JOURNAL OF MECHANICAL ENGINEERING, SCIENCE, AND INNOVATION
ITATS
Research Article

Effect of Cryogenic Treatment on Hardness Value and Microstructure of Medium Carbon Steel

Journal of Mechanical Engineering, Science, and Innovation
e-ISNN: 2776-3536
2024, Vol. 4, No. 1
DOI: 10.31284/j.jmesi.2024.v4i1.5972
ejurnal.itats.ac.id/jmesi

Dewi Izzatus Tsamroh[1], Johan Wayan Dika[2], Cepi Yazirin[3], Muhammad Ilman Nur Sasongko[1], Ardianto Prasetiyo[4]

[1]Manufacturing Engineering Technology, Universitas Negeri Malang, Indonesia
[2]Department of Mechanical Engineering, Universitas Nahdlatul Ulama Blitar, Indonesia
[3]Department of Mechanical Engineering, Universitas Islam Malang, Indonesia
[4]Department of Mechanical Engineering, Universitas Negeri Malang, Indonesia

Corresponding author:
Dewi Izzatus Tsamroh
Manufacturing Engineering Technology, Universitas Negeri Malang, Indonesia
Email: dewi.tsamroh.fv@um.ac.id

Abstract

Medium carbon steel is a kind of material that is usually used in manufacturing axles, gears, and shafts. Applying medium carbon steel to axles, gears, and shafts requires high hardness on the surface, but high ductility at the core. The mechanical properties of this material can be improved through a heat treatment process. However, in mass production there are obstacles, such is the uneven hardness on the entire surface, thus, it does not meet the hardness standards set as a commercial product. Therefore, cryogenic treatment was added which aimed to maintain the hardness value of the material after heat treatment. The material used in this research was S45C steel. The heat treatment conducted to increase the hardness of S45C steel was hardening followed by tempering. Hardening was carried out at a temperature of 900°C with a holding time of 45 minutes, followed by rapid cooling (quenching) using dromus oil as a medium. Tempering was carried out at 450°C for 15 minutes. Cryogenic treatment was carried out at -190°C for 1 hour. The research results showed that the highest hardness was obtained in specimens with hardening-quenching treatment followed by cryogenics with a hardness value of 35 HR_C (core) and 35.8 HR_C (surface). The hardness test results were in line with the microstructure test results., where the microstructure of specimens that had hardening-quenching treatment followed by cryogenics were dominated by pearlite, thus the hardness values were high.

Keywords: *Heat treatment, cold treatment, cryogenic, S45C carbon steel, hardness, microstructure*

Received: May 26, 2024; Received in revised: August 13, 2024; Accepted: August 15, 2024
Handling Editor: Vuri Ayu Setyowati

INTRODUCTION

The use of metal has increased over time, both aluminum alloys [1] and other metal alloys [2]. One metal alloy that is widely used in industry is carbon steel [3]. Carbon steel is divided into several types, one of which is medium carbon steel. S45C steel is a type of medium carbon steel that is widely used in the automotive industry, for example as a material in making shafts, gears, crankshafts, axles, and nuts and bolts. S45C steel consists of several elements, namely 0.4% Carbon (C), 0.03% Phosphate (P), 0.04% Sulfur (S), 0.2% Silicon (Si), 0.5% Manganese (Mn), 0.02% Chromium (Cr), Fe (balance)[4]. Because the carbon element content is quite high, this type of material is also called hard steel. This material can be given heat treatment such as quenching, tempering, normalizing, and annealing.

