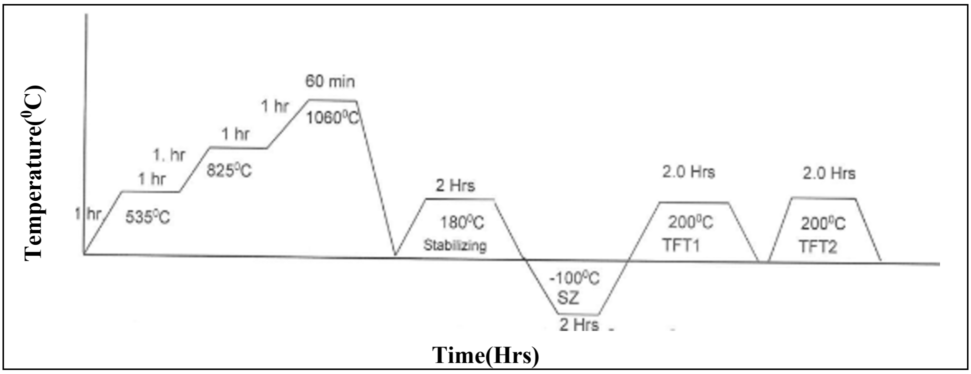


Fig. 1. Heat and cryogenic treatment process curves of CrMoV alloy steel.

Fig. 2. Various stages involved in the study.



(a) Non-treated sample (b) Cryogenic treated sample. (c) Mould samples.

Fig. 3. Untreated and treated samples.

2.3 Methods of experiments

Tensile tests were conducted on the Cr-Mo-V low alloy steel throughout a broad temperature spectrum, ranging from -195^{0}C to 600^{0}C. The tensile characteristics were examined using the ASTM E 8 standard [10]. Cylindrical specimens, designed according to ASTM standards, with a diameter of 4 mm and a gauge length of 20 mm, were taken from a vessel forging in the axial direction at a place corresponding to one-fourth of its thickness. The low temperature tests were conducted using a servo-hydraulic UTM with a capacity of 500 kN. The UTM was equipped with an environmental room that could generate temperatures ranging from -150^{0}C to 600^{0}C. A K type thermocouple was used to monitor the temperature of the test sample, which was connected to the sample gauge. Tensile tests were conducted at a temperature of -195^{0}C by submerging the test specimen along with the tie rods and grasping fittings.

For the impact tests in accordance with the ASTM E 23-20 [11] standard, utilising full-size Charpy specimens of 10 x 10 x 55 mm, with a 2 mm deep notch at a 45-degree angle. An impact pendulum equipment with a 295 J (30 kg-m) capability was used for the tests. The samples were immersed in a bath of dry ice and ethanol for the testing, which measured the impact energy absorbed throughout a temperature range of -75^{0}C to room temperature. A minimum of three valid tests were conducted at each temperature. These tests were required to be performed within a specified time frame of 6 seconds. Furthermore, it was made sure that the anvil indents on the two failed impact sample pieces were equally spaced from each other.

Utilizing the Vickers technique, the macro-hardness measurements were carried out. There were ten measurements taken for the metallographic specimens that were treated with any heat-treatment settings. Following these measurements, the mean values and standard deviations were computed. The machine specification are as follows, Wilson Rockwell Hardness Tester, Where Preliminary load :10Kg, Test load:60Kg, 100Kg, 150 Kg, Dwell Time: 1 to 50 Seconds, Test Cycle: Motorized. The final hardness was determined by averaging the results of five separate hardness tests performed on each sample.

For XRD, Diamond paste was used to polish the samples and Using a 4% Nital solution, they were etched. Metallographic sandpapers were used to mechanically grind the specimens before transmission electron microscopy (TEM, Tecani G2 F30 S-TWIN, USA) was used. The slices were next punched to three-millimeter-diameter disks and, last, electro-polished with a twin-jet polisher in a ninety-three percent ethanol and seven percent perchloric acid solution. The identification of the austenite that was maintained in the samples was accomplished by the use of X-ray diffraction (XRD) (Rigaku Dmax-2500). Figure 4 represents the entire experimental process followed in the current investigation.

