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Effect of Sub-Zero Treatment on Microstructure, Retained Austenite, and Hardness of HSLA Steel for Automotive Applications

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Abstract: This research focused on analyzing the effect of high cooling rates at cryogenic temperatures, referred to as sub-zero treatment, on the retained austenite fraction in the High Strength Low Alloy steel during the hardening heat treatment process. Sub-zero treatment was carried out after the quenching process. The treatment involved cooling the steel to $-176\text{ }^{\circ}\text{C}$, followed by a double tempering process. This study focused on analyzing the percentage of retained austenite in as-quenched and as-subzero-treated samples, as well as in as-quench double-tempered (QTT) and as-quench sub-zero double-tempered (QSTT) samples. Furthermore, the hardness of all treatment variables is also compared. The microstructure characterization was carried out using an optical microscope and a Scanning Electron Microscope (SEM), and the calculation of the percentage of retained austenite phase was performed using ImageJ software. Hardness testing was conducted using Rockwell macro hardness testing. The results of this study show that a decrease in the amount of retained austenite from 6.8% to 3.9% occurs in as-quenched samples without and with sub-zero treatment. After a double temper, the retained austenite decreases from 3.37% to 1.2% in as-QTT and as-QSTT samples. Additionally, there was an increase in hardness from 48 HRC to 49 HRC in the sample with double temper treatment. Thus, sub-zero treatment could reduce the percentage of residual austenite and increase the hardness of the HSLA steel.

Keywords: High Strength Low Alloy; Quenching; Retained Austenite; Sub-zero treatment

1. Introduction

High-strength low-alloyed steels (HSLA steels), also referred to as micro-alloyed steels, are a class of materials engineered to deliver superior mechanical performance and enhanced resistance to atmospheric corrosion when compared to conventional carbon steels. Unlike ordinary alloy steels, HSLA steels are specifically designed to achieve targeted mechanical properties rather than strictly adhering to a fixed chemical composition¹⁾. These steels typically exhibit yield strengths exceeding 275 MPa (or 40 ksi), making them suitable for a broad range of structural and engineering applications^{1,2)}. HSLA steels are characterized by relatively low carbon content (typically 0.05–0.25% C), ensuring adequate formability and weldability. Their compositions are further enhanced by microalloying elements such as niobium, vanadium, and titanium, which contribute to grain refinement and precipitation strengthening^{3–5)}.

Despite their favorable mechanical performance, HSLA steels, like other alloyed steels, are susceptible to the formation of retained austenite. Retained austenite is a metastable phase that can significantly influence service behavior and refers to the portion of austenite that fails to transform into martensite during quenching. This retained phase may remain stable under certain conditions but can transform into martensite under external stress, strain, or temperature fluctuations, resulting in delayed cracking and dimensional instability in service^{6,7)}. The volume fraction and morphology of retained austenite are influenced by several factors, including the steel's chemical composition, austenitizing temperature, quenching rate, and tempering parameters⁸⁾. In engineering applications, the presence of excessive retained austenite is often undesirable due to its role in reducing dimensional stability and increasing the likelihood of microcrack initiation over time, particularly in fatigue-loaded or thermally cycled components⁹⁾. Retained austenite plays a critical role in determining the

mechanical stability of steels, particularly under cyclic loading and thermal fluctuations. Retained austenite stability is governed by mechanical, chemical, and thermal factors, including carbon enrichment, morphology, and surrounding phase constraints¹⁰⁻¹³. From a thermodynamic perspective, the incomplete transformation of austenite during quenching is closely related to the martensite start (Ms) and martensite finish (Mf) temperatures. In many alloyed steels, the Mf temperature lies below room temperature, resulting in retained austenite after quenching¹⁴⁻¹⁷. The Koistinen-Marburger relationship describes the fraction of martensite formed as a function of undercooling below the Ms temperature^{18,19}. Its application to high-strength and other low-alloy steel demonstrated that cooling conditions below Ms strongly influenced the extent of martensitic transformation²⁰⁻²².

