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The influence of treatment on cold work tool steel SKD 11 with temperature variations on mechanical properties

Indah Uswatun Hasanah [a], ***Dedi Priadi*** [a], ***Donanta Dhaneswara*** [a], ***A. Ali Alhamidi*** [b]

[a]*Department of Metallurgical and Material Engineering, Universitas Indonesia, Depok, Indonesia*

[b]*Metallurgical Engineering Department, Universitas Sultan Ageng Tirtayasa, Cilegon, Indonesia*

[1]Corresponding author: indah.uswatun.h@gmail.com

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ABSTRACT

SKD 11 steel is a type of cold work tool steel that is popular and widely used in the manufacturing industry. SKD 11 steel is generally applied as dies, molds, cutting tools or others. Because of these applications. SKD 11 steel must have good wear resistance, hardness, dimensional stability and toughness. So to improve the characteristics of the steel, a heat treatment process is carried out, but due to the high carbon content and alloying elements in the tool steel, the martensite finish (Mf) temperature of the steel becomes lower, so that after the heat treatment process, usually the steel still leaves a lot of austenite phase. or commonly known as residual austenite. Residual austenite can affect the mechanical properties of SKD 11 steel, residual austenite can be removed by carrying out a cryogenic treatment process, this process is carried out after the quenching process. The heat treatment process this time was carried out using a vacuum furnace using an austenite temperature of 1040℃ and quenching using a medium in the form of nitrogen gas with a pressure of 3 bar. The cryogenic treatment process that was carried out afterwards was carried out by varying the temperature of -80℃, -110℃ and -140℃. The test results obtained were then analyzed using an optical microscope and XRD (X-ray diffraction), as well as hardness testing and impact testing to determine the mechanical properties of the steel. Based on the research results it is known that the resulting microstructure contains martensite matrix and carbide M_7C_3 as primary carbide and $M_{23}C_6$ as secondary carbide, then by carrying out the cryogenic treatment process can reduce the percent of residual austenite, the lowest percent of residual austenite is obtained from the results of cryogenic treatment at -140℃, which is equal to 1.15%. This is directly proportional to the hardness test results obtained, where the highest hardness value was also obtained by the sample with a value of 61.5 HRC, while the relationship with toughness is inversely proportional. The higher the hardness, the lower the resulting toughness.

ABSTRAK

Baja SKD 11 adalah salah satu jenis *cold work tool steel* yang populer dan banyak digunakan di industri manufaktur. Baja SKD 11 umumnya diaplikasikan sebagai *dies*, *moulds*, *cutting tool* ataupun yang lainnya. Karena aplikasinya tersebut, sehingga baja SKD 11 harus memiliki ketahanan aus, kekerasan, stabilitas dimensi serta ketangguhan yang baik. Sehingga untuk meningkatkan karakteristik baja tersebut dilakukan proses *heat treatment*, namun karena adanya kandungan karbon serta unsur paduan yang tinggi pada *tool steel*, maka temperatur *martensite finish* (Mf) baja tersebut menjadi lebih rendah, sehingga setelah proses *heat treatment* biasanya baja masih banyak meninggalkan fasa austenit atau yang biasa dikenal sebagai austenit sisa. Austenit sisa dapat mempengaruhi sifat mekanik pada baja SKD 11, austenit sisa dapat dihilangkan dengan melakukan proses *cryogenic treatment*, proses ini dilakukan setelah proses *quenching*. Adapun proses *heat treatment* kali ini dilakukan menggunakan *vacuum furnace* dengan menggunakan temperatur austenit sebesar 1040℃ dan di-*quenching* menggunakan media berupa gas nitrogen yang bertekanan 3 barr. Proses *cryogenic treatment* yang dilakukan setelahnya dilakukan dengan memvariasikan temperatur sebesar -80℃, -110℃ dan -140℃. Hasil pengujian yang didapatkan kemudian dianalisa menggunakan mikroskop optik dan XRD (*X-ray diffraction*), serta dilakukan pengujian kekerasan dan pengujian impak untuk mengetahui sifat mekanik dari baja. Berdasarkan hasil penelitian diketahui struktur mikro yang dihasilkan mengandung matriks martensit dan karbida M_7C_3 sebagai *primary carbide* serta $M_{23}C_6$ sebagai *secondary carbide*, kemudian dengan melakukan proses *cryogenic treatment* dapat menurunkan persen austenit sisa, persen austenit sisa yang paling rendah diperoleh dari hasil *cryogenic treatment* temperatur -140℃, yaitu sebesar 1.15%. Hal tersebut berbanding lurus dengan hasil pengujian kekerasan yang didapat, dimana nilai kekerasan tertinggi juga diperoleh sampel tersebut dengan nilai 61.5 HRC, sedangkan hubungannya dengan ketangguhan berbanding terbalik. Semakin tinggi kekerasan, maka ketangguhan yang dihasilkan semakin rendah.