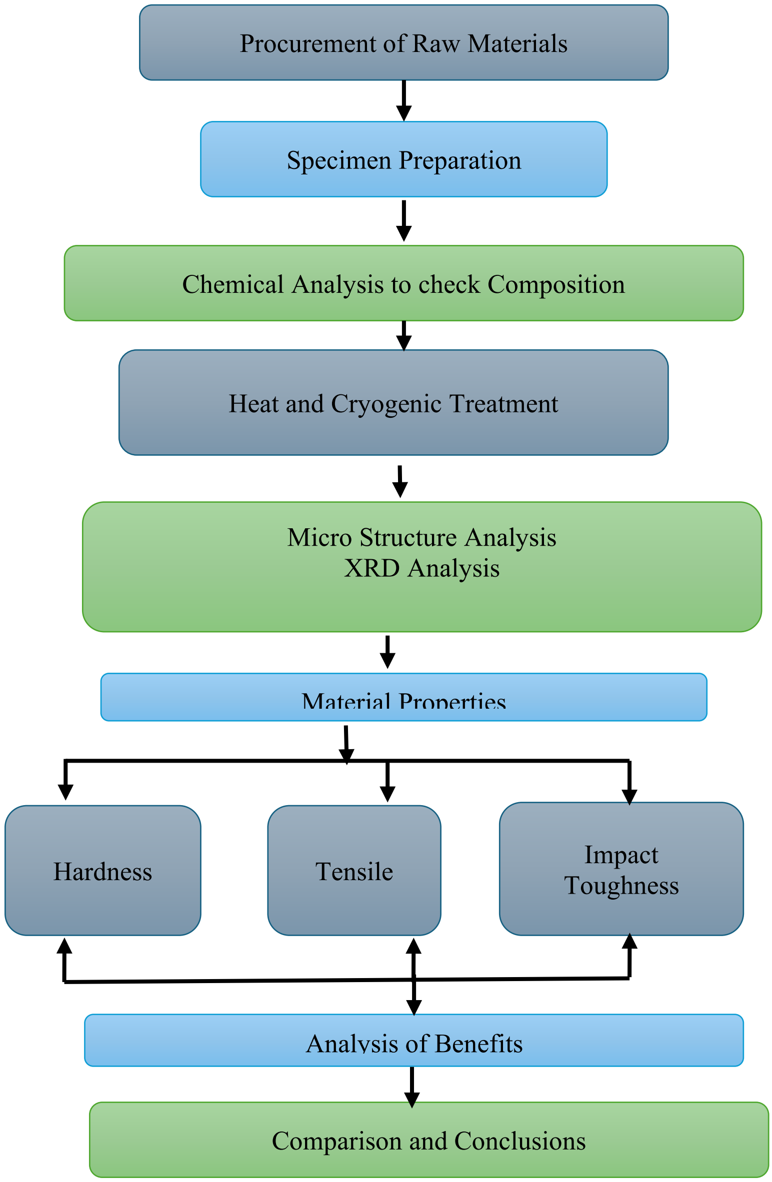


Fig. 4. Schematic of the experimental process.

3 Experimental results and discussion

3.1 Tensile test

The mechanical properties of the samples that underwent a two-hour cryogenic procedure at -100°C and a two-hour tempering process at 200°C are shown in Figure 1. Figure 5 demonstrates an increase in yield strength from 731.35 MPa to 1868.64 MPa, and a rise in tensile strength from 812.83 MPa to 1478.62 MPa. The percentage of elongation had a significant decline, dropping from 28.04 to 11.28 at a temperature of -100°C. In terms of improving tensile strength and yield strength, the results indicate that deep cryogenic treatment at -100°C produces better results than cryogenic treatment at -60°C.The deep cryogenic treatment of CrMoV steel at a temperature of -100°C improves its performance due to the fact that chilling temperatures below -80°C, as indicated in reference [12], facilitate the modification of the microstructure and dissolution of elements, as stated in reference [13]. Figure 6 shows the samples used for tensile test.

