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Review Article

Cryogenic treatment of tool steels: A brief review and a case report

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Abstract

Tool steels used in marine industries demand an effective approach to the enhancement of their properties. Normally, conventional heat treatment is widely used to increase the performance of tool steels. However, this method cannot fully enhance tool steel performance. On the other hand, cryogenic treatment is a supplemental process to conventional heat treatment; it can promote the conversion of retained austenite to martensite and accelerate the precipitation of fine carbides. In this paper, a systematic review of the cryogenic treatment of tool steels is presented. A wide range of useful investigations is reviewed, particularly in the details of the transformation of retained austenite to martensite and the precipitation of fine carbides. A case study of a tool steel subjected to conventional heat treatment, conventional cold treatment, and deep cryogenic treatment is also given and discussed in order to give an insight in the cryogenic treatment of tool steels.

1. Introduction

The improvement of tool steels to gain better mechanical properties and machinability is obviously required as a reply to the fast development of marine industries (Orlowicz et al., 2015; Singh, 2016). As a response to such demand, different methods to develop the quality of tool steels used in marine applications are presently being studied and employed. One of classical methods to increase the performance of tool steels is conventional heat treatment (Carlson, 1990; Chandler, 1995). This method is regarded as a practical method commonly applied to tool steels, performed by heating to a selected temperature, exposing a tool steel for a period of time at that temperature, and then cooling down to room temperature to gain the desired microstructure, usually martensite (Stratton, 2007; Jurci, 2017). A tempering process is then performed so that high hardness and toughness tool steels can be obtained. Conventional heat treatment is normally conducted at a temperature above 0 °C, and its application in tool steels has been known for long time. However, tool steels usually contain high amounts of carbon and alloying elements. This special characteristic of tool steels can lower the temperature of the martensite start and finish temperatures (Popandopulo & Zhukova, 1980; Baldissera & Delprete, 2008). Basically, the martensite finish temperature of such high alloy steels is always much below the environment temperature. Therefore, the traditional heat treatment fails to transform the amount of retained austenite into

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martensite (Jurci, 2017; Popandopulo & Zhukova, 1980; Baldissera & Delprete, 2008; Gavriljuk et al., 2013); as a result, the performance of tool steels cannot fully be enhanced (Jurci, 2017; Popandopulo & Zhukova, 1980; Baldissera & Delprete, 2008; Gavriljuk et al., 2013; Chopra & Sargade, 2015). Since the last century, the cryogenic treatment of tool steel has been widely recognized as an alternative approach which can be employed as a supplemental process between the hardening and tempering processes of conventional heat treatment (Chopra & Sargade, 2015; Kamody, 1999).

Cryogenic treatment is typically performed by cooling down tool steels to a designed sub-zero temperature and holding them for a certain period (Baldissera & Delprete, 2008; Gavriljuk et al., 2013; Chopra & Sargade, 2015; Kamody, 1999; Jurci et al., 2018). The aim of this approach is to obtain desired microstructures, such as the precipitation of fine carbides on the martensite matrix containing lower amount of austenite (Jurci et al., 2018; Dumasia et al., 2017). Such microstructures can increase the performance of cryogenically treated tool steels. Obviously, cryogenic treatment can change mechanical properties with modified microstructures, and the efficiency of this treatment has already been reported by several authors. However, a systematic review, with a case report, on this topic is still lacking. Thus, this paper presents a review of the modification of microstructures, especially carbide precipitation. Additionally, a case report of a tool steel subjected to cryogenic treatment and conventional heat treatment is presented and discussed, particularly in terms of modified microstructures and hardness evolution.

2. Materials and methods

2.1 Cryogenic treatment process

Since the last decade, cryogenic treatment has been extensively recognized and accepted as an effective supplemental technique to traditional heat treatment (Popandopulo & Zhukova, 1980; Baldissera & Delprete, 2008; Gavriljuk et al., 2013; Chopra & Sargade, 2015; Kamody, 1999; Jurci et al., 2017). Cryogenic treatment has also been regarded as an approach in which tool steels are cooled down and held at a sub-zero temperature for a certain period in order to obtain distinct properties, i.e., increased hardness and improved wear resistance (Das et al., 2010a; Das et al., 2010b). Nowadays, this supplemental process plays an essential role in developing the quality of tool steel (Reitz & Pendray, 2001; Prudhvi & Lakshmi, 2016). This process mostly deals with cooling tool steels to a selected subzero temperature, soaking them at that cryogenic temperature for a certain period of time, and then heating them to ambient temperature. A schematic diagram illustrating the supplemental cryogenic treatment is given in **Figure 1**.