S45C steel, which is applied as axles, crankshafts, shafts, gears nuts, and bolts, is required to have good surface hardness and good ductility at the core [5]. So, usually, this material is given heat treatment which aims to increase its surface hardness. S45C steel is a type of carbon steel that can accept heat treatment [6]. Usually, heat treatment is carried out to improve the properties of S45C steel [7]. One type of heat treatment that can be applied to increase the hardness and ductility of steel is hardening and tempering. Hardening is done to get hard properties, while tempering is done to get ductile and tough properties [8]. However, in mass production, several obstacles were found, including uneven surface hardness, so that it did not meet the standards set as a commercial product. Hardness is a property of a material that indicates its ability to resist plastic deformation or penetration when subjected to force or pressure. Harder materials tend to be more resistant to scratching, abrasion, or denting than softer materials. Hardness is often measured using various methods, such as the Mohs, Vickers, Brinell, or Rockwell scales [9]. Ductile is a material property that indicates the ability to undergo significant plastic deformation before breaking. Ductile materials can be stretched or formed into wire or other shapes without breaking. This is an important property for materials that need to be shaped or stretched without failure, such as metals such as copper, aluminum, or steel that have high ductile properties [10]. Hardness and ductility are often inversely related; very hard materials tend to be less ductile, while very ductile materials tend to be less hard [11].

Therefore, cryogenic treatment was added which aims to maintain the hardness of the material [12]. Cryogenics is a cooling treatment of a material at a temperature of up to -195°C which is held for a certain duration of time [13]. This additional treatment is carried out after the heat treatment process on the material is complete. Previous research shows that cryogenic treatment can increase the strength of high-carbon steel [14]. Cryogenics not only increase material strength but can also reduce residual stress [15].

This research is supported by several previous studies which state that cryogenic treatment can effectively improve the mechanical properties of a material [16]. Other research also concluded that adding cryogenic treatment can significantly increase the strength and hardness of the Ni-W-Co-Ta alloy [11]. Compared with other cold treatments, material processed using cryogenics is proven to have a higher value according to its strength level [17]. Other research shows an increase in material toughness by combining cryogenic and tempering treatments [18]. Thus, this research aims to investigate the effect of cryogenic treatment on the hardness and microstructure of S45C steel.

METHODS AND ANALYSIS

The method used in this research is experimental. The material used in this research is medium carbon steel (S45C steel). S45C is a medium carbon steel grade that is often

Table 1. Chemical composition of S45C

	C	Mn	Si	P	S
Composition (%)	0,42% - 0,48%	0,60% - 0,90%	0,15% - 0,35%	Max. 0,030%	Max. 0,035%

used in industrial applications due to its good mechanical properties. The name "S45C" is the Japanese standard code (JIS: Japanese Industrial Standard) for this steel [5]. S45C is often used in the manufacture of shafts, gears, bolts, and other machine components that require high strength and wear resistance [19]. The following is the chemical composition of S45C that shown in Table 1.

Below is presented Figure 1 which shows an illustration of the research scheme. This research began with the formation of specimens, where cylindrical S45C steel was cut with a diameter of 12 mm and a length of 25 mm using a cutting grinding machine for 12 specimens.

Once the specimen is ready, it is prepared for the heat treatment process. Heat treatment of the specimen begins with hardening treatment, namely the specimen is heated to austenite temperature (900°C) and held for 45 minutes [20]. The purpose of heating to this temperature is to change the microstructure of S45C steel, and holding for this duration aims to homogenize the phases formed at that temperature. Cooling of the specimen was carried out quickly using dromus oil media [21]. Dromus oil is a premium-class lubricant that provides optimal levels of lubrication and cooling for machines used in metalworking processes [22]. A total of 3 specimens after hardening treatment were followed by cryogenic treatment, while the other 3 specimens were given tempering treatment at a temperature of 450°C for 15 minutes before then being given cryogenic treatment at a temperature of -190°C for 1 hour. The tempering temperature is chosen at a medium temperature (300-550°C), this aims to increase the ductility properties but only slightly reduces the hardness of the material [6].