One well-established method to reduce the retained austenite content is sub-zero treatment, also known as cryogenic treatment. This process involves cooling the material to sub-ambient temperatures, typically ranging from -80 °C and up to -196 °C, depending on the martensite finish temperature (Mf) of the steel²³. Cryogenic treatment enhances martensitic transformation by increasing the driving force for nucleation and suppressing diffusion, while also promoting fine carbide precipitation during subsequent tempering²⁴⁻²⁷. Sub-zero treatment facilitates the further transformation of metastable austenite to martensite that was not achieved during conventional quenching, thereby increasing hardness, wear resistance, and dimensional stability^{28,29}. Studies have demonstrated that the effectiveness of sub-zero treatment is influenced by several variables, including the cooling rate, holding time, and final sub-zero temperature³⁰. When optimized, sub-zero treatment can refine the microstructure, improve hardness homogeneity, and reduce the incidence of delayed cracking caused by austenite decomposition during service³¹⁻³³. Moreover, subsequent tempering after sub-zero treatment can stabilize the newly formed martensite and reduce internal stresses, which is critical for achieving the desired mechanical performance³⁴.

Although sub-zero treatment has been extensively investigated in high-carbon and tool steels such as AISI D2 and M2^{3,28,35}, its application in HSLA steels remains relatively underexplored. Compared to tool steels, HSLA steels exhibit lower carbon content and different retained austenite stability, making the application of sub-zero treatment less explored and requiring further investigation^{36,37}. Paper on HSLA alloys commonly address the cryogenic service behavior³⁸. Given the growing importance of dimensional stability and fatigue resistance in structural applications, this gap represents a critical opportunity. Therefore, this study aims to

investigate the effect of sub-zero treatment on retained austenite reduction and hardness evolution in HSLA steel, with particular emphasis on its potential application for improving dimensional stability and reducing cracking susceptibility in structural and automotive components. By combining microstructural characterization with macrohardness measurements, the work provides new insights into how cryogenic processing can be adapted to optimize the phase stability and mechanical performance of HSLA steels.

2. Experiment Procedures

The material used in this research is High-Strength Low-Alloy (HSLA) steel, which is often applied to components of heavy equipment vehicles, with the composition as shown in Table 1.

The heat treatment process stage for this research was first carried out, starting with sample preparation, which involved cutting the sample to 40x40x1 mm. Furthermore, normalization is carried out at a temperature of 970 °C for 1 hour, followed by a pre-tempering process at 677 °C. After this, austenization is carried out for this study at a temperature of 926 °C with a holding time of 1 hour. Quenching in a water medium with 1% Polyalkylene Glycol (PAG) will reduce the sample temperature from the austenitizing temperature of 926 °C to room temperature. This treatment prevents the material from cracking and reduces distortion from extreme temperature changes. After quenching, the treatment was continued with sub-zero or cryogenic treatment. Sub-zero treatment was carried out at a temperature of -176 °C for 1 hour. The sub-zero treatment temperature of -176 °C was selected to facilitate further martensitic transformation below the estimated martensite finish (Mf) temperature of the steel. In HSLA and other alloyed steels, incomplete transformation during conventional quenching may occur because the Mf temperature can be lower than room temperature, leading to retained austenite formation. By applying sub-zero treatment significantly below room temperature, the driving force for martensitic transformation increases, enabling the transformation of metastable retained austenite into martensite and reducing the retained austenite fraction in the final microstructure. After that, it was double tempered at a temperature of 205 °C for 4.5 hours each. Samples that have been heat treated will produce steel products, namely as-QTT and as-QSTT.

Table 1: Chemical Composition of HSLA Steel

Element	C	Si	Mn	P	S	Ni
%	0.28	1.40	0.95	0.025	0.009	0.70
Element	Cr	Mo	Ca	Cu	Al	
%	1.80	0.27	0.003	0.02	0.035	