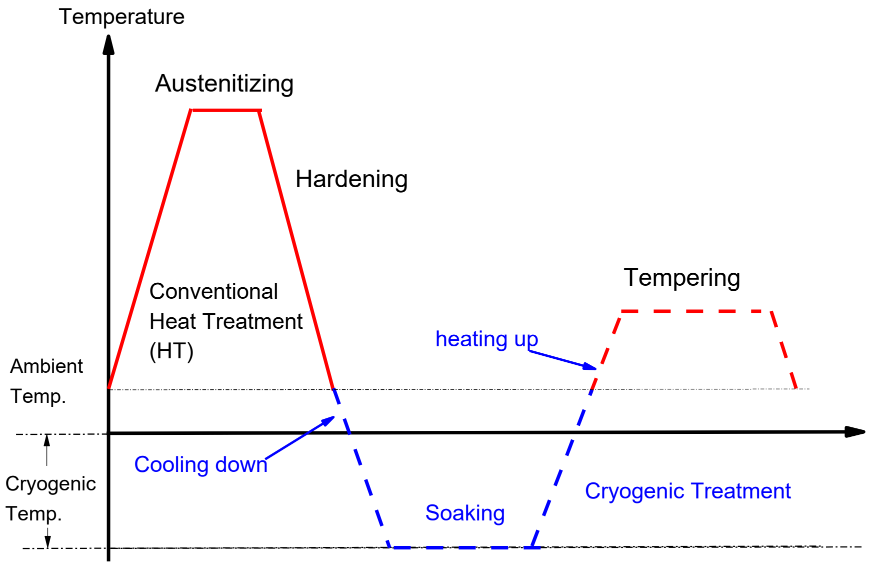

Figure 1 Schematic diagram of cryogenic treatment.

Significant parameters in cryogenic treatment affecting tool steel efficiency are tool steel type, the rate of cooling and heating, and the soaking period and temperature. Hence, cryogenic treatment of tool steels should be performed under a controlled cabinet with a cryogenic system (Diekman, 2013; Zurecki, 2005). A schematic presentation of cryogenic treatment employed in a controlled chamber with a cryogenic system is provided in **Figure 2**. Liquid nitrogen is commonly used as a cryogenic liquid in the cryogenic treatment of tool steels (Zurecki, 2005; Kalia, 2010). During treatment, cooling and heating rates should be kept constant to avoid the thermal cracking of cryogenically treated tool steels (Diekman, 2013; Zurecki, 2005; Kalia, 2010).

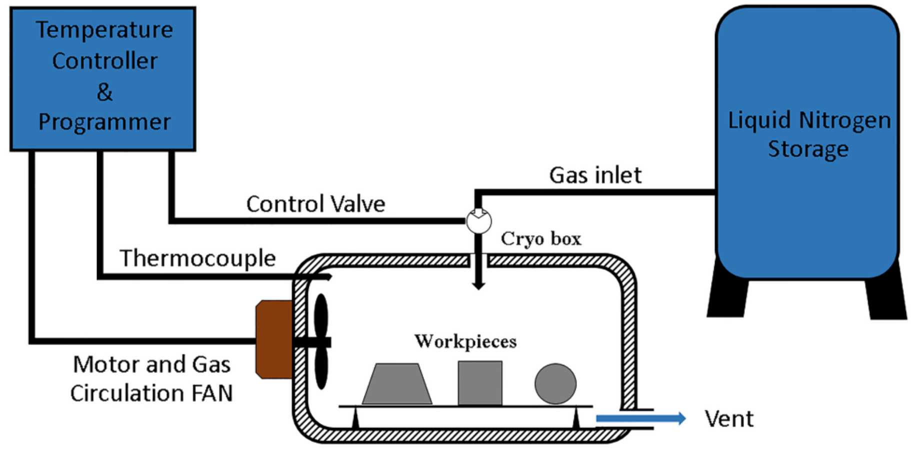

Figure 2 Schematic presentation of cryogenic treatment employed in a controlled chamber.