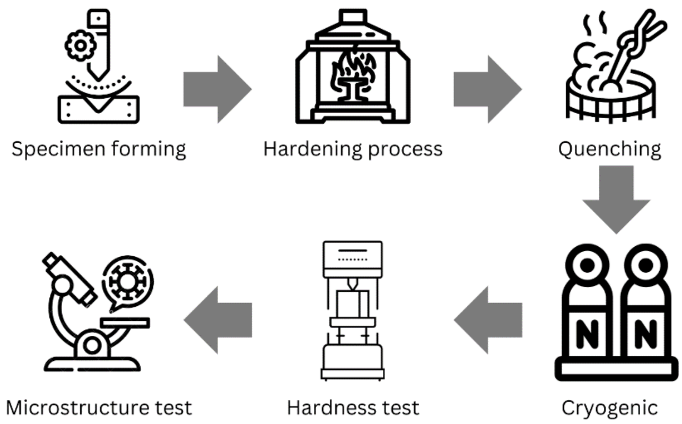

Figure 1. Illustration of the research scheme

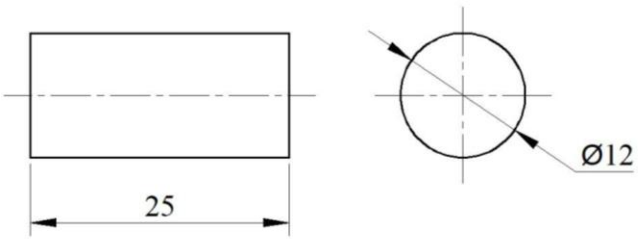

Figure 2. Dimension of specimen

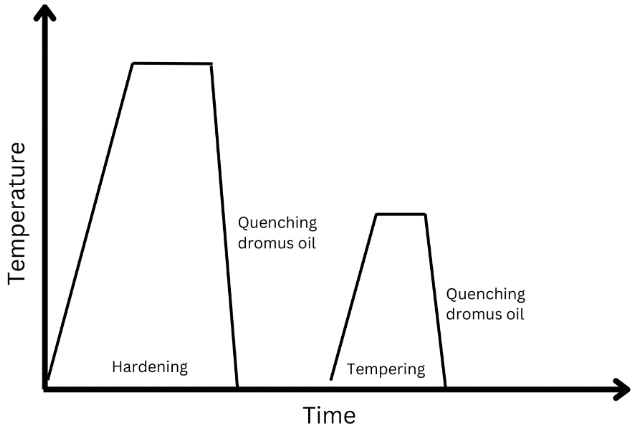

Figure 3. Heat treatment diagram

After the specimen has received heat treatment and cold treatment, the specimen is ready to be tested for hardness and microstructure. The specimen hardness test was carried out using the Rockwell method following the DIN 50103 standard as shown in Figure 2 with a major load specification of 150 kg and an indenter in the form of a diamond cone on the C scale. Meanwhile, the microstructure test was carried out at 200x magnification following the ASTM E7 standard by using an optical microscope.

RESULTS AND DISCUSSIONS

Hardness Value

Below is presented Table 2 which contains data on the hardness value of S45C steel with various treatments. The data is the average hardness taken from 3 points of each specimen. Based on Table 2, it can be seen that heat treatment can significantly increase the hardness of S45C steel [23]. This can be seen from the hardness value of the raw material which is only 7 HR_C on the core and 7.5 HRC on the surface, then increases to 30.7 HR_C on the core and 32.2 HR_C on the surface after undergoing the hardening process.

Specimens treated with hardening-quenching followed by cryogenic treatment produced the highest hardness values, namely 35 HR_C on the surface and 35.8 HR_C on the core. Meanwhile, in specimens that were subjected to tempering treatment, the increase in hardness was not higher compared to specimens that were not tempered. The following data is presented in graphical form, which shows a comparison of the hardness values of S45C steel on the core and surface.