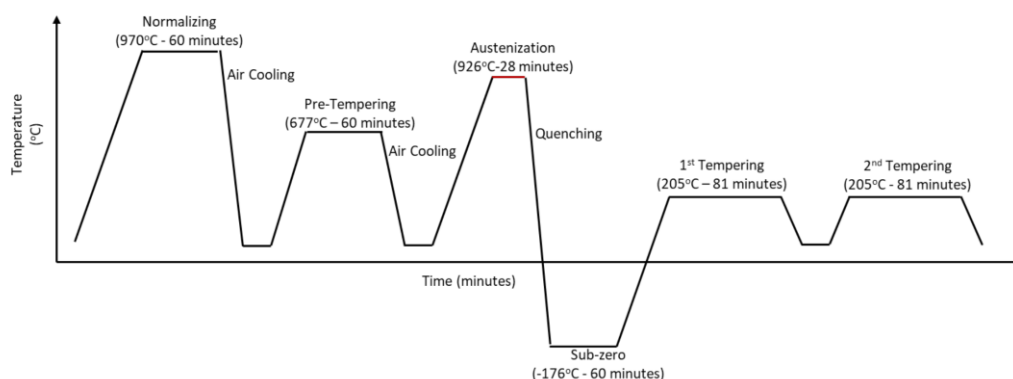


Fig. 1: Schematic diagram of the heat treatment carried out

The microstructure characterization was carried out by using an optical microscope and Scanning Electron Microscope (SEM), and the calculation of the percentage of the retained austenite phase using software of Image J. The retained austenite fraction was estimated using ImageJ image analysis based on the contrast differences observed in the microstructure. This method was employed primarily for comparative evaluation among different treatment conditions and to observe relative trends in retained austenite evolution after sub-zero treatment. Although image-based analysis provides useful microstructural information, the quantitative values obtained should be interpreted as semi-quantitative estimations due to limitations associated with phase contrast sensitivity, image thresholding, and localized observation areas. To improve measurement reliability and minimize localized observation bias, the analysis was conducted using a minimum of five measurements obtained from three different microstructure images for each treatment condition. The reported retained austenite values represent the average of these measurements, while the error bars shown in the corresponding graph indicate the variation of the measured data. Hardness testing was carried out using macro hardness testing, Rockwell C. Heat treatment carried out in this study can be briefly seen in the flow chart in Figure 1.

3. Results and Discussion

3.1. Microstructure Analysis by OM

The microstructure of the material was observed at the surface and the core of the sample. The aim is to observe the homogeneity of the microstructure on the surface and in the core, including if there is decarburization on the surface of the sample. Decarburization is the loss of Carbon on the surface of the material. The factors that influence the occurrence of this phenomenon are high temperatures, a low concentration of Carbon around the material, and a high concentration of oxygen in the atmosphere.

Prawoto et al. and Hui Wang et al.^{39,40)} identified the phenomenon of decarburization in steel (0.8 wt% C). Ferrite was found on the surface in the white/light area, and mixed pearlite and Widmanstätten ferrite in the dark area towards the core. At high temperatures, the rate of carbon diffusion from the material to the environment is very high, resulting in decarburization. Carbon atoms diffuse from the bulk material toward the surface⁴¹⁾. In Figure 2, there is no decarburization phenomenon found in all samples, which usually forms a ferrite phase on the surface. In general, the surface of the sample is dominated by the formation of a transformation zone.

The transformation zone is an area where the bainite has not fully transformed⁴²⁾. The transformation zone has a high hardness, ranging from 500 to 520 HV. The high hardness is due to the Transformation zone consisting of fresh martensite and/or retained austenite and bainite that form around it⁴²⁾. Based on this, it can be assumed that the bright phase formed in the microstructure of as-subzero consists of a mixture of martensite with retained austenite and bainite with retained austenite. Figure 3 shows the microstructure of the core parts of the as-quenched, as-subzero, as-QTT, and as-QSTT samples, which were previously austenized at a temperature of 927 °C and quenched with water and 1% Polyalkylene Glycol.

Figure 3(a) and (b) illustrate the similarities and differences in the microstructures formed from the as-quenched and as-subzero samples. The presence of martensite, bainite, and austenite phases characterizes both samples. The microstructure is dominated by martensite because the rapid cooling rate suppresses carbon diffusion. However, it can be seen that the difference exists in the martensite structure that is formed, where the as-subzero product has a finer martensite structure (i.e., it shrinks) and is more uniformly dispersed. Similar martensitic refinement after sub-zero treatment has also been reported by Bhawar et al.⁴³⁾. The refinement of martensitic structure observed in sub-zero treated samples can be attributed to increased nucleation density and restricted carbon diffusion at low temperatures, resulting in finer lath or

