Jurnal Polimesin
Department of Mechanical Engineering
State Polytechnic of Lhokseumawe
http://e-jurnal.pnl.ac.id/polimesin

e-ISSN	: 2549-1999	No.	: 3	Month	: June
p-ISSN	: 1693-5462	Volume	: 24	Year	: 2026

Processing dates: received on 2025-12-11, reviewed on 2026-2-11, accepted on 2026-05-29 and online availability on 2026-06-30

Effect of hardening holding time and tempering duration on the microstructure and hardness of CHQ 10B21 wire

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Abstract

Heat treatment parameters play an important role in controlling the mechanical properties and microstructure of bolt materials. The research was conducted on the production of bolts produced from raw material wire CHQ 10B21. This study aims to evaluate the effect of hardening holding time and tempering duration on the hardness, tensile strength, and microstructure of CHQ 10B21 wire used for bolt production. The material was heated at 870°C with varying holding times, followed by oil quenching and tempering at 480°C with different tempering durations. Hardness testing was evaluated based on the HES D3211-99A standard with a target range of 22–32 HRC, while metallographic observations were conducted using sequential grinding and polishing. The results showed a direct relationship between holding time, tempering duration, and material properties. Longer hardening holding time increased hardness, with the highest value of 29 HRC obtained at 60 minutes, compared with 14 HRC in the raw material. Increasing tempering time improved tensile strength, reaching 891.91 N/mm² at 75 minutes after 60 minutes of hardening. Microstructural analysis showed the formation of tempered martensite and tempered bainite, with more homogeneous phase distribution at longer heat-treatment durations. These findings indicate that the optimal heat treatment condition for CHQ 10B21 bolt production is 60 minutes hardening followed by 75 minutes tempering.

Keywords:
CHQ 10B21, tempering, martensite, hardening, bolting.

