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Effect of Cryogenic Treatment Duration on the Mechanical and Microstructural Properties of Manganese and Cast Steels

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Abstract

The demand for improved mechanical properties in steel such as hardness, toughness, and dimensional stability—continues to grow across critical sectors like aerospace, automotive, and heavy industries. Conventional heat treatment methods are often limited by issues such as retained austenite and residual stress. This study investigates the impact of cryogenic treatment at –80 °C for 12 and 24 hours on the mechanical and microstructural properties of manganese and cast steels. Mechanical behavior was characterized using Vickers hardness and tensile testing, while microstructural evolution was assessed through Scanning Electron Microscopy (SEM). Results show that a 12-hour cryogenic soak enhances the hardness of manganese steel by approximately 7% and slightly improves yield strength and stiffness. SEM micrographs confirmed the transformation of retained austenite to martensite and significant grain refinement. In contrast, longer soaking (24 hours) led to marginal gains and reduced ductility. Cast steel exhibited more modest improvements due to its lower alloying content. These findings confirm that cryogenic treatment, particularly with optimized exposure time, is an effective and economical method for enhancing the performance of structural steels.

Keywords
Cryogenic Treatment, Manganese Steel & Cast Steel, Vickers Hardness Test, Tensile Strength, SEM

1. Introduction
Steel is a mainstream material for automotive, construction, and aerospace industries due to its machinability, strength, and low cost. Growing demands for durability and performance, however, have revealed the shortcomings of conventional heat treatment processes, which typically leave residual stress and retained austenite, and thus affect mechanical reliability (Du,2024) (Sonar et al., 2018).

Cryogenic treatment is now widely recognized as a supplement process capable of enhancing characteristics like hardness, wear resistance, and dimensional stability. By cooling steel to below-zero temperatures, processes like retained austenite-to-martensite transformation and carbide precipitation can occur, resulting in enhanced microstructural characteristics (Wang et al., 2023) (Rauf Jamali et al., 2019).

Although research verifies its efficacy on different steel grades, the cryogenic soaking time of maximum effectiveness, especially for cast and manganese steel, is not yet well understood (Wei et al., 2024) (Fei et al., 2023). Both these steels find extensive use in high-impact and structural applications, which suit them to explore improvement by cryogenic treatment.

1.1 Objectives

1. **To obtain the optimal cryogenic parameters** to attain an ideal equilibrium between improved mechanical properties and the reduction of thermally induced defects.
2. **To improve the mechanical properties of steels**; hardness, wear resistance, toughness and dimensional stability of cryogenically treated steels.

2. Literature Review

2.1 Theoretical Background

Steel is one of the materials that is most frequently used in engineering and industrial applications due to its exceptional mechanical properties and adaptability. It plays an important role in industries such as automotive, aerospace, construction, and manufacturing. The mechanical properties of steel, including hardness, toughness, wear resistance, and dimensional stability, are crucial for its performance in demanding environments. However, as engineering challenges evolve, there is a growing demand for materials that exhibit superior mechanical performance. Advanced treatment processes, including heat treatment and cryogenic treatment, have been developed to meet these demands by modifying the microstructure of steel.

2.2 Properties of Steel

The performance of steel in industrial applications depends on several critical mechanical properties:

2.2.1 Hardness

Hardness refers to a material's resistance to localized plastic deformation (McKeen, 2016). For steel, hardness increases generally corresponds with improved wear resistance and durability, particularly in applications involving significant friction or contact stress.

2.2.2 Wear Resistance

Wear resistance decides a material's ability to withstand surface damage caused by abrasion, adhesion, or erosion (Shrivastava, 2018). Steel components subjected to high mechanical stresses, such as cutting tools or machine parts, require high wear resistance to prolong their service life.

2.2.3 Dimensional Stability

Dimensional stability is the material's ability to maintain its size and shape under changing temperatures and loads (Gheorghian & Căliman, 2013). This is critical for precision components, as dimensional changes can lead to operational failure or reduced efficiency.

2.2.4 Toughness

Toughness reflects the material's ability to absorb energy and deform plastically before fracturing ("The Basic Properties of Building Materials," 2011). It is particularly important in applications where steel must endure sudden impacts or dynamic loading conditions.The enhancement of these properties is central to improving steel's performance in critical applications. This is achieved by controlling and optimizing the microstructure of the material through advanced treatment processes.

2.3 Cryogenic Treatment

Cryogenic treatment is an advanced metallurgical process that involves exposing steel to extremely low temperatures, typically below -80°C to -196°C, to enhance its mechanical properties. Unlike traditional heat treatments, which involve heating and rapid cooling, cryogenic treatment extends the temperature spectrum to ultra-low levels, inducing unique microstructural transformations.

2.3.1 Stages of Cryogenic Treatment

1. **Cooling Stage:**
   During this stage, steel is gradually cooled from room temperature to cryogenic temperatures to

minimize thermal shock. This controlled cooling prevents structural damage that could arise from rapid temperature changes.