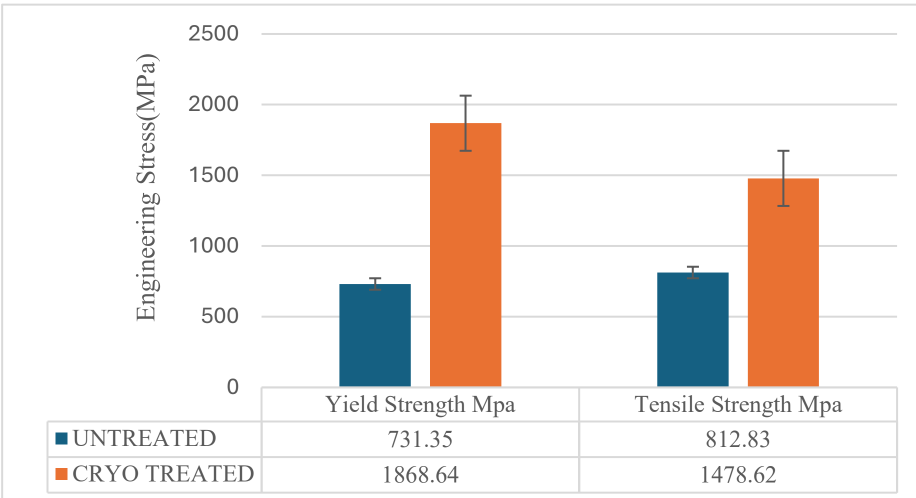
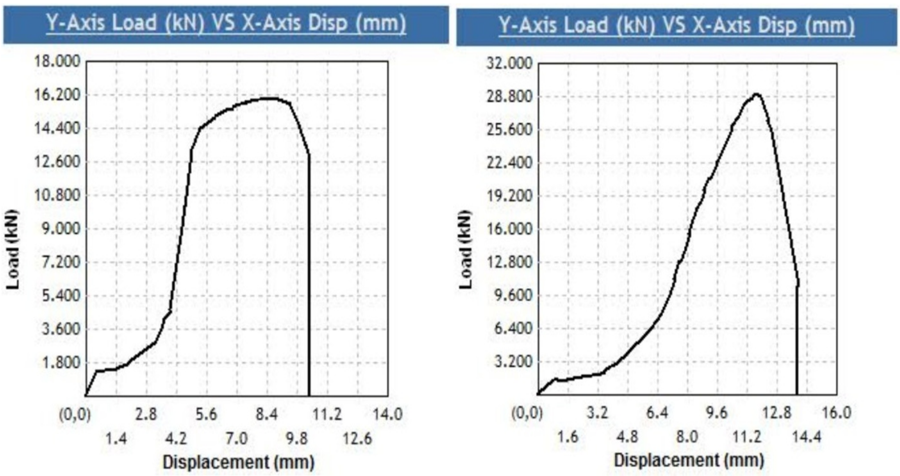


Fig. 5. Mechanical characteristics of untreated and cryo treated samples.

Fig. 6. Samples used for tensile test.

It is easy to see from the results that the mechanical properties of the steel samples are more significantly impacted by the deep cryogenic treatment and tempering. These characteristics include tensile strength, elongation, and the yield strength ratio. The change of residual austenite into martensite and the refining of martensite laths are the reasons behind this. The primary cause for this rise was attributed to the presence of densely packed secondary carbide precipitates at locations of dislocations. By retarding the coarsening kinetics of carbides, it was feasible to enhance toughness without compromising tensile strength, despite the carbide production initiated by the cryogenic procedure [14]. Following cryogenic treatment, there is a noticeable decrease in tensile elongation along with the strength and hardness increases that are seen. The microstructural changes caused by deep cryogenic cycles, such as the conversion of retained austenite to martensite, the refinement and precipitation of fine carbides, and the modification of residual stress states, are consistent with this compromise. These modifications increase yield strength and wear resistance, but they also increase the risk of brittle fracture since the material's capacity to undergo plastic deformation is diminished by the newly produced micro components and internal stresses. Therefore, when ductility, fracture toughness, or high-energy impact resistance are crucial, cryogenic processing must be used carefully even if it can be a useful method to increase load-bearing capacity and wear life. Applications like as cutting tools, dies and punches, journal bearings, wear plates, and certain rolling/sliding contacts that value surface hardness, wear resistance, and dimensional stability over bulk ductility are best suited for cryogenic treatment. Figure 7 represents the Untreated Tensile graph and Cryo treated Tensile graph.