1 Introduction

In general, the materials used according to standards for automotive bolt production are medium carbon steel (SAE 1030, SAE 1040), alloy steel (35CrMo4, 42CrMo4), stainless steel (AISI 304, AISI 316) and boron steel (10B21, 15B25) [1]. Low carbon steel material has limitations in the consistency of cold forging and heat treating characteristics of wire rod so that it is necessary to add boron in optimizing the composition of the material or process so that it is ductile in the forming process and the strength of the resulting material is better [2]. The low boron content mixture in SAE 10B21 material serves to increase hardenability through heat treatment process. The important stages of heat treatment in 10B21 steel are hardening and tempering [3]. Research on the effect of tempering holding time on piston components with SCM 420 steel material was conducted by Budiarto & Turnip with a stop tempering time of 20 seconds and a run of 5 seconds to obtain the best hardness value. The tempering process functions to soften and eliminate residual stress that still exists in the piston rod during quenching [4]. Research on the influence of various heat treatment processes on the microstructure of AISI 01 steel has been studied by Melya Sebayang and team which is used for cutting tools with a focus on changes in the microstructure of steel with quenching and tempering processes and their impact on mechanical properties followed by tempering will improve the steel structure and make it more resilient in balancing hardness and toughness which is important for cutting tool applications [5]. Research on the properties and performance of AISI 10B21 steel as a fastener has been conducted by Mehta and team [6] namely comparing the effects of various heat treatments, such as tempering, quenching, and carbonitridine on the mechanical properties of steel, namely hardness and flexibility. Testing of the heating process on 10B21 spheroidized coil material with several parameters such as preheating time, spheroidized annealing temperature, heating time, and furnace cooling temperature, and cooling time was carried out by Yang [7] to improve the mechanical properties of the material. The use of the Taguchi Method identifies optimal conditions that approach the target values for these properties at spheroidization heating parameters, thereby improving material performance in industries that require high tensile strength and good formability of 10B21 wire [7]. Research to determine the impact of tempered microstructure and hydrogen concentration on the susceptibility to hydrogen-induced brittleness of 10B21 screws at low temperatures has been carried out, showing that the microstructure after tempering significantly affects the strength of the material to hydrogen brittleness, with variations in hydrogen concentration also playing an important role [8]. The research resulted in heat treatment being able to significantly change the microstructure of 10B21 steel and affect mechanical properties such as tensile strength, ductility, and hardness with optimization of heat treatment conditions being able to improve these properties [9]. The effect of strain aging at various temperatures on the mechanical properties of 10B21 steel by observing the material structure shows that it significantly affects the mechanical properties such as tensile strength, ductility, and hardness of 10B21 steel [10]. The goals to analyze the effect of variations in heat treatment holding time on the mechanical properties of hardness, tensile strength value and microstructure of CHQ 10B21 wire and analyze the optimal conditions that combine heating holding time and tempering to achieve the best mechanical properties. The research limitation that is the focus of the researcher is that the hardening process temperature is a temperature above austenization, namely 870°C. The tests used were Rockwell C hardness test, the tempering resistance time variation was 60 minutes and 75 minutes, UTM tensile test and microstructure with optical microscope, the hardening resistance time variation was 30 minutes, 45 minutes and 60 minutes. The material specimen tested was CHQ 10B21 wire, the tempering process temperature was below the austenitization temperature, which was 480°C. The quenching media used was High Speed Quench Oil No. 1070. Early studies on 10B21 steel by Davenport [11] dan Simcoe [12] established the fundamental phase transformations in steel, which formed the basis for subsequent research on CHQ steels. This research forms the basis for further research on specific boron alloy steels such as 10B21. Research on the behavior of B27 steel material with boron alloys during heat treatment was conducted by Frydman [13] was found that the basic strength parameters of B27 steel such as R0.2 and Rm were relatively low in the original condition and after normalization but produced the highest hardening and hardening/tempering values at 200°C also increasing toughness and reducing brittle cracks in the operating temperature range of 20° to -40°C. The development of CHQ wire material bolt production with the addition of boron alloy to low carbon alloy material has undergone various tests to determine its performance carried out by Turner [14] so as to produce good bolt products. Research on slag content affecting the formation of Ds type inclusions has an impact on the quality of 10B21 steel used in cold heading (to make components such as nuts and bolts) conducted by Song and team [15]. Research using the Finite Element Method (FEM) to simulate the solidification process of 10B21 steel blanks measuring 280 mm × 325 mm under various technological conditions was carried out by Yang et al [16] resulting in an increase in casting speed set at 0.76 m/min causing the bloom solidification position to shift as far as 3.68 m backwards and the time required to achieve full solidification increased by 1 minute and when superheat increased the full solidification position also shifted by about 1.5 m. Research to determine the post-fire performance of bolts made of SAE 10B21 and 10B38 material after heat treatment and reheating at a target temperature of up to 900°C (post fire) was conducted by Yahyai [17]. Research on the effect of aging treatment at different temperatures on the strength variation of cold-hardened steel 10B21 with 20% area reduction by Dong [10] provide insight into the strengthening characteristics and mechanical properties of the steel. This study shows that the microstructure of medium carbon steel treated by the Q-P-T process mainly consists of nano-scale martensite and remaining austenite with the interface between martensite and austenite becoming irregular and unclear as the tempering time increases [18] casting of iron alloy material with high boron content and modification found that iron alloy with high boron content consists of dendritic matrix and interdendritic eutectic borides producing a matrix consisting of fine pearlite and borides having MzB crystal structure with high microhardness and distributed in the form of a continuous network. After heat treatment, the boride network breaks down and results in further ductility improvement in iron-based alloys with high boron content [19]. At below 500°C, the weight loss of HB-HSS decreased and the wear resistance of HB-HSS increased with increasing tempering temperature so that the wear loss of sample 1# (1% B and 1% Al) was greater than that of sample 2# and reached 10.5 mg and the wear resistance of HB-HSS decreased after red-hardness treatment [20]. Baking for a long duration can prevent the occurrence of Hydrogen Embrittlement (HE) susceptibility on 10B21 screws increasing with the increasing duration of baking [21]. The application of Direct Resistance Heat Treatment (DRHT) to create local variations in microstructure in high strength steel sheets (10B21) was carried out by Gould and team and resulted in a fully martensitic microstructure with a uniform hardness of about 50-Rc (500-VHN). This process allows control of the microstructure and mechanical properties through variable adjustment of heating and tempering time showing benefits for DRHT, reducing microstructural gradients and achieving a more homogeneous microstructure, the areas that have undergone DRHT transformation serve as effective crack deflectors in destructive testing, where tensile specimens with such areas successfully divert deformation and cracks to lower strength materials [22]. Materials with boron content at high temperatures will form an oxide layer in addition to experiencing HE [23]. The results showed that the formation of a mechanical mixed layer consisting of trapped abrasive particles and wear residue, supported by a hardened layer, can reduce the wear rate [24]. Huang and team studied the effect of ausforming on the kinetics of bainitic transformation in 2 wt.% Cr steel and found that the transformation rate and transformed volume fraction increased with increasing deformation at 500°C and 600°C but decreased after a large deformation of 45% at 700°C. It is concluded that the TCM II model can be used in vehicle impact simulations, and the presence of ferrite in the microstructure can provide benefits to crash performance, and can describe changes in the hardening behavior and strain rate sensitivity of multi-phase materials [25]. Research by Choi *et al.* [26] evaluated the effect of cooling factor analysis (QFA) in predicting hardness of partially quenched U-channel components made of boron steel and stated that the predicted hardness was in good agreement with the actual measurements, with a maximum error of 7.4%. This research will cover the experimental stages of the automotive component bolt manufacturing process. These stages will include raw material preparation, heat treatment under various conditions, laboratory testing, and analysis of the test results. The analysis will include a comparison of the bolt's mechanical properties before and after heat treatment, as well as an interpretation of the mechanisms responsible for these changes.