Based on Table 2, each treatment produces a higher average hardness value on the surface of the specimen, compared to the core. Following the theory that hardness is inversely proportional to ductility, these results indicate that the core has higher ductility than the surface of the specimen. Other research shows that cryogenic treatment can increase the hardness of Titanium alloys [17], although the results of other research also show that cryogenic treatment is very effective in increasing the toughness of a material

Table 2. Hardness value of S45C steel

Data	Treatments	Hardness (HR_C)	
		Core	**Surface**
1	Raw Material	7	7.5
2	Hardening-Quenching	30.7	32.2
3	Hardening-Quenching-Cryogenic	35	35.8
4	Hardening-Quenching-Tempering-Cryogenic	27.2	28.3

[15], [18]. In this study, specimens that received hardening-quenching-tempering-cryogenic treatment produced lower hardness numbers compared to specimens that only received hardening-quenching and hardening-quenching-cryogenic treatment. These results are in line with the results of other research, where the strength and toughness of low-carbon steel increased through quenching-partitioning-cryogenic-tempering (Q-P-C-T) treatment [18]. The decrease in hardness due to the tempering process is caused by the martensite structure tending to change into tempered martensite and also tending to transform into bainite or form carbide deposits [24].

Microstructure of S45C Steel

Below are presented the results of microstructure testing on S45C steel with various treatments. Figure 4 shows the results of microstructure testing on S45C steel (a) raw material, (b) S45C steel with hardening-quenching treatments, (c) S45C steel with hardening-quenching-cryogenic treatments, (d) S45C steel with hardening-quenching-tempering-cryogenic treatments. Each treatment produces pearlite and ferrite phases [13]. The pearlite phase is characterized by a dark color which is harder than the ferrite phase, while the ferrite phase is characterized by a light color which is softer than the pearlite phase [24]. In Figure 4 (a), the ferrite phase dominates, so the hardness of S45C steel tends to be low. In Figure 4 (b) the pearlite phase has formed, marked by black spots, so that the hardness of S45C steel has increased. In Figure 4 (c), the amount of pearlite is greater than in Figure 4 (b), so the hardness number is higher, besides that the grain