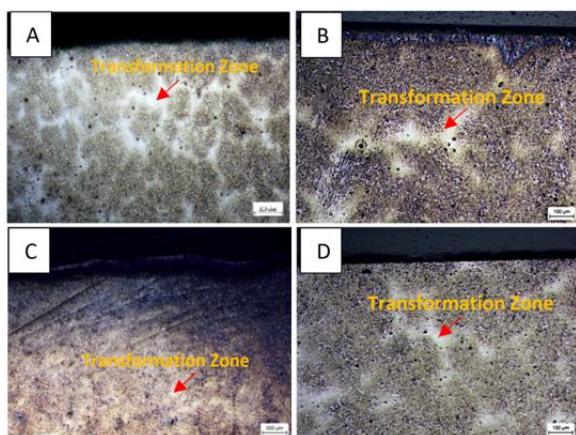


Fig. 2: Microstructure on the surface of HSLA steel (a) as-quenched (b) as-subzero, (c) as-QTT, and (d) as-QSTT. Nital 5%

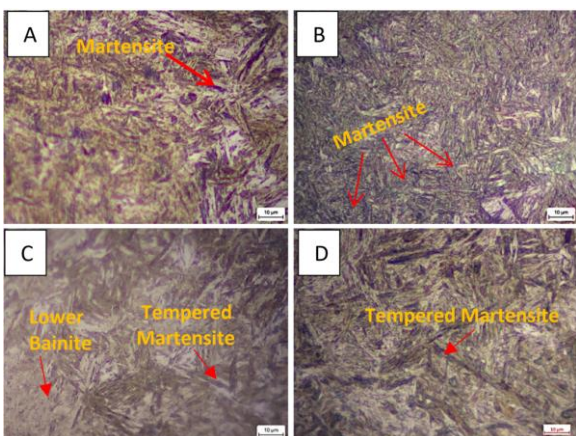


Fig. 3: Microstructure on the core of HSLA steel (a) as-quenched (b) as-subzero, (c) as-QTT, and (d) as-QSTT. Nital 5%

plate structures^{34,44,45}). Additionally, the reduction in temperature suppresses atomic mobility, leading to a more homogeneous distribution of martensite and retained austenite phases⁴⁶⁻⁴⁹).

Figure 3(c) and (d) show the differences in the microstructure formed from the as-QTT and as-QSTT samples; both samples exhibit the presence of tempered martensite and bainite phases. However, in general, the difference is evident in the formation of coarser martensite in the QSTT sample. These results are consistent with research conducted by Speich et al.⁶, who observed morphological differences in the martensite phase between the as-QTT and as-QSTT samples. Speich et al.⁶, have demonstrated that the microstructural evolution during tempering involves the coarsening of martensite and the precipitation and growth of carbides. According to the morphological comparison of the two samples, the microstructure of the samples treated with cryogenic showed a very coarse martensite structure (coarse).

3.2. Microstructure Analysis by SEM

SEM results of the sample microstructure, as-QTT, as-

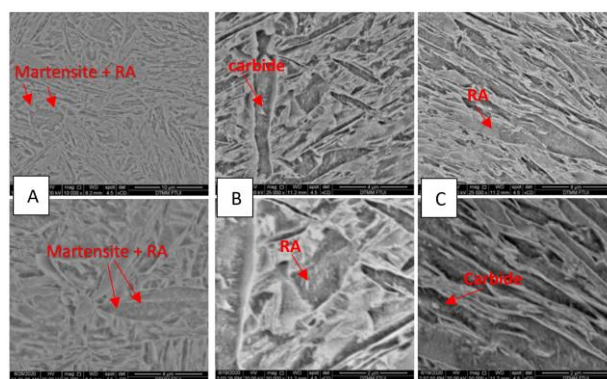


Fig. 4: SEM micrograph of the HSLA steel: (a) as-QTT (b) as-subzero, (c) as-QSTT (25000x and 50000x, nital 5%). RA indicates Retained Austenite

subzero, and as-QSTT, are shown in Figure 4. The Figure displays the SEM results of the martensite, bainite, and transformation zone areas. This is consistent with the resulting microhardness measurements of phases on the as-subzero, as-QTT, and as-QSTT microstructure samples. Phase determination in the sample is based on the literature from Campos et al., which states that the hardness value range for the martensite structure is >550 HV, lower bainite in the range of 350 – 490 HV, a mixture of martensite and lower bainite in the range of 450 – 550 HV, upper bainite in the range of 225 – 275 HV, and a mixture of bainite in the range of 275 – 350 HV⁵⁰).