2 Research methodology

2.1 Materials

The material in the research is bolt production with CHQ 10B21 wire raw material in the form of rolls which will undergo a production process to produce automotive bolts.

2.2 Chemical composition

Based on the chemical composition tests carried out, the test results for the material were as shown in Table 1.

Table 1. Chemical composition of SAE 10B21 material

Chemical composition (%)							
C	Si	Mn	P	S	Cr	Mo	B
0.212	0.203	0.758	0.0069	0.0038	0.151	0.013	0.0017

2.3 Heat treatment procedure

Before conducting material research with the research flow diagram in Fig. 1, the research tools and materials were prepared.

2.4 Mechanical testing

The tools used during the research include a B-ONE mini ceramic muffle furnace MINI-1210 product heating oven with a maximum temperature of 1200°C in the UKI FT Lab for the hardening process of the test material, a Nakazawa TNC-355AL cutting machine with a maximum workpiece diameter of 355mm in material preparation for hardness tests and metallographic tests. Polishing machine for material preparation to be tested for metallography, TNM-32A mounting press machine is used in the material preparation process for metallography testing with the test material placed in a cold-setting polyester resin mounting material consisting of 98% resin and 2% catalyst/hardener. The hardness testing machine used is the Rockwell C hardness test with the Future-Tech Rockwell Hardness Tester FR-X Series machine with a load range of up to 150 kgf with HRC units, the Universal Testing Machine (UTM) Tensilon machine with a load of up to 20,000 kgf (200kN) for tensile strength testing; Olympus BX51M optical microscope with 500× magnification for the body part of the sample and 50× for the threads in the metallographic test to observe the microscopic structure of the material. The materials used during the study such as sandpaper grit 60, grit 80, grit 240, grit 320, grit 1200 and grit 200 for polishing the surface of the sample to be tested metallography, BUCHLER alumina powder 0.3 µm is used as polishing on the surface of ferrous materials and is good for checking the cleanliness of the sample, 3% Nital Etching solution. Dilute nitric acid (3% to 5% by volume in water), hydrochloric acid (4% to 6% by volume in water), and sodium hydroxide (2% to 6% by weight in water)) (Joshua Pierre, 2021).

2.5 Microstructural analysis

In this study, there are two variables, namely the independent variable, namely the hardening holding time and the tempering holding time (Table 2).

Table 2. Material heat treatment table

Heat treatment of materials					
Media: oli quench	$t_{HARDENING}$ 30'		$t_{HARDENING}$ 45'		$t_{HARDENING}$ 60'
	$T_{tempering}$: 60'	$T_{tempering}$: 75'	$T_{tempering}$: 60'	$T_{tempering}$: 75'	$T_{tempering}$: 75'
Wire CHQ 10B21	A1	A2	A3	A4	A5