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1. Introduction

Tool steel is commonly used in the manufacturing industry for making tools in working and forming processes for various materials, such as metals, wood, plastics, and other materials typically used in the industry [1, 2]. According to the Global Tool Steel Market, the use of tool steel continues to increase every year, especially in the Asia-Pacific region, which is expected to be the most prominent region in the global tool steel market [3]. This is partly due to the increasing industrialization in countries such as Indonesia, China, India, and others. One of the most popular types of tool steel is cold work tool steel, namely SKD 11. SKD 11 steel is commonly used in several industries for dies, molds, cutting tools, or other applications. Due to these applications, SKD 11 steel must have good wear resistance, hardness, dimensional stability, and toughness [4, 5]. In order to enhance the characteristics of the steel, a heat treatment process is carried out. However, due to the high carbon and alloy content in tool steel, the martensite finish (Mf) temperature of the steel becomes lower, resulting in the presence of retained austenite, which is the austenite that remains after the transformation [6]. The presence of retained austenite in steel can affect the dimensional stability and durability of tool steel, so retained austenite needs to be eliminated before tool steel is used [7, 8]. One way to eliminate retained austenite is by conducting a cryogenic treatment process. Cryogenic treatment is an additional process to conventional heat treatment that involves freezing the steel at cryogenic temperatures (below 0°C) with the aim of improving the mechanical properties of the steel.

A research has been conducted by Yang-Yu Su et al on the cryogenic treatment process of SKD 11, which successfully showed a decrease in retained austenite from 8.19% to 1.9% [9]. In addition, several studies have been carried out to vary the cryogenic time after the hardening process [10, 11]. Cryogenic treatment has also been carried out to obtain fine carbide precipitation in martensitic matrices that contain a lower amount of austenite [12, 13]. Such a microstructure can improve the performance of cryogenically processed tool steels.

In this research, the mechanical properties of SKD 11 steel subjected to cryogenic treatment and conventional heat treatment were compared, along with their respective effects on microstructural changes. The cryogenic treatment temperature was varied to determine the most optimal temperature.

2. Research Methodology

The SKD 11 samples were cut into three different dimensions: the first one was 10x20x30 mm, the second was 10x15x15 mm, and the third one was 10x10x55 mm for impact testing. Figure 1 shows the sample dimensions.

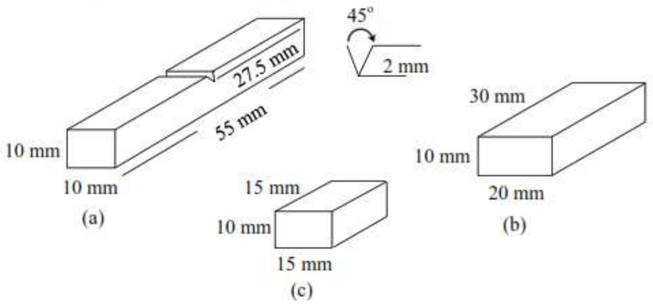

Figure 1. Sample Dimensions: (a) Impact Test Sample, (b) Hardness Test Sample, (c) Metallography and XRD Sample

The samples were subjected to two preheating procedures. The first preheat was conducted at 650°C for 30 minutes, followed by a second preheat at 840°C for 30 minutes. Then, the samples were heated at a temperature of 1040°C for 50 minutes. After the austenitization process, the samples were quenched in pressurized nitrogen gas at 3 bar until they reached room temperature. The samples then underwent cryogenic treatment by being placed in the sub-zero treatment machine and immersed in liquid nitrogen for 50 minutes, with temperature variations of -80°C, -110°C, and -140°C. Afterwards, the samples were placed in a vacuum furnace and heated at a temperature of 520°C for 6 hours.

Testing and characterization were carried out to determine the changes in mechanical properties of the samples. Micro hardness testing was conducted using the MHVS-1000AT micro hardness tester in accordance with ASTM E-384. Additionally, Charpy impact testing was performed in accordance with ASTM E23. The characterization methods used included optical microscopy to observe the microstructure of the sample surface after treatment, as well as the results of the impact test.