Untreated tensile graph Cryo treated tensile graph

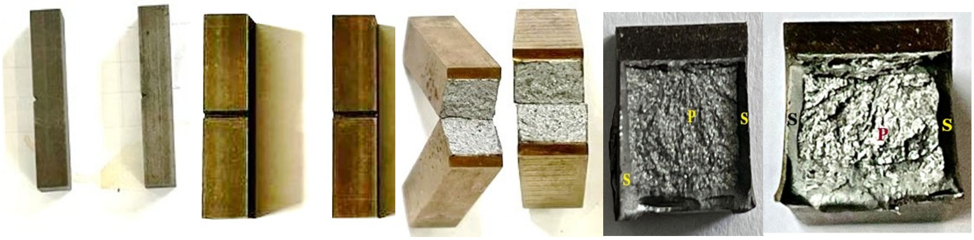
Fig. 7. Untreated and cryo treated tensile charts.

3.2 Impact test

Table 2. Impact test results.

CrMoV Tensile test	Untreated	Treated
Max	32	86
Min	28	48

To find out how DCT affected energy absorption during an impact event, an investigation was carried out. Table 2 shows that the toughness was 32 J for the usual quenching (sequence 1) and 86 J following sequence 5 (cryogenic treatment). Figure 6 demonstrates the impact of tempering on the toughness of specimens that have undergone both cryogenic and conventional treatments. It has been noted that under both situations (cryogenic and conventional), the toughness first decreases to around 11 J at 300 ˚C when the tempering temperature is raised, and then it increases to higher values at 455 ˚C. This phenomenon is sometimes referred to as "temper embrittlement" of steel, which occurs when the steel is tempered within the temperature range of 250 to 400 ˚C.



Un Treated Cryo-Treated After Impact test Untreated Cryo-Treated

Fig. 8. Samples used for impact test.

3.3 Impact fractography

The toughness was found to be 28 J following cryogenic treatment, this slight decrease in toughness could be attributed to an increase in martensite content. Upon increasing the tempering temperature, the toughness first drops to about 48 J at 300 ˚C before increasing to higher values of 86 J at cryogenic temperature. There is an increase in the impact toughness. The precipitation of finer ε-carbides and tiny secondary carbides occurs as a consequence of DCT in comparison to the untreated samples, which is in agreement with the findings of Li et al. [15]. The enhancement of the strength and impact toughness may be achieved by the refining of the ε-carbides and the precipitation of secondary carbides, both of which are mutually advantageous. When it comes to increasing the hardness of the material, the precipitation of secondary carbides is beneficial. On the other hand, the martensite matrix is easily decarbonised as a result of the precipitation of the secondary carbides, which causes the matrix's hardness to decrease. As a consequence of this, the DCT analysis reveals that the hardness of the steel under investigation is almost unaffected. The impact toughness has visibly increased, although the hardness has remained almost exactly the same. The DCT specimens' fracture properties were contrasted with those of the conventionally treated specimens (Figure 8). The shear area (S region) in the former (a) is clearly less than in the latter (b). According to a study, a greater proportion of "S" to "P" zones is a sign of increased ductility. This indicates that the conventionally treated specimen, which has more toughness, exhibits a wider shear area compared to the DCT specimens. Fracture occurs via a process that includes the development of cracks, their propagation, and sometimes rapid fracture. If the remaining cross-section can no longer sustain the imposed stress, it is said to have experienced fast fracture. Crack initiation occurred at various locations on the outer surface, eventually leading to the growth of a single dominant crack across the entire cross-section.