2.2 Classification of cryogenic treatment

Cryogenic treatment can be classified into three different temperature systems (Niessen et al., 2018; Gill et al., 2010). The first type is conventional cold treatment (CCT). In this treatment, temperature is cooled to −80 °C. CCT was the first cryogenic treatment applied in industries, and it was once believed that a cryogenic temperature around −80 °C was sufficient to convert retained austenite into martensite, resulting in improved strength, dimensional stability, and wear resistance (Niessen et al., 2018; Barron, 1982). The second is shallow cryogenic treatment (SCT), taking place between −80 and −160 °C (Sonar et al., 2018; Podgornik et al., 2016). Deep cryogenic treatment (DCT) is the third. In this treatment, tool steels are cooled to a cryogenic temperature below −160 °C (Podgornik et al., 2016; Senthilkumar & Rajendran, 2014). SCT can facilitate the transformation of residual retained austenite into martensite and increase the formation of fine carbides precipitated from the matrices of tool steels (Senthilkumar & Rajendran, 2014; Joshi et al., 2015). DCT can promote the modification of residual retained austenite into martensite and accelerate the precipitation of fine distributions of carbides (Joshi et al., 2015; Senthilkumar & Rajendran, 2011). In fact, each cryogenic treatment shows different results for different tool steels, and the usefulness of each treatment has been compared by several authors. For example, Barron carried out conventional heat treatment, CCT, and DCT on a set of different tool steels. His work reported that, in comparison to conventional heat treatment, the wear resistance of tool steels increased by CCT, and increased significantly by DCT. Meng et al. (1994) investigated the effects of cryogenic treatment on AISI D2 tool steel and pointed out that hardness increased by cryogenic treatment as compared to conventional heat treatment. The hardness of AISI D2 tool steel was found to be increased in the following order: CCT, SCT, and DCT. Collins and Dormer (1997) examined the hardness and abrasive wear rate of AISI D2 subjected to different cryogenic conditions in comparison to those with conventional heat treatment. Their results indicated that increased hardness and decreased wear rate were gained by all of the cryogenic treatments, and the effects of cryogenic treatment was more pronounced in deep cryogenically treated AISI D2. From the examples of the works mentioned above, it would be understandable that deep cryogenic treatment is considered to be a new challenge in the world of heat treatment which can provide considerable improvement in the hardness and wear resistance of tool steels.

3. Microstructure and mechanical property modification

3.1 Conversion of retained austenite

Technically, tool steels contain high amounts of carbon and alloying elements, resulting in lowering their martensite start and finish temperatures. In particular, martensite finish temperature becomes much lower than the environment temperature for tool steels (Das et al., 2010a; Das et al., 2010b; Das et al., 2009). Thus, the microstructure obtained from the hardening process of the traditional heat treatment contains a mixture of retained austenite and martensite. Retained austenite is soft in nature (Ahmad et al., 2018; Mohammed et al., 2020; Peeratatsuwan & Chowwanonthapunya, 2020). Hence, this phase produces negative effects on the desired properties, i.e., hardness, strength, and wear resistance (Chowwanonthapunya & Peeratatsuwan, 2020). Additionally, this phase is unstable, and can potentially be converted into martensite when tool steels are in service, resulting in change in the dimensions and in distortion of tool steels (Das et al., 2009; Kara et al., 2020). Hence, reduction of the amount of retained austenite is of great significance for the improvement of tool steel performance.

The cryogenic treatment of tool steels was introduced by Scott (1920). The works from Scott have illustrated the positive effects gained from cryogenic treatment, particularly from the point of stability and the life time of tool steels. Vanvlack (1998) reported the effects of cold treatment on a tool steel, in which an increased amount of martensite in the tool steel could be achieved when the quenching temperature further decreased to −78 °C. Mohan et al. (2001) showed that the transformation of retained austenite into martensite occurs at a constant temperature process. Bensely et al. (2005) investigated hardness, wear resistance, and variation in the amount of retained austenite of En 353 steel treated by traditional heat treatment, CCT, SCT, and DCT. His works displayed that, with respect to conventional heat treatment, CCT, SCT, and DCT enhanced the hardness and wear resistance of EN 353 steel. In particular, DCT improved wear resistance by 372 % and hardness by 3.5 %. Reduction in the amount of retained austenite was also found in CCT, SCT, and significantly in DCT. From all of works provided above, it can be concluded that the quality of tool steels can be improved by cryogenic treatment, especially by DCT. One of major mechanisms is the transformation of retained austenite into martensite, resulting in improvement in the dimensional stability, hardness, and wear resistance of tool steels.

3.2 Precipitation of carbide

Several papers conducted by pioneer metallurgists suggest that cryogenic treatment promotes the formation of carbide precipitated out from martensite, thus enhancing hardness and wear resistance. Bensely et al. (2011) investigated the effects of cryogenic treatment on residual stress. The results exhibited that the reduction of residual stress was observed in CCT and SCT, but more significantly in DCT. The reduced residual stress in cryogenic treatment was contributed to by the precipitation of fine carbides. Thus, the precipitation of fine carbides can be promoted by CCT and SCT, but particularly by DCT. Akhbarizadeh et al. (2009) examined the influences of cryogenic treatment on the hardness and wear resistance of D6 tool steel using SCT and DCT. The results reported that both cryogenic treatment conditions reduced the amount of retained austenite, resulting in improvement of the hardness and wear resistance of the cryogenically treated D6 tool steel. Since more uniform distribution of precipitated carbides was found in deep cryogenically treated D6 tool steel, DCT for D6 tool steel showed more enhancement in hardness and wear resistance in comparison to SCT for D6 tool steel. Zhu et al. (2008) conducted cryogenic treatment on Fe-Cr-Mo-Ni-C-Co alloy. They found precipitation of very fine carbides (η-Fe_2C) in the alloy steel subjected to DCT. These very fine carbides and their uniform distribution were attributed to the increased harness and wear resistance of deep cryogenically treated alloy steel. Das et al. (2010a) studied the effect of cryogenic treatment on D2 tool steel and focused on carbide precipitation. They indicated that cryogenic treatment accelerated the formation of secondary carbides, which are very fine in nature. The formation of these secondary carbides was found to be considerably higher in D2 tool steel with DCT than in that with CCT and SCT. Clearly, the examples of research given above suggest that cryogenic treatment facilitates the formation of fine carbides. In particular, DCT offers more improvement than CCT and SCT in the hardness and wear resistance, because this treatment promotes the formation of more residual fine carbides and increases carbide density.