2. **Soaking Stage:**
   The steel is held at the cryogenic temperature for a specific duration. This soaking period allows the microstructural transformations to stabilize, particularly the conversion of retained austenite to martensite and the precipitation of fine carbides.
3. **Warming Stage:**
   After the soaking stage, the steel is slowly cooled to room temperature to avoid thermal stress induced. In some cases, post-treatment processes, such as tempering, are applied to further refine the microstructure (Figure 1).

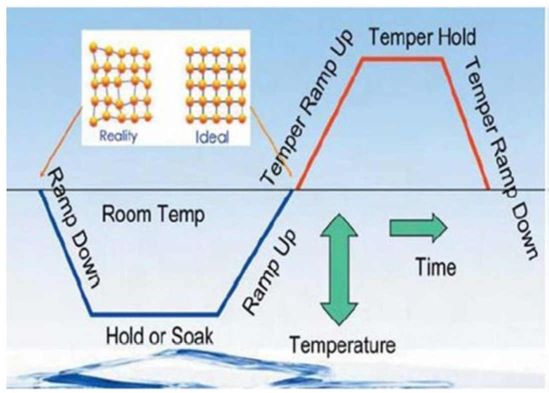


Figure 1. Generalized Cycle for Cryogenic processing (Sonar et al., 2018)

2.4 Microstructural Changes

The effectiveness of cryogenic treatment lies in the microstructural changes it induces within steel. These transformations directly influence the mechanical properties and overall performance of the material:

2.4.1 Transformation of Retained Austenite

Retained austenite refers to a relatively soft phase in steel which can compromise hardness and wear resistance. Cryogenic treatment facilitates the transformation of retained austenite into martensite which is a harder and more stable phase (Fei et al., 2023). This transformation enhances the material's overall strength and wear resistance (Figure 2).

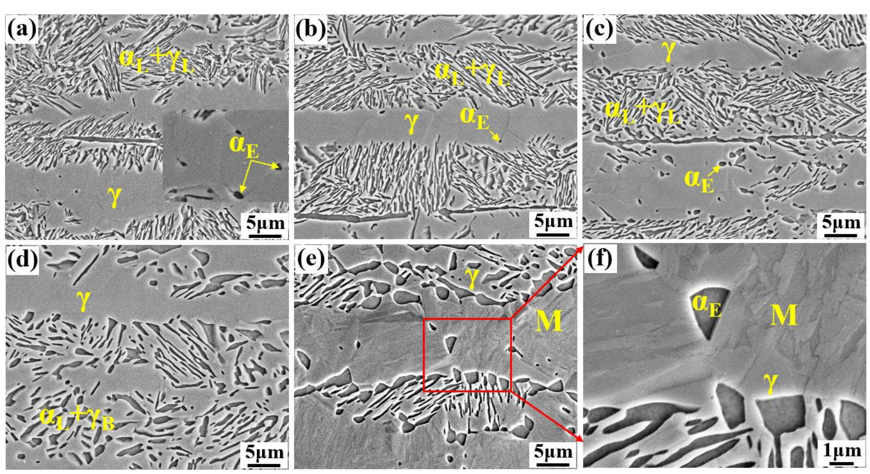


Figure 2. Transformation of retain austenite into martensite (Fei et al., 2023)

2.4.2 Carbide Precipitation

At cryogenic temperatures, the precipitation of fine carbides is promoted. Because of their uniform distribution throughout the steel matrix, these carbides help to improve hardness and improved wear resistance (Rauf Jamali et al., 2019). This phenomenon is particularly beneficial for tools and components exposed to high friction or abrasive conditions (Figure 3).

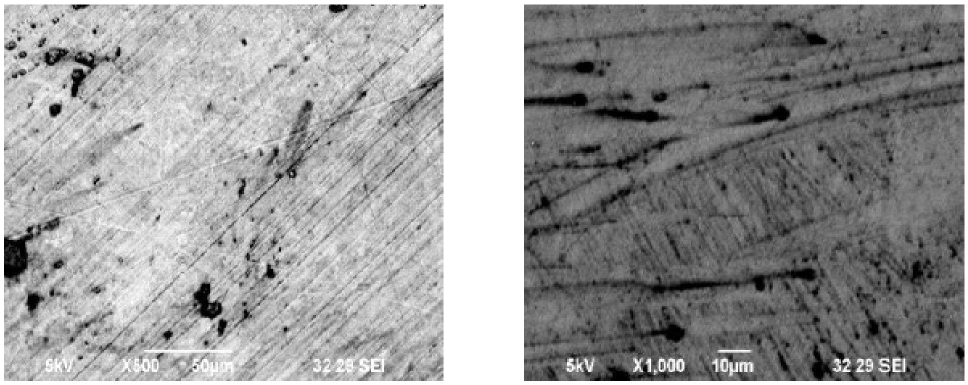


Figure 3. Carbide Precipitation of steels after cryogenic treatment (Rauf Jamali et al., 2019)

2.4.3 Reduction of Residual Stresses

Manufacturing processes often introduce residual stresses within steel, which can lead to distortion, cracking, or premature failure. Cryogenic treatment alleviates these stresses, improving the material's dimensional stability and ensuring long-term reliability (Yang et al., 2024).