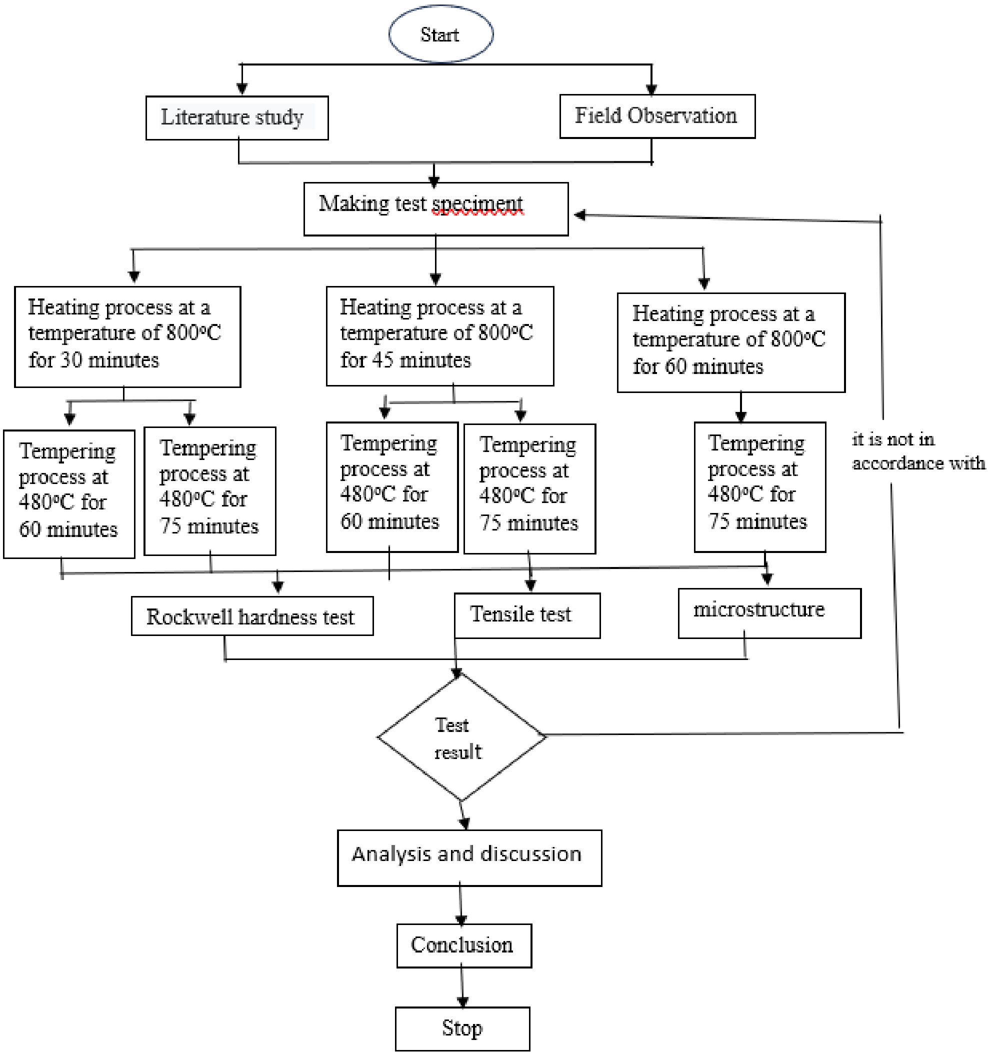

Fig. 1. Research flow diagram.

The dependent variable such as the hardness value of the bolt production according to the reference standard, namely HES D3211-99A where the hardness range is 22-32 HRC, the hardening temperature is 870°C and the tempering temperature is 480°C and shown in Table 2.

2.6 Experimental variables

The bolt manufacturing process goes through several long processes that will form bolts from raw materials into final products that meet standards as shown in Fig. 2.

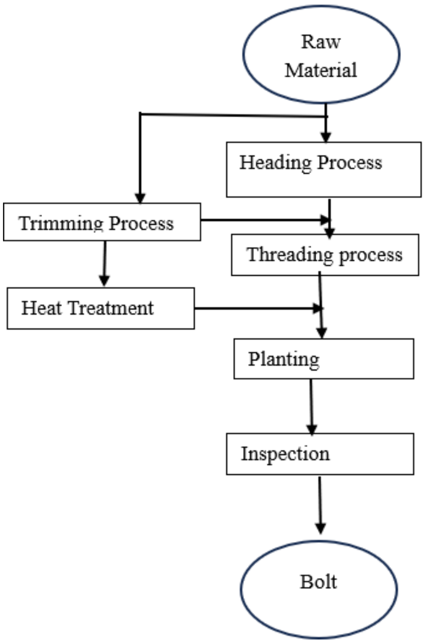

Fig. 2. Bolt manufacturing process.

3 Results and discussion

Continued collection of all data such as heat treatment process, the composition of raw materials required, parameters that need to be controlled in the heat treatment process, physical and mechanical properties of production that are in accordance with standards and ends with the analysis obtained can be applied to actual production conditions. With the completion of a series of tests from the production process to hardness testing, tensile strength testing and microstructure testing, the research data were obtained. The sample was heated for 30 minutes then quenched and then tempered with a holding time of 60 minutes, the results of the bolt body hardness test were obtained where the average value obtained was 16.6. HRC where the value is not included in the standard range of strength division 8.8 bolts, namely 22-32 HRC, the tensile strength value is only 598,038 N/mm^2 and the results of the sample thread microstructure test are as in Fig. 3 so that the sample is declared not good.

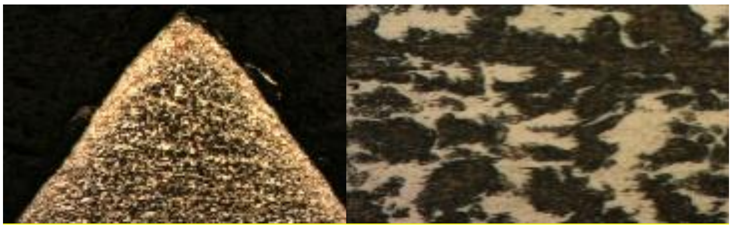
(a) (b)

Fig. 3. Microstructure of core area 500× magnification (a) sample A1, (b) thread area 50× magnification with 3% nital etching.

From the microstructure observation, it was found that after heating for 30 minutes and tempering holding time of 60 minutes after the quenching process, a tempered martensite structure was formed which was formed from the heating process above the austenitization temperature of 870°C and then quickly dipped (a). In image (b) in the thread section there is a difference in the surface and core sections. The thread section that is closer to the surface will experience faster cooling during quenching compared to the core.