3.4 Hardness

When cryogenic treatment was performed right after quenching, the mean hardness increased somewhat from 33 HRC to 39 HRC (before tempering), according to the influence of cryogenic treatment prior to any further tempering procedures as shown in Figure 9. The marginal enhancement may be ascribed to the conversion of a limited quantity of the remaining austenite into martensite as previously described. The DCT samples exhibited greater hardness, which may have been a contributing factor to their reduced toughness. At a temperature of 455 degrees Celsius, cryo-treatment results in a little increase in hardness but also causes a significant decrease in toughness.

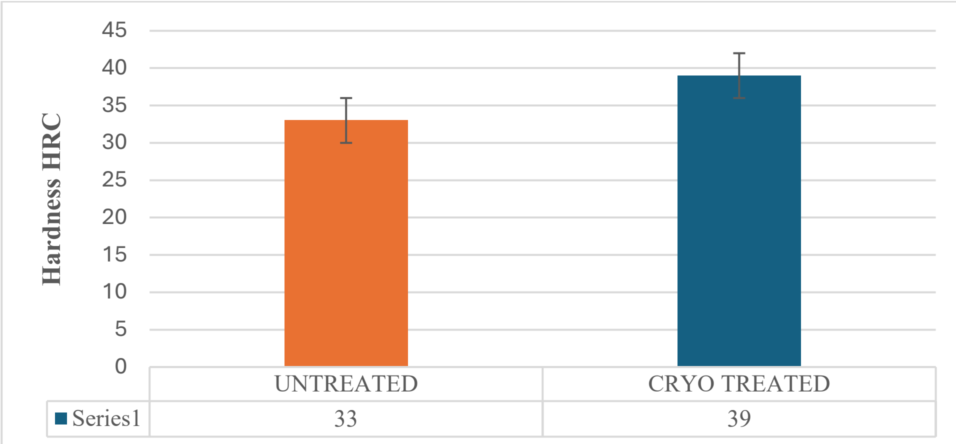


Fig. 9. Untreated and cryo treated test values of hardness HRC.

It has been observed that the cryogenically treated samples are marginally harder throughout the tempering temperature range than the conventionally heat-treated samples. The greater volume fraction of martensite is probably what caused this increase in hardness. However, it is possible that cryogenic treatment may have also facilitated the formation of carbides during tempering, leading to further hardness enhancement. This aspect requires additional investigation. It is worth mentioning that previous studies have reported hardness improvements in tool steels as a result of cryogenic treatment, where the amount of retained austenite is significantly higher than that found in the current steel.

Tempering temperature is the primary factor in determining CrMoV steel's final hardness. Measurements of CrMoV steel are hardness before and after thermal stabilization processes. The samples possessed an average hardness of 20 HRC immediately after the vacuum quenching and tempering process (Q-T), but before the stabilisation step. After the first phase of stabilisation, the material's hardness increased from 20 HRC to 36 HRC when the thermal treatment temperatures were raised. A 55.55% Hardness Improvement were observed in cryogenic sample. The development of the microstructure during tempering at various temperatures is responsible for these alterations in behaviour. In tempered CrMoV steel, the matrix and precipitates work together to increase the material's hardness. The matrix is significantly weakened because to the martensite's near-complete decomposition into ferrite and cementite during high-temperature tempering. As a result of the carbide transition, the previously coherent relationship between the ε-carbides, and the martensite matrix becomes fractured. And as a consequence, after tempering at temperatures higher than 380ºC, the effect of the precipitated carbides of the alloying elements (Cr, Mo, and V) compensates for the weakening effect caused by the decomposition of martensite, which results in a slight increase in the steel's hardness as the temperature is raised from 430ºC to 530ºC. The results of Li et al. were verified by the findings of this sample, which demonstrated that CT causes smaller secondary carbides and finer ε-carbides to precipitate in comparison to the samples that were only tempered. The purification of beta-carbides and the precipitation of secondary carbides are two processes that may help in material's impact toughness and strength. Purifying -carbides and precipitating secondary carbides are two steps that may help improve a material's strength and impact toughness. Secondary carbide precipitation helps increase hardness, which is useful.

