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# The Effect of Temperature in Cryogenic Treatment on the Mechanical Properties and Microstructure of High-Speed Steel SKH 51

Indah Uswatun Hasanah[1*], A. Ali Alhamidi[2], Hardy Andriyansyah[1], Suprapto Suprapto[3]

[1] *Department of Metallurgical Engineering, Faculty of Engineering, Universitas Sultan Ageng Tirtayasa, Cilegon, Indonesia*
[2] *Department of Mechanical Engineering, Faculty of Engineering, Universitas Sultan Ageng Tirtayasa, Cilegon, Indonesia*
[3] *Research Center for Accelerator Technology, Research Organization of Nuclear Energy, National Research and Innovation Agency (BRIN), Yogyakarta, Indonesia*

*Corresponding author: *indah.uswatun.h@untirta.ac.id*

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## ABSTRACT

High-speed steel SKH 51 has excellent mechanical properties, including high hardness and wear resistance. This material is widely used for cutting tools and drills. Service life is also a crucial factor that is often improved to extend the material's usability. To enhance the service life of SKH 51, a heat treatment modification was applied, specifically cryogenic treatment, to increase its hardness and wear resistance. The process began with austenitization, followed by quenching using $N_2$ gas. Subsequently, cryogenic treatment was conducted at varying temperatures of -80°C, -110°C, and -140°C, with a holding time of 30 minutes. Finally, a tempering process was done at 540°C with a holding time of 3 hours. The microstructure and mechanical properties after the applied heat treatment were studied. In the as-received condition, the microstructure consisted of pearlite, ferrite, and coarse carbides. After the heat treatment, martensite and carbides formed in significant amounts, with only a small fraction of retained austenite remaining after the cooling process. X-ray diffraction (XRD) analysis revealed carbide phase peaks such as MC, $M_2C$, and $M_6C$. The hardness value increased as the cryogenic treatment temperature decreased over the 30-minute duration. Wear resistance was measured using the Ogoshi wear test method, showing an improvement as the cryogenic treatment temperature decreased. The increase in hardness and wear resistance of SKH 51 was attributed to the greater formation of martensite and carbides, which reduced the amount of retained austenite.

**Keywords:** *SKH 51, cryogenic treatment, martensite, carbide, retained austenite*

## 1. INTRODUCTION

The manufacturing industry, particularly in sectors such as automotive, aerospace, heavy machinery, and electronics, heavily relies on the durability of materials used in production. In manufacturing, the longevity or service life of materials is crucial for enhancing operational efficiency, reducing maintenance costs, and ensuring sustainable production. Moreover, industrial development also demands improvements in the mechanical properties of materials, such as wear hardness and resistance. Tool steels are widely used in manufacturing and machining industries, particularly for molds in forming processes and as cutting tools in machining operations. Among the various materials used in machining, high-speed steel (HSS) is one of the most commonly used. One of the most widely used types of tool steel is high-speed steel SKH 51 [1]. However, to optimize its mechanical characteristics, such as hardness, toughness, and wear resistance, an appropriate heat treatment process, including tempering, quenching, or annealing, is required. This heat treatment can alter the microstructure of the steel, thereby extending the service life of SKH 51 steel [2] [3].

The heat treatment process for tool steel, which begins with austenitization followed by quenching, introduces a new challenge: the formation of residual stress and retained austenite, which can degrade its mechanical properties. Therefore, an additional treatment is required to eliminate residual stress and retained austenite in the steel [4]. One of the effective methods for this purpose is cryogenic treatment [5] [6]. Cryogenic treatment has been recognized as an effective method for improving microstructure and dimensional stability, and it has been proven to enhance the mechanical properties of machine components, thereby extending the service life of tools [7] [8]. After undergoing cryogenic treatment, high-speed steel SKH 51 becomes extremely brittle, necessitating a tempering process to reduce its brittleness. A study on shallow cryogenic treatment conducted by Gill et al. at a temperature of -110°C with double tempering at 150°C showed that this process was less effective in improving the hardness and wear resistance of SKH 51 [9]. Subsequently, Lakhani and Raut conducted a study in 2017 on shallow cryogenic treatment at -80°C with a holding time of 5 to 8 hours [1]. In a later study, cryogenic treatment was performed at -70°C with a holding time of 30 to 60 minutes, successfully enhancing the mechanical properties of SKH 51 steel [10]. In this study, a heat treatment process in the form of cryogenic treatment is carried out to improve the mechanical properties of high-speed steel SKH 51 by varying the cryogenic treatment temperature.