These microstructural changes make cryogenic treatment an effective method for enhancing steel's mechanical properties beyond what traditional heat treatments can achieve.

2.5 Review of Previous Studies

Cryogenic treatment has been extensively studied as an advanced metallurgical process for enhancing the mechanical properties of steel. Over the past decades, researchers have explored its effects on various grades of steel, focusing on critical properties such as hardness, wear resistance, dimensional stability, and toughness. These studies showed that cryogenic treatment can offer immense opportunities for improving performance of steels, especially those used in severe industrial applications.

The process is often studied in comparison to conventional heat treatment methods, with findings indicating that cryogenic treatment gives better performance due to more profound changes in the microstructure. However, the degree of improvement varies depending on the steel composition, treatment parameters, and post-treatment conditions. Understanding these factors is essential for optimizing the cryogenic treatment process and tailoring it to specific applications.

This section summarized some of the key findings from previous studies, categorizing them into the effects of cryogenic treatment on mechanical properties, the role of microstructural transformations, and the optimization of process parameters. By analysing existing research, this review aims to highlight the advancements made in the field, identify inconsistencies or gaps in the literature, and establish the basis for further investigation in this study.

2.5.1 Effect of Cryogenic Treatment on Hardness

Cryogenic treatment significantly enhances the hardness of steel through the facilitation of microstructural transformations. The process enables the total conversion of retained austenite into martensite, a more durable and stable phase. Research on high-manganese austenitic steels indicates that cryogenic treatment facilitates the precipitation of fine carbides, thereby enhancing hardness by strengthening the steel matrix (Wang et al., 2023).

In addition, deep cryogenic treatment (DCT) has also demonstrated the ability to refine grain structures which results in increased hardness. As compared to traditional heat treatments, the improvements in hardness obtained through cryogenic treatment are more consistent and enduring due to stabilized microstructures (Fei et al., 2023).

2.5.2 Effect on Wear Resistance

Cryogenic treatment is commonly known for improving the wear resistance of steels. By optimizing grain structures and diminishing residual stresses, it enhances the material's capacity to endure surface damage. For high-manganese steels, cryogenic treatment alters wear mechanisms, reducing material loss during abrasive contact (Du, Yuan, et al., 2024).

Cryogenic treatment promotes the development of fine carbides, thereby increasing wear resistance of steels by creating a harder and more resilient surface layer (Rauf Jamali et al., 2019). Comparative studies reveal that cryogenically treated steels exhibit reduced wear rates relative to conventionally treated steels, especially under high-load conditions.

2.5.3 Impact on Dimensional Stability

Cryogenic treatment improves the dimensional stability of steel by reducing residual stresses induced during production operations (Yang et al., 2024). This is achieved by stabilizing microstructural phases, mainly through the transformation of retained austenite to martensite.

For precise applications, such as in aerospace or tool manufacture, dimensional stability is important to avoid distortions while in use. Studies demonstrate that cryogenic treatment performs better than conventional heat treatments in maintaining dimensional accuracy over extended service times.

2.5.4 Influence on Toughness

The influence of cryogenic treatment on toughness changes depending on the steel composition and treatment conditions. In high-manganese steels, cryogenic temperatures boost toughness by accelerating the production of deformation twins and increasing dislocation density, which improves the material's ability to absorb energy before fracturing (Wei et al., 2024).

However, at ultra-low temperatures, excessive carbide precipitation can occasionally weaken toughness, demanding careful adjustment of cryogenic treatment parameters (Rauf Jamali et al., 2019). Comparative studies with conventional heat treatment demonstrate that cryogenic treatment generally offers a better balance between hardness and toughness, especially for steels utilized in dynamic or impact-prone environments.

2.5.5 Comparative Studies

Cryogenic treatment provides certain advantages over conventional heat treatment in overcoming limitations such as incomplete phase transformation and residual stresses. Although conventional treatments such as quenching and tempering is very effective in improving hardness, these processes often leave retained austenite that can compromise wear resistance and dimensional stability. (Huang et al., 2024).

Cryogenic treatment extends the temperature spectrum to ultra-low levels, ensuring more complete phase transformations that improves properties like wear resistance, hardness, and toughness. Studies show that cryogenically treated steels demonstrate superior performance in industrial applications, particularly in terms of wear resistance and long-term dimensional stability.