3.5 XRD analysis

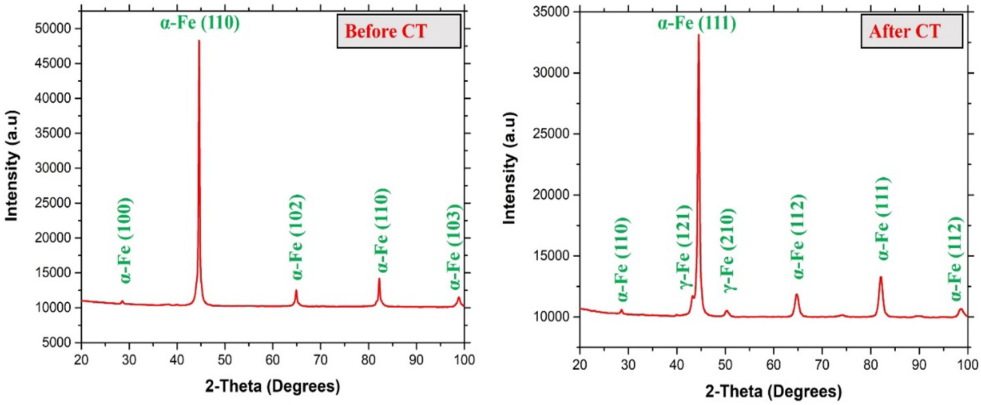


Fig. 10. X-ray diffractograms of before and after CT samples.

Figure 10 represents that X-Ray Diffractograms of CrMoV Low Alloy Steel Before and After Cryo Treatment Samples, which are plotted between Intensity (Cts) on Y-Axis and Position (°2θ) on X-Axis. We could not found Austenite Phase in Un-Treated (Before CT) sample, perhaps due to low content of the retained Austenite. We found only Iron-Alfa phase at highest and Lowest peaks. Iron-Alfa Phase more in Un-Treated (Before CT) sample as compared to Cryogenic Treated (After CT). Sample Uncertainty surrounds the cryogenic technique' role in promoting carbide to precipitate in martensite. As indications, we just have two engineering observations. First, the temperature at which austenite starts to cool and transform into martensite must be much lower than Ms. Second, the holding duration is greater the higher the population and volume proportion of carbides. We propose that tiny internal stress during cooling causes crystal flaws like twins and dislocations. The transformation of martensite from retained austenite and the geographical heterogeneity in composition and microstructure, which results in variable thermal contraction, are twain responsible for this stress. The material must be cooled below a particular temperature in order to induce internal stress in the martensite that is high enough to result in crystal defects. A localized diffusion mechanism, presumably including the grouping of alloying and carbon elements around the flaws, is suggested by the prolonged holding period that is necessary. With a drop in temperature, the martensite becomes more supersaturated.

3.6 Optical micrographs

Optical Microscopy (OM) photographs of untreated are shown in Figure 11 a,b,c, and d and those that were treated with cryogens were shown in Figure 12 a,b,c, and d. From Figure 12(b) blue colour arrow represented the Austenite phase and red colour arrow represented the Martensite phase. From Figure 12(d) white colour arrow represents Austenite phase, yellow colour arrow represents carbides and red colour arrow shows the Martensite phase and also blue coloured arrow represents Grain boundaries. The optical microstructure of tempered CrMoV steel is mostly made up of lath-shaped martensite, retained austenite, and precipitated phases. Additionally, some typical optical photographs of steel that has been tempered at a variety of temperatures are also displayed. After being subjected to tempering, the samples reveal a preponderance of carbides with a strip-like shape as well as a few spherical ones. at the lath contact between martensite grains and at grain boundaries is where carbides are most prone to develop.