Other samples were carried out under actual conditions in the field where the 10B21 bolt sample was heated for 60 minutes then quenched and then tempered with a holding time of 75 minutes showing the results of the hardness test of the bolt body where the average value obtained was 29.0 HRC, where the value is in accordance with the strength division 8.8 bolt standard, namely between 22-32 HRC so that production is declared OK and the tensile strength of the material with a tensile strength value of 891,915 N/mm^2 is still below the standard so it is declared OK.

In the microstructure shown in Fig. 4, there is a change in phase shape after heating-quenching-tempering heat treatment.

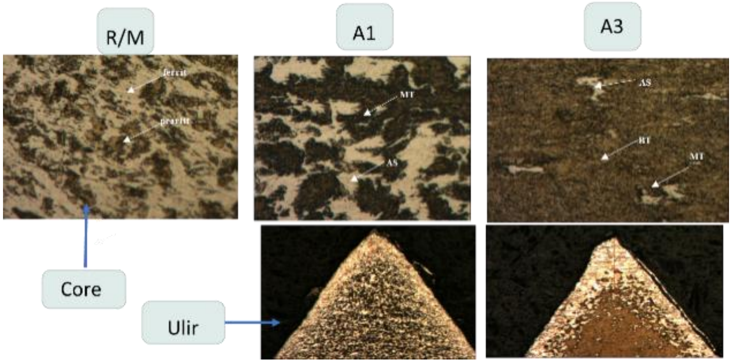

Fig. 4. Microstructure of samples at variations in hardening time.

The microstructure changes observed were in the core and thread sections. In the core of the test sample, changes in the structure of the raw material before heat treatment are visible, where the observed phases are dominated by pearlite and ferrite, as is the case with cold heading materials in general. The hardening was held for 30 minutes, then quenched and tempered. The material phase changed from austenite to martensite due to quenching and became tempered martensite after tempering, as shown in the dark colored part of the image. Then, due to the imperfection of the transformation conditions, residual austenite is formed, namely the part of the austenite that does not change into martensite, indicated by the light-colored area. At a holding time of 30 minutes, the phases formed are martensite and residual austenite. While at a holding time of 45 minutes, the material structure is more homogeneous. Remaining austenite is slightly visible so that the structure is more even in the tempered martenite phase and the presence of tempered bainite. Longer holding times provide a more even phase transformation time. The amount of remaining austenite can reduce the hardness value so that sample A1 with more remaining austenite than A3 has a lower hardness value. In the tests that have been carried out on samples with various hardening heat treatment resistance times and tempering heat treatment times, different mechanical properties of the material were obtained between samples as well as differences in the microstructure obtained as shown in Fig. 5.

Processing CHQ 10B21 material to achieve optimal mechanical properties requires hardening and tempering processes that require a combination of toughness, strength, and hardness. The test results show changes in the mechanical properties of 10B21 wire material. The hardness of 10B21 boron material during heat treatment compared to the hardness value of the raw material is shown in Fig. 6.

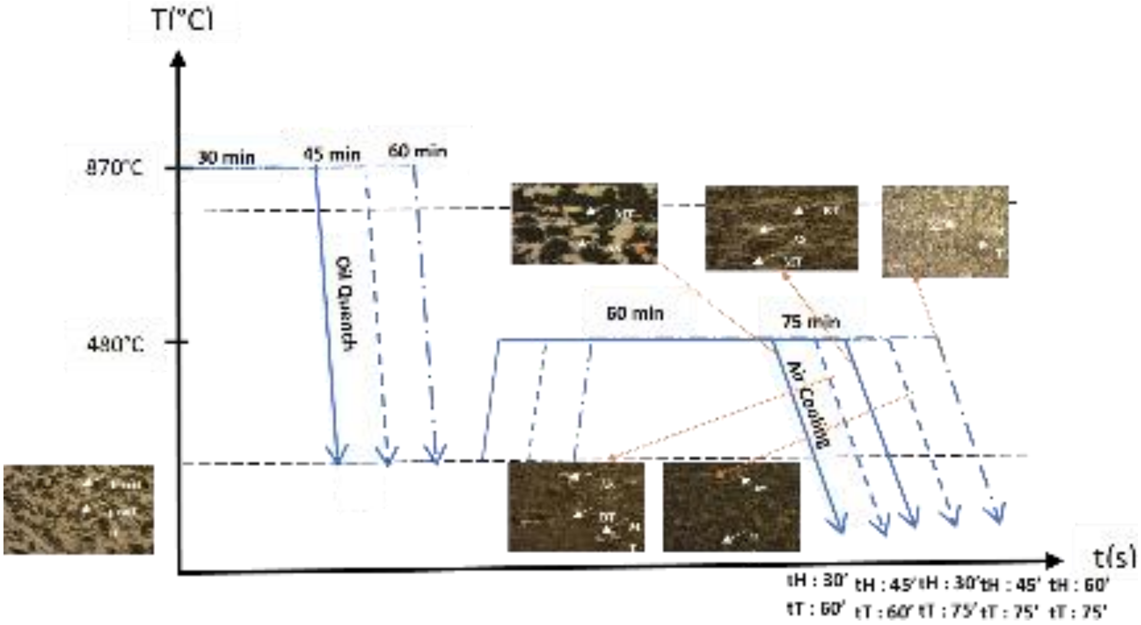

Fig. 5. Differences in microstructure in various heat treatments (hardening and tempering times).