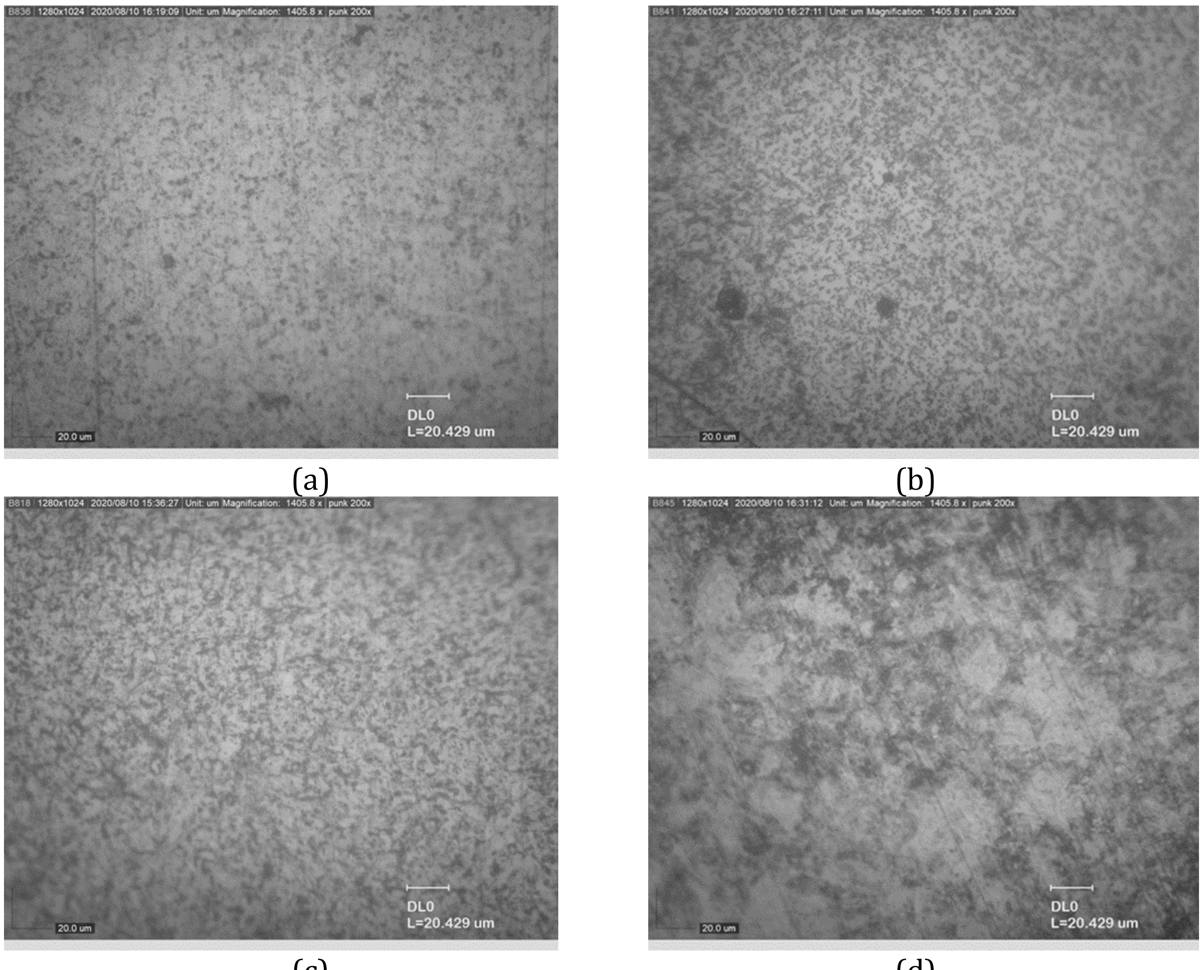

Figure 4. Microstructure of (a) raw material S45C steel, (b) S45C steel with hardening-quenching treatments, (c) S45C steel with hardening-quenching-cryogenic treatments, (d) S45C steel with hardening-quenching-tempering-cryogenic treatments.

size looks more homogeneous. Figure 4 (d) is dominated by the ferrite phase, which is shown in light color, besides that the grain size looks larger compared to Figure 4 (c), so it can be concluded that the hardness value is lower. Ferrite is formed due to the slow cooling process of the austenite of hypoeutectoid steel when it reaches A3. Ferrite is very soft, ductile, and has high conductivity. If austenite is cooled below A3, austenite which has a very low C content will transform to Ferrite [25], [26].

According to observations on the microstructure of S45C steel, it is known that the highest hardness was found in specimens with hardening-quenching-cryogenic treatment. In this treatment, the phase formed is dominated by hard pearlite. Meanwhile, the specimens that were given tempering treatment were dominated by the ferrite phase, which caused the hardness number to decrease. Visually, it is known that specimens with hardening-quenching-cryogenic treatment have a microstructure that tends to be more homogeneous, so this is also thought to cause the specimen to have high hardness [11], [24].

The discussion regarding microstructure is closely related to the resulting hardness value [15], [27]. Specimens that have more pearlite phases and a more uniform structure will have a higher hardness value compared to specimens that have more ferrite phases.

CONCLUSION

Based on the research results and discussion in the previous section, it can be concluded that giving cryogenic treatment to the S45C material can increase its hardness. The highest hardness figure was obtained in specimens that received hardening-quenching treatment followed by cryogenic treatment of 35 HR_C (core) and 35.8 HR_C (surface). Compared to raw material, this treatment can increase hardness by 400%. The results of microstructure testing also show that the specimens that received hardening-quenching followed by cryogenic treatment were dominated by pearlite, which causes the material to have high hardness.

ACKNOWLEDGEMENTS

DECLARATION OF CONFLICTING INTERESTS

The author(s) declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

FUNDING

The author(s) disclosed receipt of no financial support for the research, authorship, and/or publication of this article.

REFERENCES

[1] D. I. Tsamroh, “Comparison finite element analysis on duralium strength against multistage artificial aging process,” *Arch. Mater. Sci. Eng.*, vol. 109, no. 1, pp. 29–34, 2021, doi: 10.5604/01.3001.0015.0512.

[2] A. A. Rosidah, K. R. S. Roni, and M. Yunus, “Dried Clove Leaf as Corrosion Inhibitor for Medium Carbon Steel in Acidic Media,” *J. Mech. Eng. Sci. Innov.*, vol. 3, no. 2, pp. 78–85, 2023, doi: 10.31284/j.jmesi.2023.v3i2.5160.