## 2. METHODOLOGY

*2.1. Sample preparation*

The sample used in this study was tool steel SKH 51, which was pre-processed to achieve the required dimensions. The preparation process began with an initial cutting using a bandsaw machine to obtain dimensions of 4 cm × 4 cm × 4 cm. This was followed by further cutting using a hand grinder until the sample met the standard requirements for testing.

*2.2 Heat treatment*

The heat treatment process began by heating the sample in a vacuum furnace at a temperature of 1190°C for 30 minutes. However, before reaching this temperature, the sample was held at 650°C and 950°C for 60 minutes each to ensure homogenization. Following the heating process, quenching was performed to obtain the martensitic phase, which enhances the hardness of the steel. This was achieved using $N_2$ gas at a pressure of 1.9 bar. Once the sample reached room temperature, it was placed in a sub-zero treatment machine, where it was cooled using liquid $N_2$ at varying temperatures of -80°C, -110°C, and -140°C for 30 minutes to eliminate retained austenite formed during quenching. Finally, the sample was reheated in a vacuum furnace at 540°C and held for 3 hours to reduce brittleness and residual stress in the steel.

*2.3 Testing and characterization*

Before conducting the hardness test, the sample underwent a grinding process using sandpaper with grit sizes of 80# and 100# to achieve a flat and uniform surface, facilitating microstructural analysis and hardness testing. The sample was then placed in a microhardness tester, model MHVS-1000AT, equipped with a diamond pyramid indenter.

The wear test was done to evaluate the abrasive resistance of the HSS SKH 51 sample using the Oghoshi method, which follows the ASTM G99 standard. During this test, the sample was rubbed against a wear ring with a ring diameter of 30 mm, a ring thickness of 3 mm, a ring speed of 1.97 m/s, and an applied load of 3.16 kg.

Microstructural analysis was conducted using an Olympus BX41M-LED optical microscope to examine the phases and grain structures formed in the HSS SKH 51 sample through the metallographic method. The process began with grinding to smooth the steel surface using sandpaper with grit sizes of #80, #120, #240, #320, #500, #800, #1200, and #1500. Next, the sample was applied with diamond paste and underwent polishing using a polishing machine and polish wool. The final step before analysis involved etching the sample using Amberg's reagent, which consists of 20 ml HCl, 65 ml ethanol, 15 ml water, and 5 g $CuCl_2$. After etching, the sample was analyzed using an optical microscope at magnifications of 200X and 500X. Furthermore, X-ray diffraction (XRD) characterization was done to analyze the phases formed in the sample. The recorded XRD data produced a diffractogram, which represents the relationship between intensity and 2θ..

## 3. RESULTS AND DISCUSSION

*3.1. Raw material characterization*

The material used in this study is High-Speed Steel (HSS) SKH 51, produced by Hitachi Metals, Ltd., Japan. The chemical composition of this steel is presented in Table 1 Based on its carbon content, this steel is classified as high-carbon steel, with the addition of alloying elements such as Cr (Chromium), Mo (Molybdenum), W (Tungsten), and V (Vanadium). This type of steel is also categorized as tool steel, specifically high-speed steel (HSS), which is designed for high-speed applications and is required to have high hardness to maintain durability and performance.