In Figure 4 (a), the microstructure is dominated by tempered martensite and several granular bainite matrices, in which several granules are thought to be retained austenite and/or fresh martensite. This is consistent with a study from Campos⁵⁰. The existence of a significant amount of bainitic matrix can be due to the high Si content of the steel, which lowers the bainite start temperature and decreases the free energy of the ferrite, thereby increasing the driving force for bainitic reactions^{51,52}). Furthermore, Cr and Mn are known to support the formation of bainite in steel during continuous cooling. The microstructural results obtained in this study are similar to those of the research conducted by Sebastien TP et al.⁵³) regarding carbide investigation on 0.3 wt% C as-QTT steel. The results show the presence of a retained austenite phase, SEM test results on samples subjected to the sub-zero process are shown in Figure 4(b). As seen in Figure 4(b), the SEM microstructure, with a magnification of 50000x, reveals the formation of a martensite. On the inside, as shown in Figure 4(b) at 25000x magnification, there are several irregularly shaped granules, which are retained austenite and the presence of carbides. The SEM test results on the as-QSTT sample are shown in Figure 4 (c). In this Figure, a martensite containing retained austenite (irregular shape) and carbide on the inside, as shown in Figure 4(c). Based on morphological comparison, the martensite observed corresponds to lath martensite typical of low-carbon steels, while bainite may be classified as

upper or lower depending on carbide distribution⁵⁴⁻⁵⁷). The presence of retained austenite in blocky or film-like morphology also influences mechanical behavior and transformation stability⁵⁸⁻⁶⁰).

3.3. Analysis of the Percentage of Retained Austenite and Macro Hardness

HSLA steels that have been cooled (as-quenched, as-subzero) and double tempered (as-QTT, as-QSTT) undergo quantitative phase calculations using ImageJ. In the core of the sample, there is a phase that is brightly colored and in the shape of an irregular shape, which is the retained austenite phase, the percentage of which is calculated based on % area after three measurements. The illustration of ImageJ measurements can be seen in Figure 5.

Figure 6 shows a graphical comparison of the retained austenite percentage from the ImageJ measurements. The as-quenched sample exhibited the highest retained austenite fraction, with an average value of 6.8%. Sub-zero treatment reduced the retained austenite fraction to an average value of 3.9%. Without temper process, the retained austenite amount is relatively high in the sample. On the other hand, the sample with a double temper exhibited a lower retained austenite. The average amount of retained austenite is at 3.37% average and 1.2% average for the sample with and without subzero treatment respectively. It should be noted that the retained austenite values obtained in this study are based on image analysis and therefore represent semi-quantitative estimations. Future investigations using XRD analysis are recommended to provide more precise phase quantification. The sample resulting from the as-QTT treatment has a greater amount of retained austenite when compared to the results of the as-QSTT treatment because the low sub-zero temperature will increase the value of the cooling rate, which will ultimately suppress the diffusion process because the low cooling temperature forces the material below the martensite start temperature and

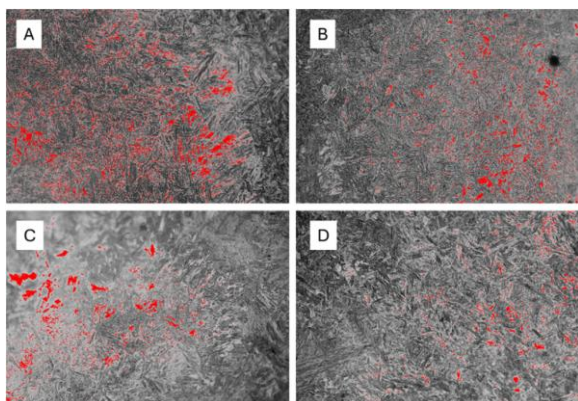


Fig. 5: ImageJ measurement on retained austenite phase (marked in red color). (a) as-quenched (b) as-subzero, (c) as-QTT, and (d) as-QSTT