[3] D. A. Utama, “Pengaruh Quenching Terhadap Laju Korosi pada Baja Karbon Tinggi,” Univeristas Sriwijaya, 2022.

[4] S. Zhang, K. Gao, S.T. Hong, et al., "Electrically assisted solid state lap joining of dissimilar steel S45C and aluminum 6061-T6 alloy," J. Mater. Res. Technol., vol. 12, pp. 271–282, 2021, doi: 10.1016/j.jmrt.2021.02.091.

[5] D. R. Eo, J. Yoon, and H. Lee, "Heterostructure effect at the interface of maraging steel deposited upon carbon steel via directed energy deposition," *Mater. Des.*, vol. 225, p. 111574, 2023, doi: 10.1016/j.matdes.2022.111574.

[6] M. Bajt Leban, B. Zajec, B. Podgornik, *et al.*, "Susceptibility of X17CrNi16-2 martensitic stainless steel to hydrogen embrittlement after conventional and deep cryogenic heat treatment," *Eng. Fail. Anal.*, vol. 162, no. May, 2024, doi: 10.1016/j.engfailanal.2024.108403.

[7] Z. H. Song, H. Y. Song, J. X. Liu, et al., "Tuning heterogeneous microstructure and improving mechanical properties of twin-roll strip cast low carbon steels by on-line heat treatments," *J. Mater. Res. Technol.*, vol. 30, no. 11, pp. 5894–5904, 2024, doi: 10.1016/j.jmrt.2024.04.143.

[8] Y. M. Wang, Y. Liang, Y. D. Zhai, *et al.*, "Study on the role of cryogenic treatment on corrosion and wear behaviors of high manganese austenitic steel," *J. Mater. Res. Technol.*, vol. 24, pp. 5271–5285, 2023, doi: 10.1016/j.jmrt.2023.04.100.

[9] L. M. Rymer, L. Winter, K. Hockauf, et al., "Artificial aging time influencing the crack propagation behavior of the aluminum alloy 6060 processed by equal channel angular pressing," *Mater. Sci. Eng. A*, vol. 811, p. 141039, Apr. 2021, doi: 10.1016/J.MSEA.2021.141039.

[10] B. T. Ogunsemi, T. E. Abioye, T. I. Ogedengbe, et al., "A review of various improvement strategies for joint quality of AA 6061-T6 friction stir weldments," *J. Mater. Res. Technol.*, vol. 11, pp. 1061–1089, 2021, doi: 10.1016/j.jmrt.2021.01.070.

[11] K. hao SHU, Y. XIONG, Y. LI, *et al.*, "Cryogenic rolling impacts on microstructures and properties of a novel Ni–W–Co–Ta medium-heavy alloy," *Trans. Nonferrous Met. Soc. China (English Ed.*, vol. 34, no. 4, pp. 1214–1225, 2024, doi: 10.1016/S1003-6326(24)66465-4.

[12] X. Li, Z. Wei, X. Wang, *et al.*, "Effect of cryogenic temperatures on the mechanical behavior and deformation mechanism of AISI 316H stainless steel," *J. Mater. Res. Technol.*, vol. 22, pp. 3375–3386, 2023, doi: 10.1016/j.jmrt.2022.12.190.

[13] M. Villa and M. A. J. Somers, "Cryogenic treatment of an AISI D2 steel: The role of isothermal martensite formation and 'martensite conditioning,'" *Cryogenics (Guildf).*, vol. 110, no. May, p. 103131, 2020, doi: 10.1016/j.cryogenics.2020.103131.

[14] J. kuan Ren, D. shun Mao, Y. Gao, et al., "High carbon alloyed design of a hot-rolled high-Mn austenitic steel with excellent mechanical properties for cryogenic application," *Mater. Sci. Eng. A*, vol. 827, no. March, p. 141959, 2021, doi: 10.1016/j.msea.2021.141959.