3. Result and Discussion

3.1. Microstructure Analysis

Figure 2 shows the microstructure of the as-received SKD 11 steel. As can be seen in the Figure 2, the phase formed is *pearlite phase* (ɑ + Fe3C), in addition to the *large primary carbide* and *fine secondary carbide* that are distributed in the *ferrite matrix* (ɑ). The *large primary carbide* is the M_7C_3 *carbide type*, while the *fine secondary carbide* is the $M_{23}C_6$ carbide type.

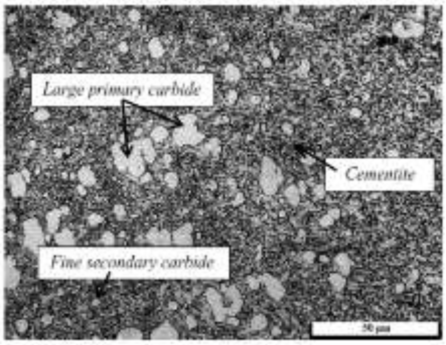

Figure 2. Microstructure of as-received SKD 11 steel

It can be seen from Figure 3 that there are two types of carbides present in the microstructure of the sample after conventional heat treatment. The carbides formed are of the M7C3 type, which is the primary carbide (PC), and M23C6 type, which is the secondary carbide. Secondary carbides can be distinguished into 2 based on their sizes, namely large secondary carbide (LSC) and small secondary carbide (SSC). M7C3 type carbides are usually recognized by their rod-shaped form, have large sizes, and are distributed in regions with high cooling intensity.

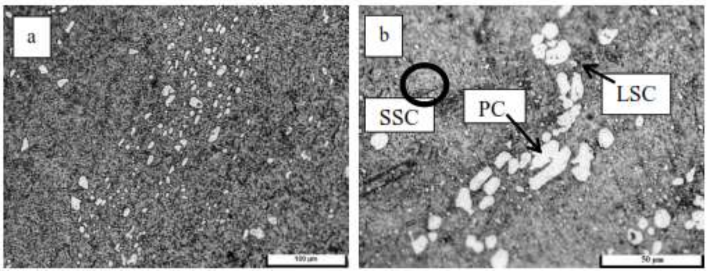

Figure 3. Microstructure of Conventional Heat Treatment Treatment at Magnification (a) 200x, and (b) 500x

The high carbon content leads to a high amount of primary carbide (M7C3) that does not dissolve during the austenitizing process. This is also influenced by the austenitizing temperature where at 1040°C, not all M7C3 can be dissolved. Type M23C6 carbide is a very fine type of carbide and is evenly distributed along the matrix. The M23C6 carbide present in the microstructure of the conventional heat treatment is formed due to precipitation during tempering process, where the carbon trapped in the martensite phase is released and reacts with chromium alloying elements to form Cr23C6.

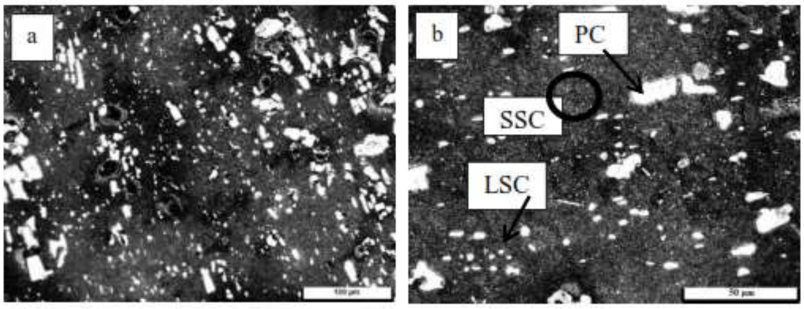

Figure 4. Microstructure of Cyrogenic Treatment at -80 °C - (a) 200x magnification, and (b) 500x magnificatio