Moreover, the combination of cryogenic treatment with other processes, such as intercritical annealing or intensive quenching, can further refine microstructures and enhance mechanical properties, offering a more versatile and effective approach than conventional methods alone (Fei et al., 2023).

2.6 Optimization of Cryogenic Treatment Parameters

Optimization of the cryogenic treatment parameters is important for the required mechanical properties enhancement in steel. In this study, the main influencing parameters of the treatment process are the cryogenic temperature, soaking time, and post-treatment processes. Every parameter has a significant role in deciding the extent of the different microstructural transformations induced and the resultant material properties.

2.6.1 Soaking Duration

The soaking duration is the time that the steel is held at cryogenic temperatures which affects the uniformity and completeness of microstructural transformations.

- Short Soaking Times (<1 Hour):

Limited transformations occur, leading to partial conversion of retained austenite and less significant improvements in mechanical properties (Fei et al., 2023).

- Optimal Soaking Times (1–24 Hours):

Most studies recommend soaking durations in this range to allow sufficient time for austenite-to-martensite transformation and carbide precipitation (Wei et al., 2024).

- Extended Soaking Times (>24 Hours):

Longer durations can improve property enhancement but may have diminishing returns in terms of cost-effectiveness and energy consumption (Du, Hu, et al., 2024).

2.6.2 Combination with Other Treatments

The integration of cryogenic treatment with other thermal processes, such as intensive quenching or intercritical annealing, can further enhance mechanical properties.

- Intensive Quenching (IQ):

Preceding cryogenic treatment with IQ promotes grain refinement and the formation of uniform martensite, which enhances hardness and toughness (Fei et al., 2023).

- Intercritical Annealing:

Combining cryogenic treatment with intercritical annealing stabilizes retained austenite and optimizes strain-hardening behavior through the TRIP effect (Fei et al., 2023).

2.7 Research Gap

Properties of steel, including hardness, wear resistance, toughness, and dimensional stability. While substantial work has been made in understanding its effects and adjusting its parameters, some problems remain. Existing research frequently focuses on certain steel grades or limited parameters, leaving gaps in the overall understanding of the treatment's processes, scalability, and long-term performance. Addressing these limitations is critical for promoting cryogenic therapy as a diverse and cost-effective solution for industrial applications.

2.7.1 Incomplete Understanding of Microstructural Transformations

While the transformation of retained austenite to martensite and carbide precipitation are well-documented effects of cryogenic treatment, the exact mechanisms governing these transformations under varying cryogenic parameters remain insufficiently understood.

For high-manganese steels, there is limited clarity on how cryogenic treatment impacts grain coarsening and its interaction with deformation mechanisms like twinning-induced plasticity (TWIP) and stacking fault energy (SFE).
The role of cryogenic treatment in controlling the mechanical stability of retained austenite in medium-manganese lightweight steels needs further investigation, particularly in conjunction with post-treatment processes like intercritical annealing.

2.7.2 Trade-Offs Between Properties

Cryogenic treatment enhances hardness and wear resistance but may reduce toughness or increase grain coarsening, particularly in high manganese steels.

There is a need for systematic studies that optimize cryogenic parameters to achieve an ideal balance between hardness, toughness, and wear resistance.

2.7.3 Lack of Industrial Feasibility Studies
Most research is conducted on a laboratory scale, with limited exploration of the economic feasibility and scalability of cryogenic treatment for industrial applications.The high cost of ultra-cryogenic media (e.g., liquid nitrogen) and extended treatment durations poses challenges for widespread adoption in industries.

3. Methods
3.1 Material Selection and Preparation
The cast steel and manganese steel materials procured commercially and utilized in the research were selected because of their toughness and their potential for industrial application. The two materials were machined to the standard test specimens for tensile and hardness tests per ASTM requirements. The specimens underwent preliminary normalization prior to being cryogenically treated to give a consistent baseline microstructure (Table 1 and Table 2).

Table 1. Material Composition of Manganese Steel

Element	C	Mn	Si	P	S	Fe
Wt. %, min	1.05	11.00	0.30	-	-	Bal.
Wt. %, max	1.35	14.00	1.00	0.07	0.05	Bal.

Table 2**.** Material Composition of Cast Steel

Element	C	Mn	Si	P	S	Fe
Wt. %, min	0.15	0.50	0.20	-	-	Bal.
Wt. %, max	0.25	1.00	0.80	0.04	0.05	Bal.

3.2 Cryogenic Treatment Process
The cryogenic treatment was performed within a New Brunswick Innova U725 ultra-low temperature freezer with the environment held constant at –80°C. Two treatment times of 12 hours and 24 hours were selected. Specimens were cooled slowly to avoid thermal shock, soaked at the desired condition, and then brought back to ambient temperature under controlled conditions without tempering to isolate the cryogenic effects (Figure 4).