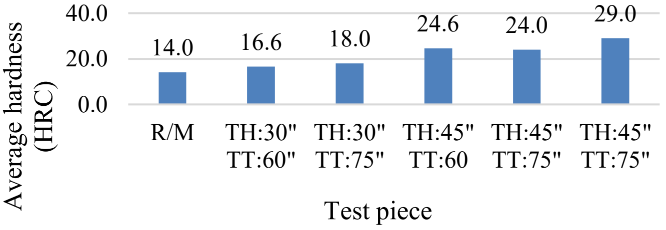

Fig. 6. Graph of material hardness under various heat treatments.

The hardening process with a longer holding time resulted in an increased hardness value with the highest hardness value produced at a holding time of 60 minutes, which was around 29.0 HRC from raw material with a hardness value of 14.0 HRC. The hardness value is high but the bolts must not be brittle so that the ductility properties of the research material must be improved. The increase in the ductility of the material is carried out by tempering at a temperature below the heating temperature so that a finer carbide dispersion is produced and the resulting material is harder. The tempering process is carried out to reduce hardness, increase ductility, and eliminate residual stress so that the graph shows the difference in tempering resistance time on the material resulting in different properties. Tempering treatment can increase the toughness of the material, as seen in Fig. 7, the results of material testing show that with the tempering process the tensile strength value of the material increases with the greatest tensile strength being in the material with a heating resistance of 60 minutes and tempering of 75 minutes, which is around 891,915 N/mm^2.

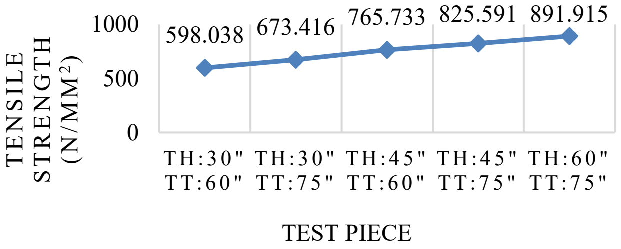

Fig. 7. Graph of tensile strength test results.

The increase in the tensile strength value of the material after the tempering process can be seen from the difference in tempering resistance time, namely at tempering times of 60 minutes and 75 minutes there was an increase in the tensile strength value which means an increase in bolt toughness.

A slightly longer tempering holding time of 75 minutes compared to 60 minutes resulted in a more extensive tempering resulting in a slight increase in ductility, reduction in internal stresses and a slight decrease in hardness compared to the shorter tempering duration. Longer tempering times allow more carbide precipitation and stabilization of the martensite structure, increasing ductility and reducing brittleness. The tempering process produces a material that has a balance between toughness and hardness. In the graph in Fig. 8, the hardening treatment for 45 minutes with a tempering time of 60 minutes and 75 minutes has a different hardness value, although not significant, from 24.6 HRC at a tempering time of 60 minutes to 24.0 HRC at a tempering time of 75 minutes, but the tensile strength has the opposite value where at 75 minutes it is greater than the tensile strength at a tempering time of 60 minutes. A different thing happened to the sample with a heating time of 30 minutes, namely the hardness value increased from 16.6 HRC at a tempering time of 60 minutes to 18.0 HRC at a tempering time of 75 minutes and the tensile strength value increased from 598,038 N/mm^2 at a tempering time of 60 minutes to 673,416 N/mm^2 at a tempering time of 75 minutes. This difference is influenced because during tempering the steel experiences microstructural changes, namely martensite changes into tempered martensite and carbides precipitate. Longer tempering times allow the formation of more stable carbides, increasing hardness and tensile strength. The presence of alloying elements such as boron, carbon, and manganese in 10B21 steel supports transformations that contribute to the resulting mechanical properties.