**Table 1.** Chemical Composition of HSS SKH 51 Sample

| Element | % Weight |
|---|---|
| C | 0.87 |
| Cr | 3.95 |
| Mo | 5.00 |
| W | 6.24 |
| S | <0.001 |
| V | 1.82 |
| Si | 0.26 |
| Mn | 0.31 |
| P | 0.02 |
| Fe | Bal. |

*3.2. Microstructure analysis*

Figure 1 illustrates the microstructure of the SKH 51 sample at 200× magnification. The white phase is identified as carbide (C), the grey phase as retained austenite (γ'), and the black phase as martensite. In Figure (A) (NCT), the white phase (carbide) accounts for 7.059%, while the retained austenite is 21.719%. The NCT (non-cryogenic treatment) sample has the lowest carbide content and the highest retained austenite content among the samples. In Figure (B) (CT-80), which represents the cryogenic treatment sample at -80°C, the carbide content is 14.199%, while the retained austenite content is 12.662%. It can be concluded that the cryogenic treatment process successfully reduced the amount of retained austenite while increasing the carbide content. In Figure (C) (CT-110), which represents the cryogenic treatment sample at -110°C, the carbide fraction was measured at 15.156%, while the retained austenite content was identified as 9.787%.

In Figure (D) (CT-140), which represents the cryogenic treatment sample at -140°C, the carbide fraction increased to 16.485%, while the retained austenite content decreased to 8.172%. These results indicate that as the cryogenic treatment temperature decreases, the retained austenite content is further reduced, while the carbide formation is enhanced.

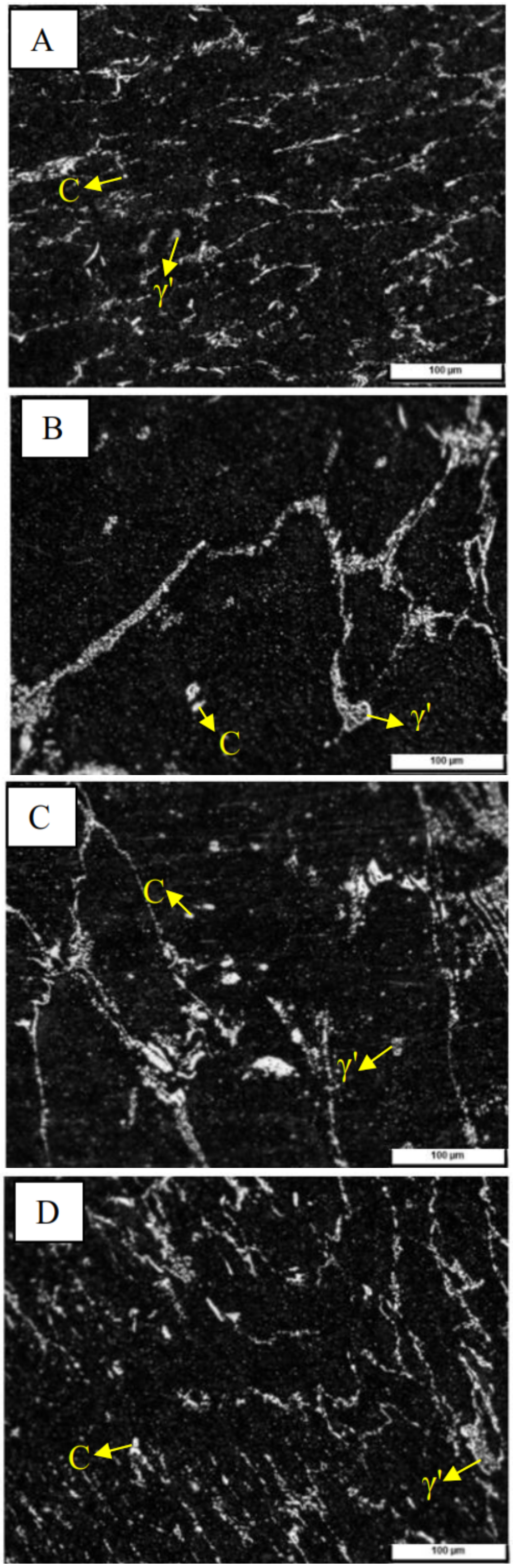


**Figure 1.** Microstructure of SKH 51 Sample Observed Under an Optical Microscope at 200× Magnification. (A) NCT; (B) CT-80; (C) CT-110; (D) CT-140 (γ' = retained austenite, C = carbide)