Figure 4. Cryogenic Freezer

3.3 Mechanical Testing
3.3.1 Vickers Hardness Test
In this study, hardness was tested by a Vickers Hardness Tester under 30 kgf load in accordance with ASTM E384. Three indentations were induced on each specimen and averaged for consistency. After indentation, the diagonals of the resulting impression are measured using a microscope, and the Vickers Hardness Number (VHN) is calculated.
The Vickers Hardness Number was calculated using the following formula (Figure 5):

$$HV = \frac{1.8544 \times F}{d^2}$$

Where HV is the Vickers Hardness Number, F is the applied force in kilogram-force (kgf) and d is the average length of the two diagonals of the indentation in millimetres (mm).


(a)


(b)

Figure 5. (a) Vickers Hardness Machine; (b) Specimen undergoes Vickers Hardness Test

3.3.1 Tensile Test

Tensile behaviour was quantified with the help of INSTRON 600DX Universal Testing Machine following ASTM E8 standard test practices. Flat dogbone specimens of 50 mm gauge length and 12.5mm width were utilized to ensure stress distribution during the test. Critical parameters recorded were Ultimate Tensile Strength (UTS), yield strength (0.2% offset), Young's modulus, and elongation at break (Figure 6).


(a)


(b)

Figure 6. (a) Universal Testing Machine INSTRON 600DX (b) Flat Dog Bone specimen for Tensile Test

3.4 Microstructural Analysis

Scanning Electron Microscopy (SEM) was applied to examine the microstructural change before and after cryogenic treatment. Etched specimens were analyzed to verify the refinement in grains, phase transformation (martensite from austenite) and precipitation of carbides(Figure 7).

Figure 7. SEM Machine

4. Results and Discussion

4.1 Vickers Hardness

The Vickers hardness tests registered a clear rise in hardness following the cryogenic treatment, particularly in the case of manganese steel. In untreated manganese steel, the mean hardness was 126.57 HV, which increased to 132.57 HV after 12 hours and 135.53 HV after 24 hours of cryogenic soaking at −80 °C. This is a predicted 7% improvement for which credit goes to retained austenite transformation into martensite and fine carbide precipitation (Du et al., 2024) (Wang et al., 2023). Both martensitic transformation and low stacking fault energy are promoted by high carbon and manganese content and add to the hardness through microstructural refinement.

On the other hand, cast steel with lower alloying content showed minimal improvement in hardness—from 129.50 HV untreated to 132.07 HV treated for 24 hours. The low rise (~2%) agrees with previous research [6], who reported low hardening in low retained austenite steels. This suggests that the cryogenic treatment effect is highly dependent on steel composition, particularly the availability of transformable phases and elements promoting carbide formation (Table 3 and Table 4).

Table 3. Result of Vicker Hardness Test of Manganese Steel

Condition	HV1	HV2	HV3	HVavg
Untreated	125.20	126.00	128.50	126.57
12hrs Cryogenic	132.20	133.80	131.70	132.57
24hrs Cryogenic	137.30	132.90	136.40	135.53

Table 4. Result of Vicker Hardness Test of Cast Steel

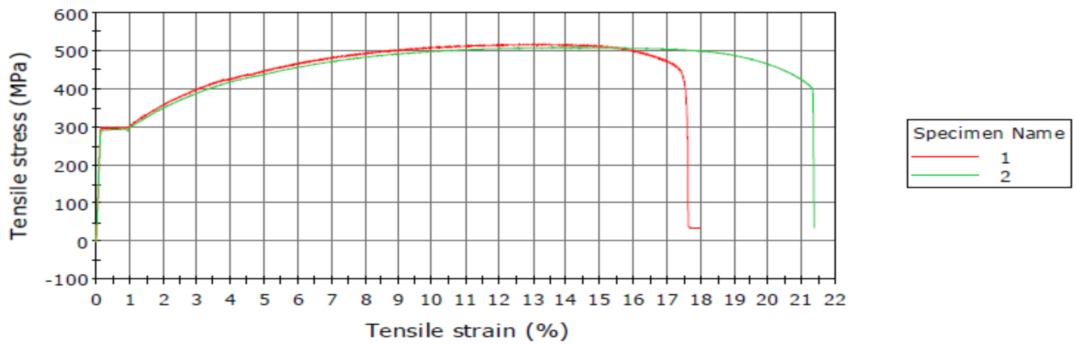
Condition	HV1	HV2	HV3	HVavg
Untreated	131.00	128.00	129.50	129.50
12hrs Cryogenic	131.10	129.80	133.10	131.33
24hrs Cryogenic	133.60	130.50	132.10	132.07