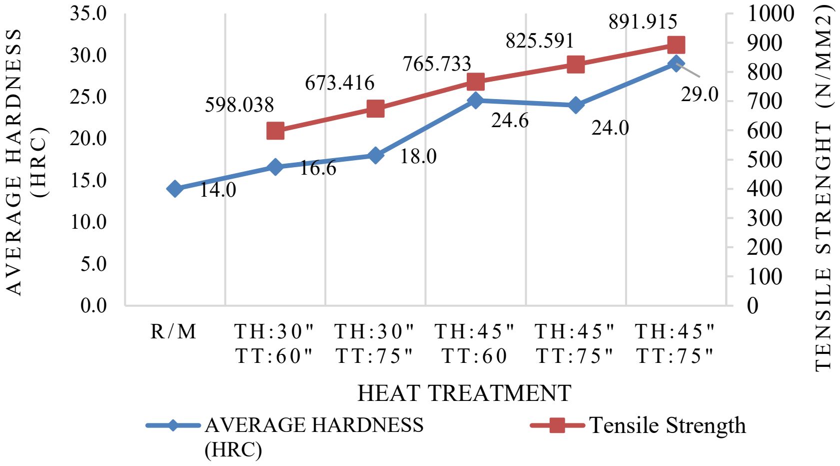

Fig. 8. Combined graph of hardness and tensile strength in various heat treatments.

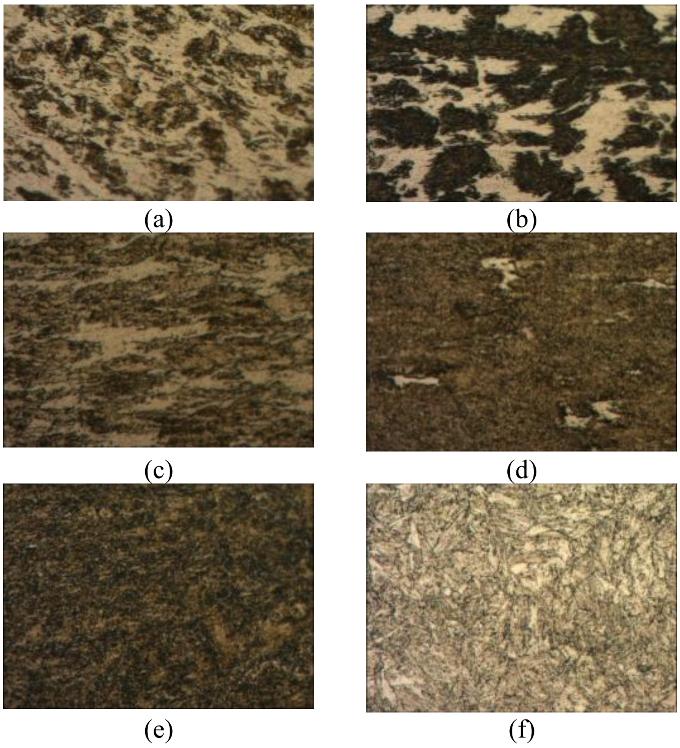
(a) (b) (c) (d) (e) (f)

Fig. 9. The microstructure of CHQ 10B21 material at various heat treatments (a) Raw Material (b) tH: 30'; tT: 60' (c) tH: 30'; tT: 75' (d) tH: 45'; tT : 60'(e) tH: 45'; tT: 75'(f) tH: 60'; tT: 75'.

Observation of changes in the structure of CHQ 10B21 wire material was carried out using an optical microscope with a magnification of 500×. In Fig. 9, changes in material structure during heat treatment changes such as heating to 870°C, the wire is heated to a temperature above the critical temperature (A3) for low carbon steel, causing the transformation of all microstructures (ferrite and pearlite) into austenite (Face-Centered Cubic (FCC) structure) which is able to dissolve more carbon compared to ferrite. This also applies to samples with a heating time of 60 minutes where the final microstructure is much better than the previous sample. This also applies to samples with a heating time of 60 minutes where the final microstructure is much better than the previous sample. After the austenitization process the material is rapidly cooled in oil (quenching) to prevent significant carbon diffusion, causing martensite transformation. Martensite is a metastable phase with a Body-Centered Tetragonal (BCT) crystal structure formed from austenite and has high hardness but tends to be brittle.

The material is then tempered at a temperature of 480^{0}C so that it aims to reduce the hardness of the martensite and increase its toughness. In this tempering process, residual stress will be relaxed where the internal stress in the martensite is reduced and the material will change into a strong and tough phase. This is because some of the tempered martensite changes into tempered martensite which is a mixture of ferrite and fine carbides. In addition, carbide formation usually occurs throughout the martensite and contributes to the hardness and strength of the material. Where excess carbon in the martensite diffuses out and forms fine carbide particles. The microstructure of CHQ 10B21 raw material before heat treatment is dominated by the ferrite phase, which is a light-colored area and pearlite, which is a dark-colored area and is lamellar in shape. This is influenced by the CHQ 10B21 wire manufacturing process where this material has been designed to have good mechanical properties for the cold heading process.