When the temperature of the tempering process is elevated to $400^{\circ}C$, the strip-like M7C3carbides and the spherical M23C6carbides both undergo a transformation into M2C-type and MC-type carbides, respectively. The carbides that look like strips are thermodynamically unstable, and they precipitate in the martensite that has been tempered. Steel's tensile strength is determined in large part by its martensitic laths, it has been discovered. The martensitic laths become finer with increased tempering temperature. During tempering, the coarsening of the martensite laths and the removal of lath borders are caused by the recombination of sub grain boundaries. Concurrently, dislocations recover significantly as the tempering temperature rises, resulting in a markedly lower dislocation density and a clearly diminished strengthening effect of dislocations. In addition, the carbides that form during tempering contribute significantly to the steel's durability. Due to their predominance, the strip-like carbides have a direct impact on the steel's mechanical qualities. Because -carbides, which have a mean width of 18 nm, are extensively disseminated in tempered martensite, the matrix is greatly reinforced as long as the tempering temperature is below $200^{\circ}C$. When the temperature of tempering gets around $230^{\circ}C$, however, the -carbides gradually dissolve and transition into M_7C-type and M_2C-type ones. This destroys the coherent relationship between the -carbides and the martensite matrix and greatly weakens the impact of strengthening. The density of precipitation of all carbides can be increased by up to 25% with cryogenic treatment. Additionally, the carbide is often smaller, with an average size reduction of 18%. Figure 13 shows the SEM images of Untreated CrMoV and Cryo treated CrMoV samples.

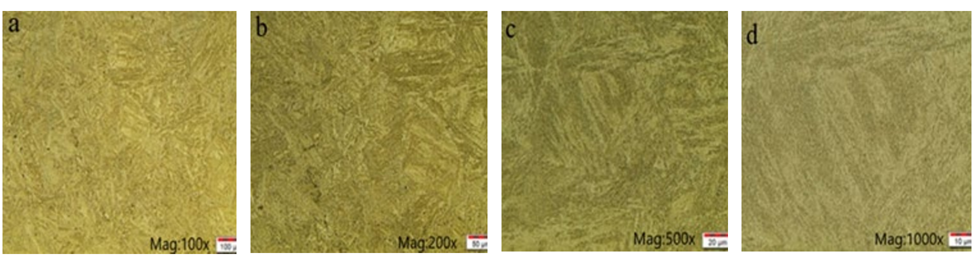
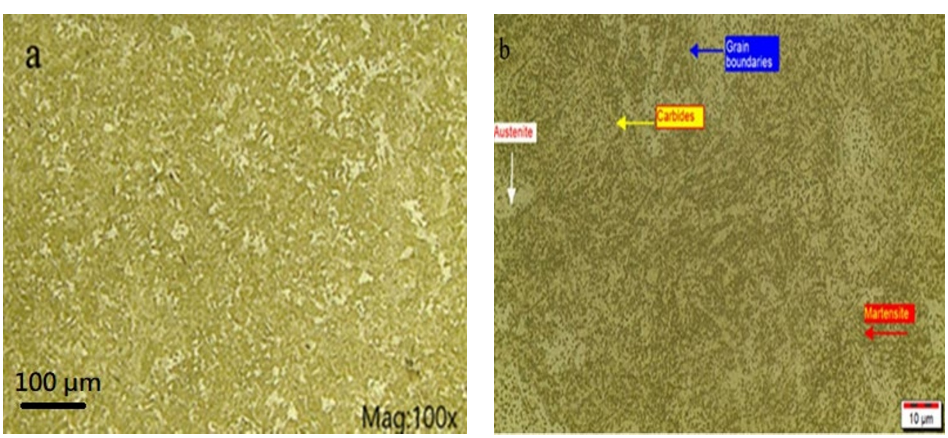


Fig. 11. Optical microstructures of CrMoV low alloy steel prior to cryogenic treatment at (a) 100x (b) 200x (c) 500x , and (d) 1000x.