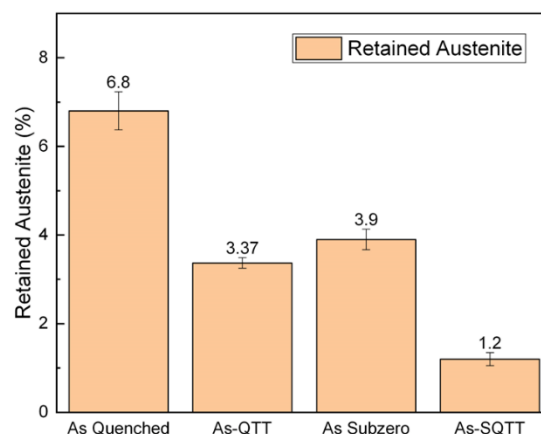


Fig. 6: Comparison of retained austenite fractions between conventional and sub-zero treatments

touches the martensite finish line before transforming into bainite⁶¹).

The transformation kinetics of retained austenite are strongly dependent on temperature and carbon content. Sub-zero treatment accelerates the transformation of metastable austenite into martensite by increasing undercooling below M_{f3} ^{7,61-63}). During tempering, retained austenite can become destabilized and decomposed, with its stability depending on the tempering conditions and surrounding microstructure⁶⁴⁻⁶⁶). Rapid or high-temperature tempering has also been reported to alter retained austenite stability and the microstructural evolution of bainitic steels^{67,68}). The thermal and mechanical stability of retained austenite during cryogenic and tempering treatments have been investigated in relation to its surrounding microstructure and phase condition^{69,70}). Carbon enrichment and partitioning during quenching and tempering can further modify retained austenite stability and microstructural evolution⁷¹⁻⁷³). The observed reduction in retained austenite is consistent with thermodynamic descriptions of martensitic transformation^{74,75}). Kinetic framework models have also been used to describe austenite decomposition and evolution in low-alloy and high-strength steels⁷⁵⁻⁷⁸).

The decrease in the amount of retained austenite occurred after double tempering at 205 °C for 5 hours. During the first tempering process, precipitation of ϵ -iron carbide or other carbide phases occurs, accompanied by a reduction in the excess carbon content present in martensite. The Carbon is then slowly partitioned into the retained austenite. During the second tempering process, the retained austenite that has not been transformed into the material decomposes into bainitic ferrite or ferrite carbide. Talebi et al. specifically found that the retained austenite will decompose to lower bainite during the tempering process⁷⁹).

Macro hardness testing was conducted on the HRC scale. Figure 7 shows a graph of macro hardness between conventional treatment and sub-zero treatment. The as-

quenched sample has a hardness of 51 HRC. After tempering at a temperature of 205°C, the hardness is decreasing to 48 HRC. The same trend also occurred for the sample with subzero treatment. The hardness for the subzero sample with and without double tempering process is 54 HRC and 49 HRC respectively. The decrease in hardness value after the double tempering process is caused because the tempering process produces carbide precipitates, which bind the solid solution elements to the matrix, thereby decreasing the material's hardness⁸⁰.

There are differences in hardness values between the as-QTT sample and the as-QSTT sample; the as-QSTT sample has a slightly higher hardness than the as-QTT hardness^{81,82}. The as-QTT hardness value is 48 HRC, while the as-QSTT hardness value is 49 HRC. Although the increase in hardness after sub-zero treatment is relatively small, the primary objective of this study is the reduction of retained austenite rather than maximizing hardness improvement. The transformation behavior of retained austenite during sub-zero treatment and subsequent tempering is complex, as the retained austenite may transform not only into martensite but also contribute to the formation of bainitic ferrite and carbide precipitates during tempering. Consequently, the increase of overall hardness may appear limited. Nevertheless, the slight increase in hardness observed in the as-QSTT sample remains consistent with the reduction of retained austenite, which is generally softer and less stable compared to martensitic phases. These results are consistent with research conducted by Das D. et al.²³. The research explained the effect of sub-zero treatment on the microstructure and hardness of AISI D2 steel. The increase of hardness in AISI D2 steel after sub-zero treatment has been associated with changes in the martensitic microstructure and carbide precipitation⁸¹. Related studies have also reported changes in microstructure and mechanical properties following sub-zero treatment in maraging stainless steel, and Cr-V ledeburitic steels^{83,84}.