In heat treatment with a hardening holding time (t_H) of 30 minutes with a tempering time (t_T) which has a different morphological structure. At a tempering time of 60 minutes, temper martensite is formed from the quenching process, namely the dark colored area with the lighter area being carbide precipitation. A slightly longer tempering time compared to 60 minutes (i.e. 75 minutes) results in a more extensive tempering resulting in a slight increase in ductility, reduction in internal stresses, and a slight decrease in hardness compared to the shorter tempering duration and allows more carbide precipitation and stabilization of the martensite structure, increasing ductility and reducing brittleness. For samples with a hardening time (t_H) of 45 minutes with a tempering time (t_T) of 60 minutes and 75 minutes, the tempered martensite structure is more homogeneous because the material has more time than before and produces a more homogeneous phase so that the hardness and strength of the resulting material are better. The best material properties are obtained from materials with a hardening time (t_H) of 60 minutes with a tempering time (t_T) of 75 minutes where the microstructure formed is more homogeneous with a very optimal holding time to produce production with strong and ductile properties likes Fig. 10.

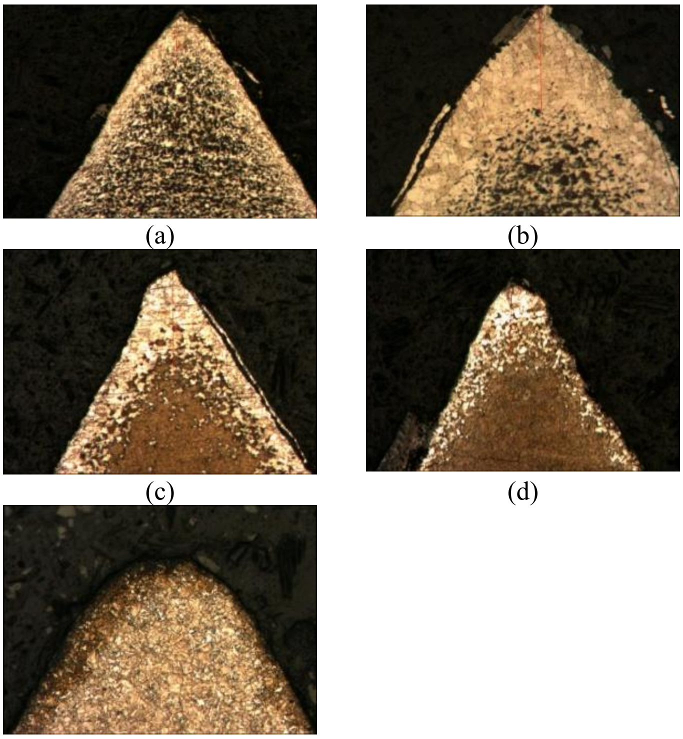
(a) (b) (c) (d) (e)

Fig. 10. Microstructure photos of CHQ 10B21 material in the threaded part of the bolt at various heat treatments (a) t_H: 30'; t_T: 60' (b) t_H: 30'; tT: 75' (c) t_H: 45'; tT: 60' (d) t_H: 45'; t_T: 75' (e) t_H: 60'; t_T: 75'.

The threaded part of the bolt is one of the critical points that must ensure the desired properties because the bolt produced must be strong but not brittle because the strength of the bolt provides the ability to withstand loads, while ductility provides the ability to resist deformation and failure under a wide range of operational conditions. The combination of these two properties results in a reliable and durable bolt thread, which is essential for machine performance and safety. Observations of the structure of the bolt section were also carried out where the thread section in all heat treatments had differences in the core and surface sections.

Fig. 10(a) to Fig. 10(e) shows the gradation from the core (thread center) to the surface. In conditions (a) and (b) with a heating holding time of 30 minutes and tempering time of 60 minutes, it is very clear that the core is darker in color compared to the surface because it has less martensite and more tempered ferrite, resulting in slightly lower hardness. Meanwhile, the tempered martensite part is more located on the surface with a lighter color and is more uniform. The thread regions closer to the surface generally experience faster cooling during the quenching process compared to the core. This results in higher hardness near the surface due to the finer martensite structure.

Fig. 10(e) shows a homogeneous structure with the core and surface having the same and even structure. The heat treatment carried out resulted in the statement that the CHQ 10B21 material has optimal mechanical properties for high-load applications such as bolt threads that require a combination of hardness and resistance to wear and impact loads. The optimal heat treatment to produce bolts with standards is at a hardening time of 60 minutes and a tempering time of 75 minutes.

4 Conclusions

Based on the test results and data processing that has been carried out, the conclusions can be drawn: (1) Hardening holding time affects material hardness. Increasing the holding time increased the hardness value, with the highest hardness of 29 HRC obtained at 60 minutes; (2) Tempering duration influences tensile strength. Longer tempering time increased tensile strength, with the maximum value of 891.91 N/mm² achieved at 75 minutes; (3) Microstructural observations showed the formation of tempered martensite and tempered bainite. The most homogeneous phase distribution was observed at 60 minutes hardening and 75 minutes tempering; (4) The optimal heat treatment condition for CHQ 10B21 bolt production was achieved at 60 minutes of hardening and 75 minutes of tempering, meeting the required hardness standard and providing improved mechanical performance.

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