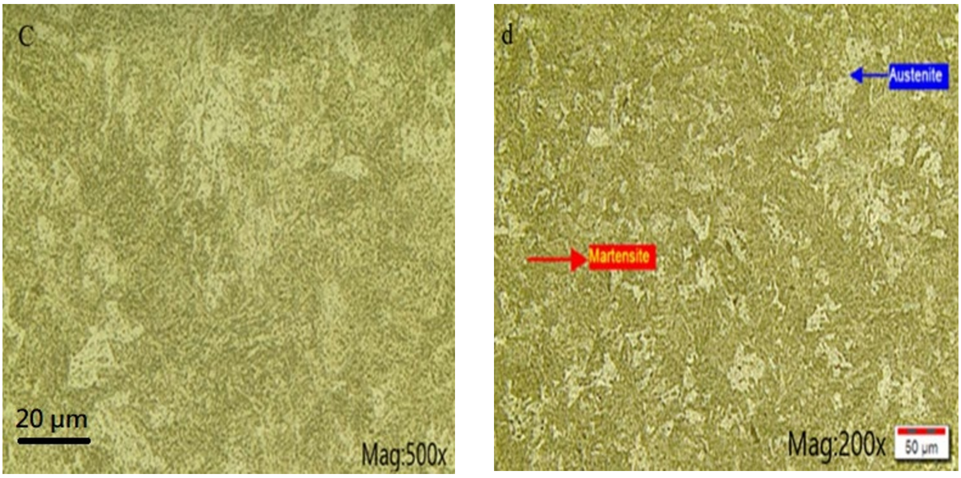


Fig. 12. Optical microstructures of CrMoV low alloy steel after cryogenic treatment at (a) 100x (b) 1000x (c) 500x (d) 200x

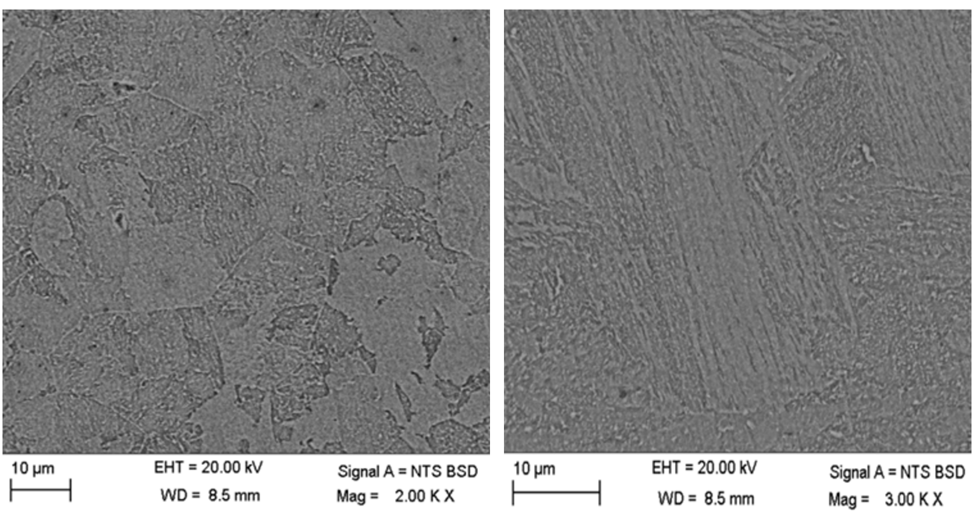


Fig. 13. SEM images (a) Untreated CrMoV sample. (b) Cryo treated CrMoV sample.

4 Conclusion

The results shown that, in contrast to normal operations, cryogenic operations enhance attributes such as hardness, tensile strength, toughness.

- The cryogenically treated tensile samples, followed by tempering, exhibited superior performance compared to untreated samples that were tempered beforehand. The sub-zero treatment with lower temperatures proved to be more successful in enhancing the mechanical qualities.
- The rise in impact toughness values in steel materials is specifically attributed to the formation of primary carbides in the inter-dendritic areas. The interactions between the carbide structures and the matrix play a crucial role in determining

the impact toughness mechanism.

- The hardness and impact values of the samples rose concurrently due to the locking mechanism generated by the carbide structures at the dislocation locations.
- Microstructure of Cryogenically Treated CrMoV specimen shows the presence of Martensite along with some austenite and carbide precipitation in the matrix. The fundamental process behind impact toughness is the alteration of the microstructure, and the impact toughness is affected by the formation of carbide precipitates during cryogenic processing
- Cryogenic temperature-induced microstructure changes facilitate the nucleation of carbides and the conversion of austenite into martensite, which results in the improvement. Further evidence that tempering either before to or after a cryogenic operation significantly affects material characteristics and aids in better carbide dispersion was found in the findings.

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