*3.3. Hardness testing analysis results*

The hardness test was conducted in accordance with the ASTM E-92 standard, using the Vickers microhardness method with a diamond indenter, and the results were recorded in HV units, which were subsequently converted into HRC units. The study produced a graph illustrating the relationship between heat treatment and hardness values, as shown in Figure 2. Cryogenic treatment is considered to play a significant role in enhancing the hardness of SKH 51 samples, primarily due to the increased formation of martensite during the cryogenic process. The formation of martensite is affected by the available transformation time from austenite. This phenomenon occurs because the martensite finish temperature (Mf) is significantly below room temperature, allowing martensite to continue forming at temperatures below room temperature [11]. The NCT and CT-80 samples show the same HRC values, which has a minimal impact since the cryogenic treatment temperature used for CT-80 was too high to induce significant changes. This condition falls into the category of shallow cryogenic treatment [12]. As the cryogenic treatment temperature was further lowered, an increase in hardness was observed in the CT-110 and CT-140 samples, reaching 65 HRC and 66 HRC, respectively. This finding is further supported by the microstructural analysis presented in Figure 3, which shows that the retained austenite content has decreased to less than 10% in the CT-110 and CT-140 samples. Concurrently, the martensite fraction increased to 73% in CT-110 and 75% in CT-140. In addition to martensite, carbides also play a crucial role in enhancing hardness [13]. The formation of carbides is affected by the alloying elements present in SKH 51, contributing to the improvement of its mechanical properties [14].

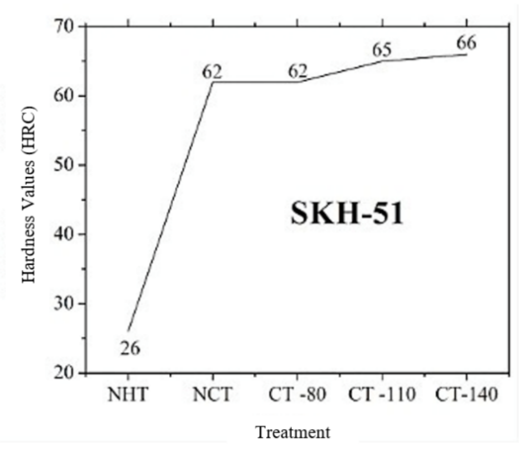


**Figure 2.** Effect of Treatment on Hardness

*3.4. Wear Test Analysis*

The samples that underwent treatment were tested according to ASTM G99 using the Ogoshi method, resulting in the graphs shown in Figures 3 and 4. From Figure 3, it can be observed that the abrasive resistance values decrease as the cryogenic treatment temperature is lowered. This decrease in abrasive resistance values indicates a reduction in mass loss during the wear test. The reduction in mass loss signifies that the material has become more resistant to friction [15] .