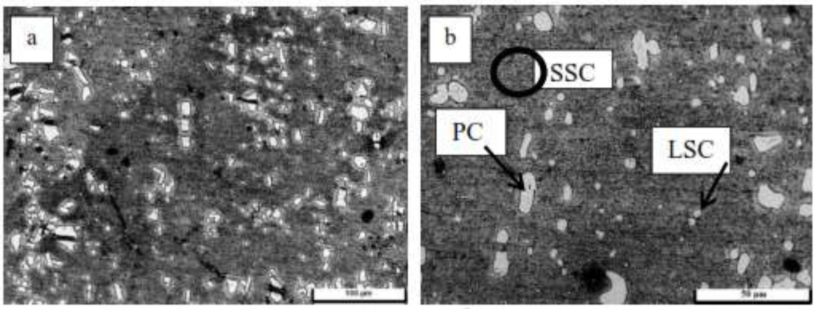

Figure 5. Microstructure of Cyrogenic Treatment at -110°C - (a) 200x Magnification and (b) 500x Magnification

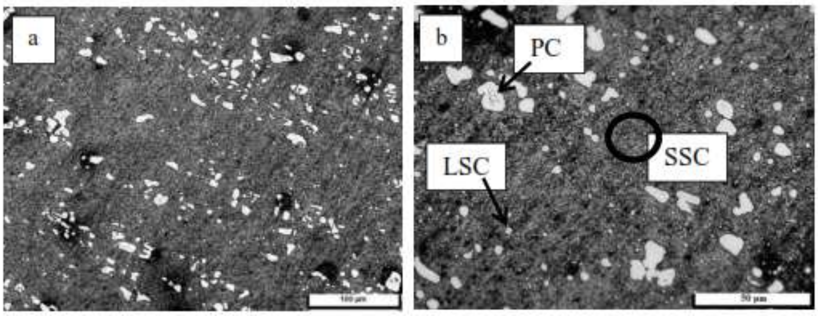

Figure 6. Microstructure of Cyrogenic Treatment -80\140OC Treatment (a) 200x, and (b) 500x Magnification

The microstructure generated from each cryogenic temperature variation of the sample is shown in Figures 4, 5, and 6. From these three microstructures, it can be observed that there is martensitic matrix and carbides with different percentages of residual austenite and volume fraction of carbides.

In Figure 7, the relationship between treatment types and the percentage of carbide volume and retained austenite volume is shown. It can be observed that the lower the cryogenic treatment temperature, the higher the percentage of carbide volume in the steel's microstructure, and conversely, the lower the temperature, the lower the percentage of retained austenite in the steel after cryogenic treatment. In cryogenic treatment at -140℃, the highest carbide volume fraction of 14.5% and the lowest remaining austenite volume percentage of 1.15% were produced.

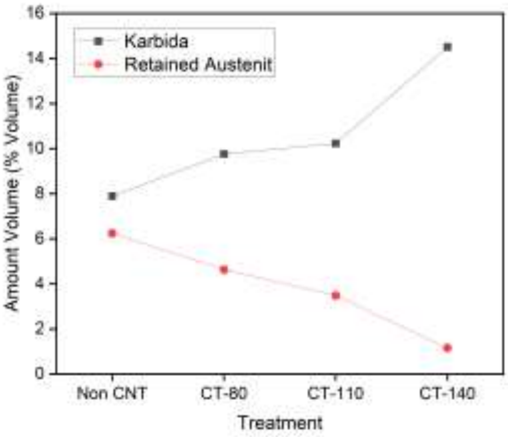

Figure 7. The Influence of %Volume of Carbide and Remaining Austenite on the Type of Treatment Performed

Table 1 shows the size of primary carbides, large secondary carbides, and small secondary carbides for several treatments. Samples treated with cryogenic *treatment* at a temperature of -140℃have the smallest carbide size compared to the other three treatments. As the *cryogenic treatment* temperature decreases, the percentage of carbide volume increases but the size of the carbide decreases. The small size of the carbide in the cryogenic treatment at -140℃ is due to the decrease in the remaining austenite percentage contained in the sample. The smaller the percentage of retained austenite, the more interstitial carbon in the martensitic matrix, which will diffuse and form carbides with alloying elements. The carbides formed at cryogenic temperature of -140℃undergo repeated nucleation, resulting in smaller carbide size.

Table 1. Results of PC, LSC, and SSC Measurements on Samples with Different Treatments

Sample	Primary carbide (PC)	Secondary carbide (SC)	
		Large Secondary carbide (LSC)	Small Secondary carbide (SSC)
	µm	µm	µm
Conventional Heat treatment	68.45	3.92	0.79
Crygonenic Treatment (-80℃)	55.45	3.85	0.63
Crygonenic Treatment (-110℃)	49.42	3.5	0.5
Crygonenic Treatment (-140℃)	42.76	2.81	0.4

3.2. *Identification of Carbide Types*

XRD (x-ray diffraction) analysis was conducted to identify the phases and types of carbides formed in SKD 11 steel after treatment as directed by Figure 8. The XRD pattern of as-received SKD 11 shows peaks of α-iron (ferritic), γ-iron (austenitic), M7C3 carbide, and M23C6 carbide phases. Cryogenic treatment at -140°C shows the same XRD pattern as that of as-received, with a decrease in the intensity of (111)γ and (004)γ peaks. This indicates that a certain amount of austenite phase decreased after the cryogenic treatment process at a temperature of -140°C, which is due to the transformation of the austenite phase into the martensite phase.