4.2 Tensile Properties

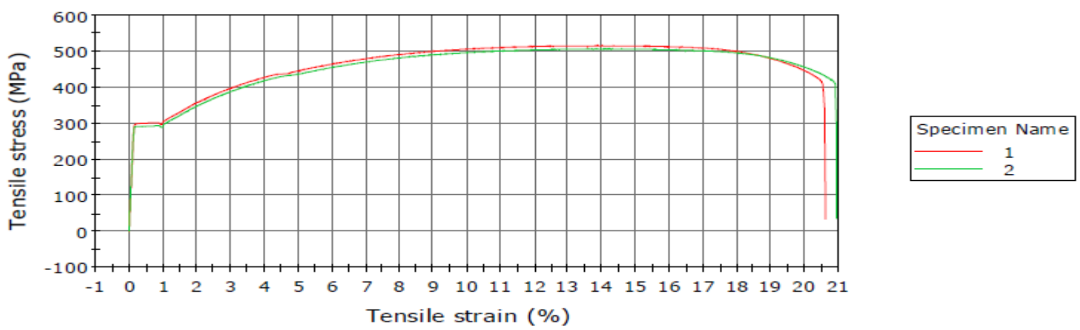
4.2.1 Manganese Steel

Tensile testing revealed ultimate tensile strength (UTS) was unaffected in treatments: 513.02 MPa (untreated), 511.04 MPa (12 hrs), and 511.47 MPa (24 hrs), which shows that cryogenic treatment will have no adverse effect on peak load-carrying capacity. Yield strength, though, did rise barely at all with treatment—from 294.68 MPa to 296.82 MPa—due to pinning of dislocations and precipitation of retained austenite to martensite (Fei et al., 2023). Young's modulus also increased from 215.05 GPa (untreated) to 221.55 GPa (24 hrs), reflecting improved stiffness (Yang et al., 2024).

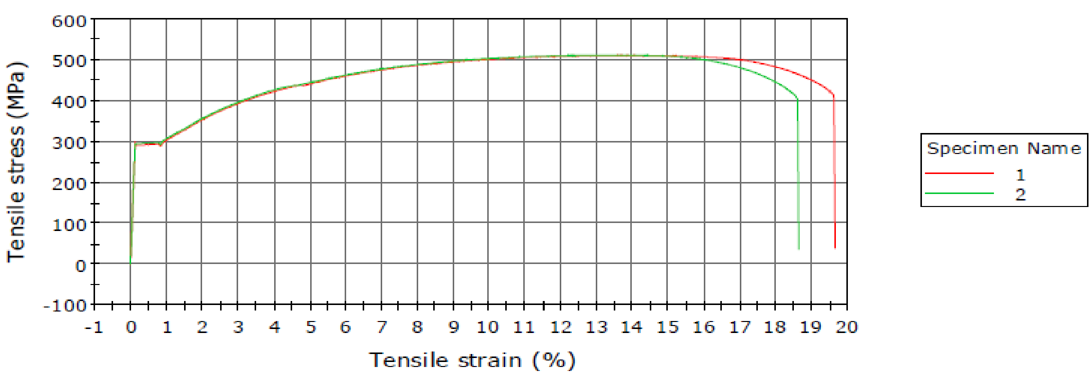
Notably, ductility was increased slightly after 12 hours but later reduced after 24 hours, as witnessed in the decrease of elongation at break (from 3.54 mm to 3.37 mm) (Figure 8). The trend indicates that over-cryogenic soaking can cause over-hardening (Table 5), which undermines toughness (Rauf Jamali et al., 2019).



(a) Untreated



(b) 12hrs Cryogenic



(c) 24hrs Cryogenic

Figure 8. Stress-Strain Curves of Manganese Steel

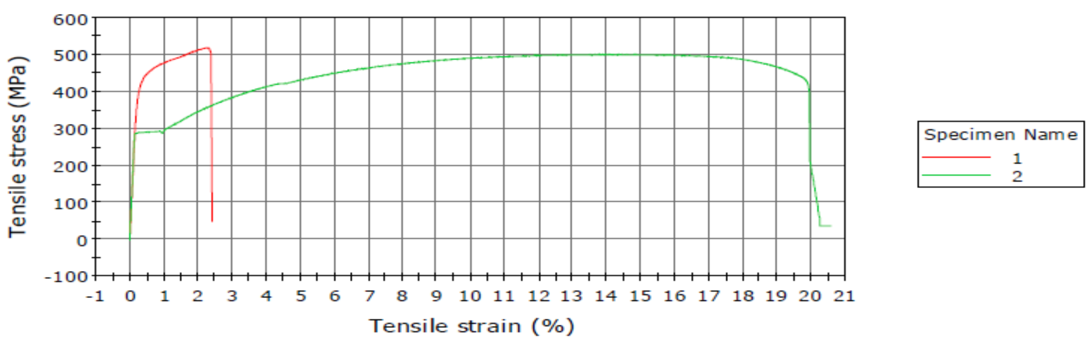
Table 5. Result of Tensile Test of Manganese Steel