Reviews and experimental studies further discuss the microstructure-toughness relationship and strengthening behavior of sub-zero treated steels⁸⁵⁻⁸⁷. Precipitation strengthening occurs due to the formation of transition carbides such as ϵ -carbide during tempering, which impede dislocation motion⁸⁸⁻⁹¹. The relationship between martensite fraction and hardness is well established, with higher martensite content leading to increased hardness values^{61,92-95}.

The reduction in retained austenite has significant implications for engineering applications, particularly in automotive and structural components subjected to cyclic loading⁹⁶⁻⁹⁹. Lower retained austenite content improves dimensional stability and reduces the risk of delayed cracking caused by stress-induced martensitic transformation^{97,100,101}. In service conditions, reducing retained austenite provides several advantages, particularly in lowering the risk of cracking. Metastable retained austenite can transform into martensite under applied stress, strain, or hydrogen exposure, leading to local volume expansion and stress concentrations that may cause delayed cracking and dimensional instability¹⁰². By minimizing unstable retained austenite, these transformation-induced strains are reduced, thereby decreasing the likelihood of cracking. Moreover, sub-/deep-cryogenic treatments are widely reported to decrease retained austenite and improve dimensional stability, both of which contribute to a lower susceptibility to service-related cracking¹⁰³. In short, the overall mechanism of retained austenite reduction involves three main stages: (1) incomplete martensitic transformation during quenching, (2) further transformation of retained austenite into martensite during sub-zero treatment, and (3) carbide precipitation and phase stabilization during tempering^{3,43,54,68,74,88,90,104}. Our findings of reduced retained austenite after sub-zero treatment are consistent with these observations.

4. Conclusion

Based on the test results, sub-zero treatment significantly influences the retained austenite content and hardness of HSLA steel. Cooling the material to -176 °C effectively reduces the retained austenite, lowering it from 3.37% in the as-QTT sample to 1.2% in the as-QSTT sample which means a reduction of 2.17% compared to conventional treatment. This reduction is due to the increased cooling rate at extremely low temperatures, which suppresses diffusion and drives the transformation process fully past the martensite start and toward the martensite finish line before any bainite forms. As a result, the hardness improves, reaching 49 HRC which is attributed to the formation of a more uniform carbide structure. Furthermore, the reduced retained austenite lowers the isothermal transformation rate and the resulting strain

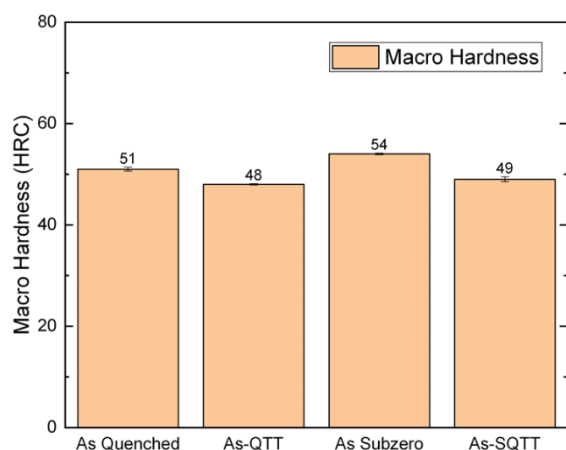


Fig. 7: Comparison of macrohardness values between conventional and sub-zero treatments

volume, helping to minimize the risk of delayed cracking after heat treatment.

Importantly, these findings highlight the practical implications of applying sub-zero treatment to HSLA steels. By reducing retained austenite and enhancing hardness the treatment may improve dimensional stability and reduce susceptibility to microcrack initiation, both of which are critical for components exposed to cyclic loading or thermal fluctuations. This suggests that sub-zero treatment has the potential to extend fatigue life and enhance long-term reliability of HSLA steels in demanding structural and engineering applications.

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