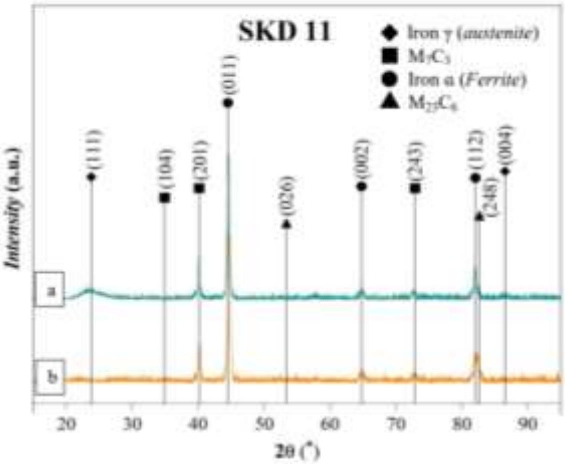

Figure 8. X-Ray Diffraction Patterns of (a) *As-Received* SKD 11 Steel Sample and (b) SKD 11 Steel Sample After *Heat Treatment* + *Cryogenic Treatment* at -140°C

3.3. Hardness

As seen in Figure 9, there is a graph of the type of treatment against the hardness values. The values obtained from the four steel samples are 61.5, 59.8, 59, and 58.3 HRC, respectively. The increase in hardness values generated by each sample is not significantly different. The increase in hardness values is influenced by the amount of remaining austenite phase that transforms into the martensite phase. The increase in hardness value in SKD 11 steel is also influenced by carbide precipitation and carbide fraction distribution in the sample. As mentioned earlier, the lower the cryogenic treatment temperature, the higher the percentage of carbide volume. The increase in carbide volume will decrease the percentage of remaining austenite contained in the steel. This is consistent with the data on the hardness value of samples treated with cryogenic treatment at a temperature of -140°C, which has the highest hardness value.

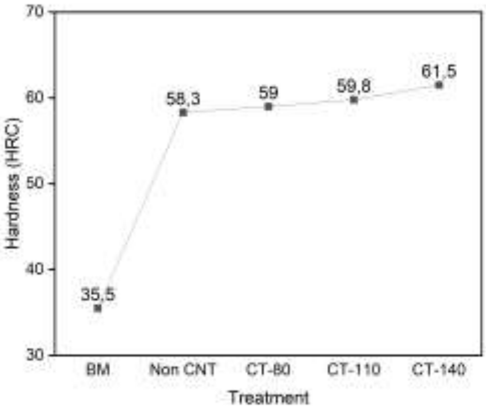

Figure 9. The Effect of Hardness Results on SKD 11 Steel for Different Types of Treatment

3.4. Impact Testing

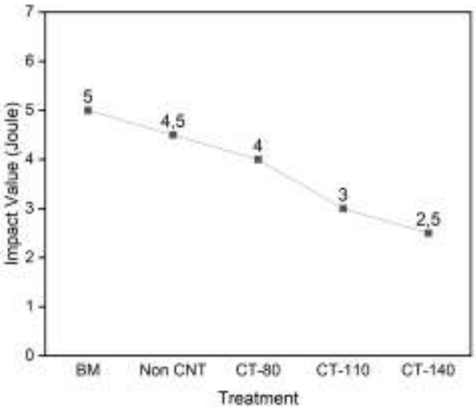

Figure 10. The Effect of Toughness Results on SKD 11 Steel for Different Types of Treatment

Impact testing aims to determine the toughness property of a material. In impact testing, data on absorbed energy values are obtained, and this energy value indicates how tough the material is in receiving shock loads. Figure 10 shows a graph of each treatment against impact value.