Condition	UTS (MPa)	Yield Strength (MPa)	Young's Modulus (GPa)	Strain at Max Load (mm/min)	Elongation at Break (mm)
Untreated	513.02	294.68	215.05	0.1367	3.42
12hrs Cryogenic	511.04	296.82	209.72	0.1415	3.54
24hrs Cryogenic	511.47	295.06	221.55	0.1349	3.37

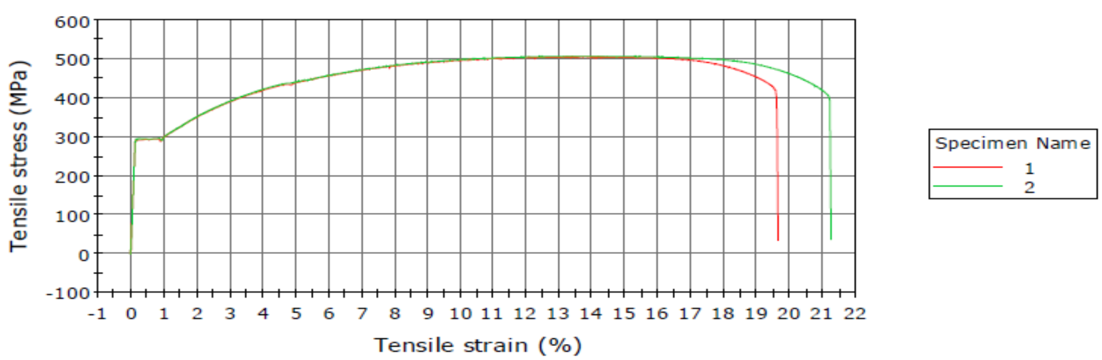
4.2.2 Cast Steel

For cast steel, UTS increased slightly from 500.56 MPa to 506.31 MPa after 12 hours but decreased to 496.60 MPa after 24 hours. Yield strength also followed the same trend. The results show that there is a small strengthening effect of the exposure by cryogenic, but indicate potential embrittlement with prolonged treatment. Young's modulus increased from 207.83 GPa to 217.98 GPa as anticipated, with increased structural stiffness (Table 6).

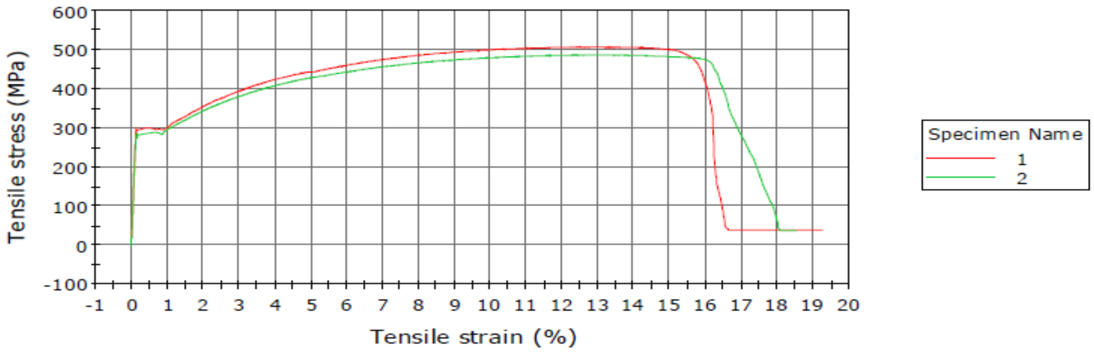
Ductility, in terms of strain at maximum load and elongation at break (Figure 9), declined monotonically with rising cryogenic exposure. This highlights the trade-off between ductility and hardness/stiffness again to maximize treatment time to more equally balance these properties (Du et al., 2024).



(a) Untreated



(b) 12hrs Cryogenic



(c) 24hrs Cryogenic

Figure 9. Stress-Strain Curves of Cast Steel

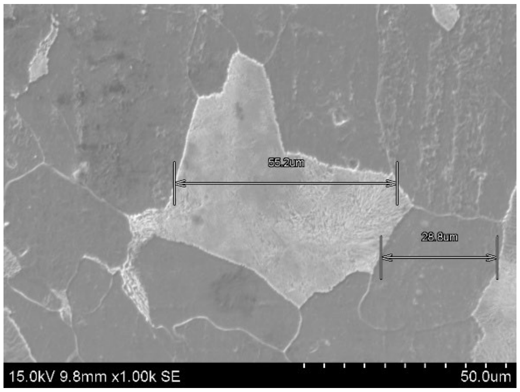
Table 6. Result of Tensile Test of Cast Steel

Condition	UTS (MPa)	Yield Strength (MPa)	Young's Modulus (GPa)	Strain at Max Load (mm/min)	Elongation at Break (mm)
Untreated	500.56	290.68	207.83	0.1428	3.57
12hrs Cryogenic	506.31	294.93	217.98	0.1351	3.38
24hrs Cryogenic	496.6	291.98	216.78	0.1319	3.30