[15] M. A. Essam, A. Y. Shash, M. K. El-Fawakhry, et al., "Influence of micro-alloying elements and deep cryogenic treatment on microstructure and mechanical properties of S5 cold work shock resisting tool steel," *Results Mater.*, vol. 17, no. January, p. 100374, 2023, doi: 10.1016/j.rinma.2023.100374.

[16] X. Yang, R. Li, Y. Wang, et al., "Improvement of mechanical and tribological performances of carbon nanostructure films by cryogenic treatment," *Tribol. Int.*, vol. 156, no. September 2020, p. 106819, 2021, doi: 10.1016/j.triboint.2020.106819.

[17] K. Mertová, P. Salvetr, and M. Duchek, "Comparison of cryogenic and cold deformation of commercially pure titanium," *Mater. Today Proc.*, vol. 28, no. xxxx, pp. 916–919, 2019, doi: 10.1016/j.matpr.2019.12.324.

[18] K. Wang, K. Gu, J. Miao, *et al.*, "Toughening optimization on a low carbon steel by a novel Quenching-Partitioning-Cryogenic-Tempering treatment," *Mater. Sci. Eng. A*, vol. 743, pp. 259–264, 2019, doi: 10.1016/j.msea.2018.04.104.

[19] R. I. Riza and K. A. Ilman, "Data on the cooling rate using nano carbon-fluid quenching medium and its effect on the hardness of S45C steel," *Data Br.*, vol. 46, p. 108867, 2023, doi: 10.1016/j.dib.2022.108867.

[20] C. Cui, Z. Weng, K. Gu, et al., "The strengthening role of post-welded cryogenic treatment on the performance and microstructure of 304 austenitic stainless steel weldments," *J. Mater. Res. Technol.*, vol. 29, no. January, pp. 5576–5584, 2024, doi: 10.1016/j.jmrt.2024.02.220.

[21] D. I. Tsamroh, P. Puspitasari, A. Andoko, et al., "Optimization of multistage artificial aging parameters on Al-Cu alloy mechanical properties," *J. Achiev. Mater. Manuf. Eng.*, vol. 87, no. 2, pp. 62–67, 2018, doi: 10.5604/01.3001.0012.2828.

[22] R. N. Fattah, S. Sugiyanto, B. H. Priyambodo, et al., "Rekayasa Peningkatan Kekerasan Permukaan Gear Sprocket Sepeda Motor dengan Metode Quenching Variasi Media Pendingin," *Quantum Tek. J. Tek. Mesin Terap.*, vol. 5, no. 1, pp. 8–13, 2023, doi: 10.18196/jqt.v5i1.19418.

[23] C. T. Parcianello, D. G. Fantineli, T. S. Rosendo, et al., "Influence of the heat treatment on the mechanical and tribological properties of cryogenically treated AISI M2 steel," *J. Mater. Res. Technol.*, vol. 26, pp. 6462–6475, 2023, doi: 10.1016/j.jmrt.2023.08.309.

[24] P. Jovičević-Klug, M. Jovičević-Klug, L. Thormählen, *et al.*, "Austenite reversion suppression with deep cryogenic treatment: A novel pathway towards 3rd generation advanced high-strength steels," *Mater. Sci. Eng. A*, vol. 873, no. April, 2023, doi: 10.1016/j.msea.2023.145033.

[25] T. Surdia and S. Saito, "Pengetahuan Bahan Teknik," p. 372, 1999.

[26] N. Nurkim, S. Sukanto, D. I. Tsamroh, et al., *Pengantar Material Teknik*, 1st ed. Padang: CV. HEI PUBLISHING INDONESIA, 2024.

[27] J. Li, X. Zhang, H. Bu, *et al.*, "Effects of deep cryogenic treatment on the microstructure evolution, mechanical and thermal fatigue properties of H13 hot work die steel," *J. Mater. Res. Technol.*, vol. 27, no. November, pp. 8100–8118, 2023, doi: 10.1016/j.jmrt.2023.11.236.