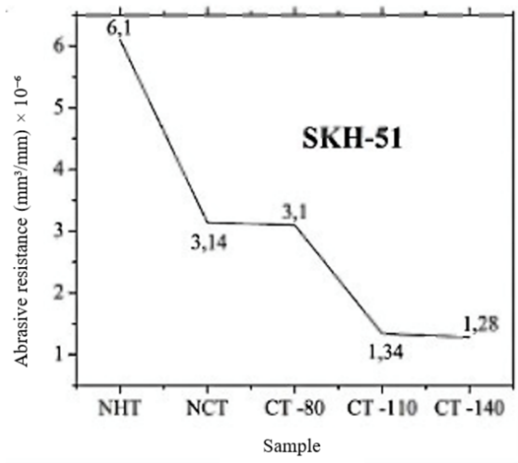


**Figure 3.** Effect of Treatment on Abrasive Resistance Values

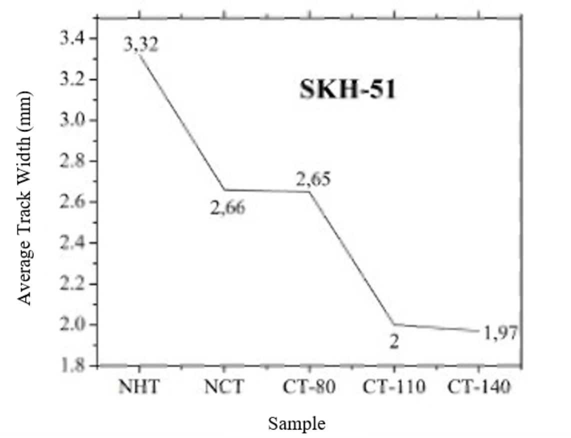


**Figure 4.** Effect of Treatment on Average Track Width

As shown in Figure 4, cryogenic treatment plays a significant role in reducing the average track width formed during testing. The use of the same abrasive media and applied pressure resulted in different average track widths, indicating that the area affected by wear decreased, thereby improving the wear resistance of the material [16]. From Figure 3 and Figure 4, it can be concluded that the lower the cryogenic treatment temperature, the greater the improvement in wear resistance, as evidenced by the reduced area affected by wear. The CT-140 sample, which underwent cryogenic treatment at the lowest temperature, showed the smallest average track width.

*3.5. XRD analysis results*

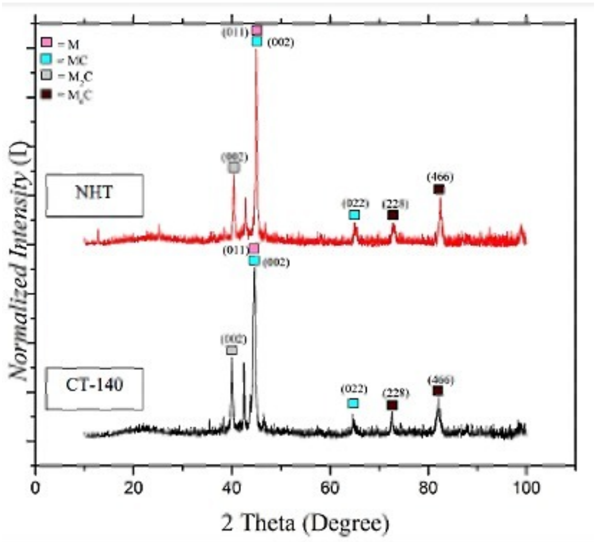


**Figure 5.** X-ray diffraction Pattern for CT-140 and NHT Samples

XRD testing was conducted to identify the compounds present in the material subjected to cryogenic treatment at a temperature of -140°C, as shown in Figure 5. In addition, the result for the NHT sample (non-heat treatment) as received are also shown in Figure 5 for contrast. From Figure 5, it is identified that for cryogenic treatment at a temperature of -140°C the compounds MC (002 and 022), $M_2C$ (002), and $M_6C$ (228 and 466) are present at the following 2-theta values: MC (44.3 and 64.4), $M_2C$ (41), and $M_6C$ (72.6 and 81.7). In Figure 5, it can also be observed that a shift in the diffraction pattern of the $M_2C$ compound occurred after the treatment process, with a shift of 0.1 degrees from 40.1 to 40, caused by microstrain. The calculation of microstrain was performed using the Williamson-Hall method [17], resulting in a value of 8.36 with a crystal size of 156.72771 Å. Microstrain itself refers to the plastic deformation happening in the material due to strain [18]. This microstrain is caused by the expansion of the material during heating, particularly during the austenitization process, followed by contraction during the quenching process. Furthermore, the material continues to contract during the cryogenic treatment due to the decreasing temperature, leading to shifts in the crystal structure of the material [19]. The shift in the diffraction pattern is also caused by the phase transformation from austenite to martensite. This phase transformation changes the crystal structure of the material from BCC (Body-Centered Cubic) to BCT (Body-Centered Tetragonal), leading to the phenomenon of microstrain [20]. This microstrain induces internal stress, which can easily initiate cracks when the material is in use.

## 4. CONCLUSION

The lower the cryogenic treatment temperature, the higher the hardness and wear resistance of the material. Additionally, the retained austenite content decreases, while the carbide formation increases. The best hardness and wear resistance results from the cryogenic treatment were observed in the CT-140 sample, with a hardness value of 66 HRC and wear resistance of $1.28 \times 10^{-6}$ $mm^3/mm$, accompanied by a retained austenite content of 5.37% and carbide content of 12%. These variations are caused by the uneven distribution of phases in the material.

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