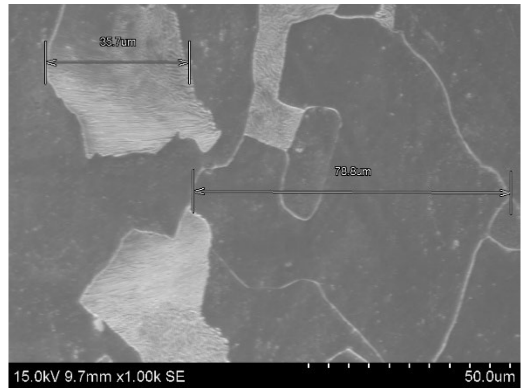
4.3 SEM Microstructure Correlation

SEM micrographs confirmed grain refinement and phase transformation in the two steels after 12 hours cryogenic treatment. Tapered grain boundaries and martensite formation indications were shown in manganese steel, in accordance with indications of enhanced hardness and stiffness. Partial grain refinement and non-uniform grain morphology with bimodal grain size distribution (31.5 µm and 85.3 µm) were present in cast steel, in accordance with moderate mechanical improvement.

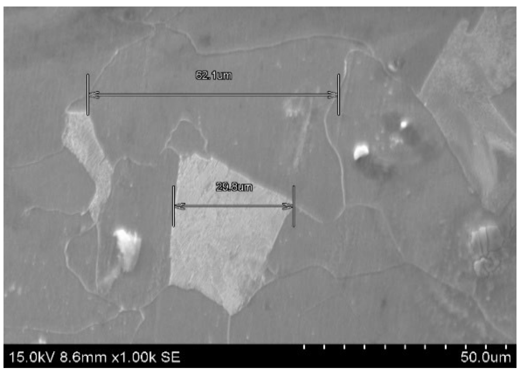
The Hall–Petch relationship of smaller grains corresponding to higher strength is in agreement (Wei et al., 2024). In both cases (Figure 10), cryogenic treatment promoted uniformity and reduced microstructural defects, though to varying degrees based on composition.



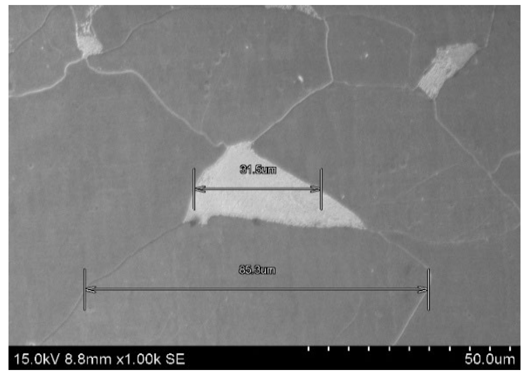
[1] Untreated



[2] 12hrs Cryogenic



[3] Untreated



[4] 12hrs Cryogenic

Figure 10. SEM Micrograph: (a) and (b) Manganese Steel, (c) and (d) Cast Steel

4.4 Proposed Improvements

To complement this research, several areas are proposed for additional study. Firstly, the use of advanced characterization techniques such as X-ray Diffraction (XRD), Electron Backscatter Diffraction (EBSD), and Transmission Electron Microscopy (TEM) is strongly suggested to more precisely identify the phases after treatment and confirm the extent of retained austenite transformation and carbide formation.Second, a wider range of cryogenic soaking times, including shorter durations (e.g., 6 or 8 hours) and intermediate durations (e.g., 18 hours and 48 hours), can be examined with a view to establishing the kinetics of the transformation and determining the saturation point for property enhancement. Stepwise cooling or controlled ramp-down cycles, rather than straight putting in cryogenic freezer, can also be examined in future work.

Thirdly, continuation of the mechanical testing regimen to impact toughness, fatigue strength, and wear analysis at different service temperatures would provide a better understanding of the effect of cryogenic treatment on long-term performance and durability in real service conditions.

Lastly, replication of this study on other steel alloys—like high-carbon tool steels or stainless steels—would help to broaden the generality of applicability of cryogenic treatment to a broader industrial material base.

6. Conclusion

Results indicate that cryogenic treatment significantly enhances manganese and cast steel mechanical and microstructure properties. In manganese steel, hardness was enhanced by an estimated 7% after 24 hours of treatment through retained austenite transformation to martensite and precipitation of fine carbides. Cast steel showed much smaller improvements due to its lower carbon and alloying content.

Tensile tests revealed that cryogenic treatment maintained or even increased the strength and rigidity of the two steels. Of special interest, 12-hour treatment provided the best strength to ductility ratio, and 24-hour treatment produced minimal losses in formability due to over-hardening.

SEM analysis confirmed grain refinement and microstructural alteration associated with the mechanical improvements observed. Typically, 12 hours cryogenic soaking at −80 °C is found to be optimum, yielding better performance with minimal sacrifice of ductility.

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