Based on Figure 11, it can be concluded that the highest toughness value is found in the non-cryogenic treated sample, which is 4.5 J, while the lowest toughness value is found in the steel resulting from cryogenic treatment at -140°C, which is 2.5 J. The toughness value is influenced by the size of the carbide; the smaller the carbide size and the higher the volume fraction of carbide, the lower the obtained toughness value. Furthermore, in Figure 14, there is a microstructure of the fracture surface of the five SKD 11 steel samples at 50x magnification. From Figure 14, it can be concluded that the smaller the carbide size and the higher the carbide volume fraction, the lower the resulting toughness value. It can also be seen from Figure 12 that the four samples from cryogenic treatment have smaller carbide sizes and homogeneous distribution, which results in a decrease in toughness value.

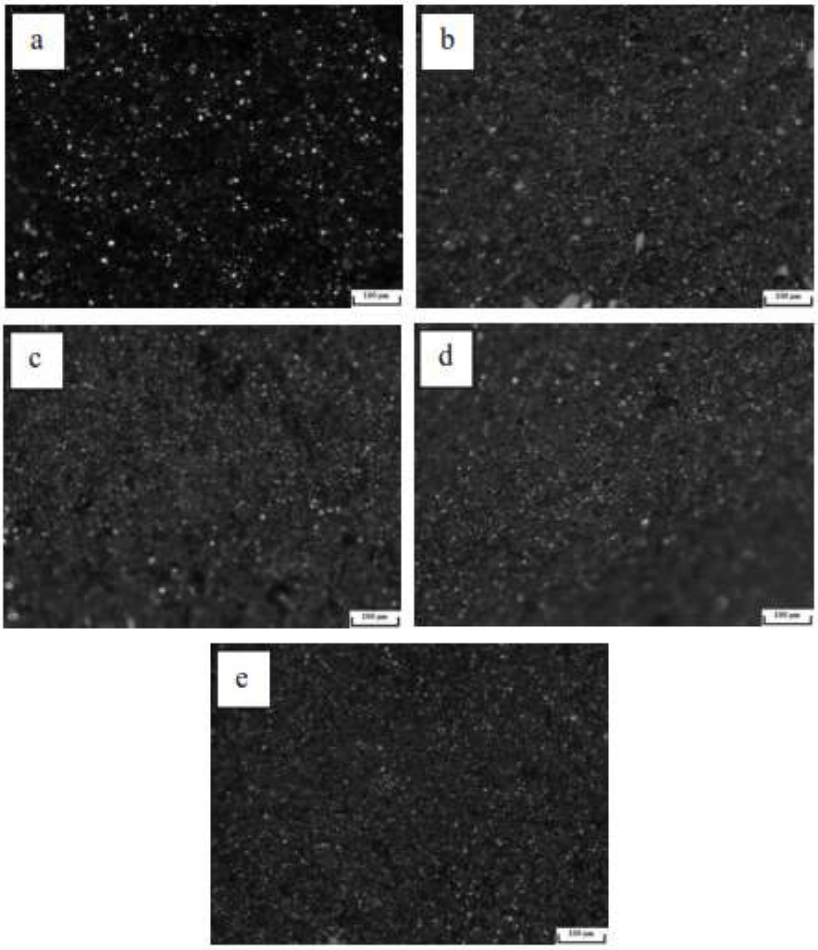

Figure 11. Microstructure of Fracture Surface in Toughness Test Results of (a) As-Received SKD 11, (b) Conventional Treatment, (c) Cryogenic Treatment at -80℃, (d) Cryogenic Treatment at -110℃, (e) Cryogenic Treatment at -140℃

4. Conclusion

Based on the research conducted on the effect of cryogenic treatment on SKD 11 with various temperatures on mechanical properties and microstructure, it can be concluded that:

1. The microstructure of the heat treatment and cryogenic treatment samples contains a matrix that is mostly martensitic phase with the dispersion of M7C3 and M23C6 carbides along the matrix.
2. The results of the hardness values obtained from each sample are directly proportional to the volume fraction of carbides produced, where the higher the volume fraction of carbides in the sample, the higher its hardness, however, conversely, the sample hardness will increase as the residual austenite percentage and carbide size decrease. The highest hardness value was obtained in the cryogenic treatment sample at -140℃, which is 61.5 HRC with a residual austenite percentage calculated according to ASTM E975-00 standards of 1.15%.
3. The results of the impact values obtained from each sample are inversely proportional to the hardness values obtained, where the higher the hardness value of the sample, the lower the toughness value obtained from the sample, The highest toughness value was obtained from the sample with conventional heat treatment, which was 4.5 J with only a hardness value of 58.3 HRC.

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