4. A case study

SKD 11 tool steel, with the chemical compositions as listed in **Table 1**, was used in this case study because it can represent one of the most common tool steels used for typical applications in marine industries (Chowwanonthapunya, 2019).

Table 1 Chemical compositions of SKD11 tool steel in this study.

Specimens	Chemical Compositions (wt.%)							
	C	Mn	Cr	Si	Ni	Mo	V	Fe
SKD 11	1.52	0.47	12.1	0.4	0.53	1.1	0.42	Bal.

In the case study, investigation of the effects of CCT and DCT on the microstructure of SKD 11 tool steel was conducted. Specimens were prepared, of a size of 30×60×15 mm^3, and then subjected to conventional heat treatment (HT), CCT, and DCT. For conventional heat treatment (HT), specimens were austenitized at 1,100 °C for 30 min and then hardened by air cooling. For cryogenic treatments, liquid nitrogen was used to further cool hardening specimens from room temperature to each designed sub-zero temperature condition. CCT and DCT were performed in a liquid nitrogen chamber and held in specific cryogenic temperatures of –20 and –175 °C, respectively, in the closed chamber for 5 h. All conventionally heat treated and cryogenically treated specimens were then tempered at a temperatue of 300 °C for 2 h to eliminate residual stress from the hardening process. Light optical microscopy was employed to examine the microstructures of samples subjected to CCT and DCT in comparison to those that experienced conventional heat treatment. The hardness test was conducted on all treated specimens using an MMT-X3A Rockwell hardness tester. This test was carefully performed with 150 kg for major load with a dwell time of 15 s to obtain the average hardness value of treated specimens, estimated from five measurements. Wear test was also carried out using a pin-disc machine to evaluate the wear resistance of samples treated by different treatment conditions. The sample was placed perpendicularly on a stationary plate where its surface could be pressed against the abrasive actions provided by a moving disc covered with abrasive paper (Emery, 80-grade size). The perpendicularly-fixed load of this test was set at 15 N with a constant speed of the moving disc of 160 rpm. The radius of the abrasive wheel was set at 6 mm with a sliding distance (S) of 350 m. Each sample prior and after the test was weighed and then divided by the density of steel to obtain the wear volume loss (ΔW). ΔW could further be calculated to identify wear factor (K), the reciprocal of which could reflect the wear resistance performance of each sample. According to the Archard equation, a set of equations used to evaluate the wear resistance performance (WP) of this present case study was given as follows (Reitz & Pendray, 2001; Prudhvi & Lakshmi, 2016):

$$\Delta W = (W_{\text{initial}} - W_{\text{final}}) / D \tag{1}$$

where W_{initial} and W_{final} mean the initial and final weight after wear test, and density of steel (D) was assumed to be 7.83 g/cm^3) (Das et al., 2010a).

$$\Delta W = KFS \tag{2}$$

$$WP = 1/K = FS / \Delta W \tag{3}$$

Figure 3 displays micrographs at 50× and 100× magnifications of samples subjected to conventional heat treatment, CCT, and DCT. As cleary seen from **Figures 3(a)** and **3(b)**, conventionally heat treated samples contained non-uniform distributions of coarse carbides on the matrices of the specimens. As indicated by arrows in **Figure 3(b)**, the large white particles represent primary carbides (PC), which are coarse in nature. **Figures 3(c)** and **3(d)** displays the micrographs of samples obtained from CCT. As indicated in circles 1 - 4, **Figure 3(c)** shows the initial evolution of secondary carbides precipitated from the matrix of a conventionally cold treated sample. **Figure 3(d)** exhibits secondary carbides with a higher magnification, as marked in ovals 1 and 2. It is clear from **Figure 3(d)** that the secondary carbides were smaller than the primary carbides. **Figure 3(e)** displays micrographs at 50× of samples obtained from DCT. Obviously, more fine secondary carbides were precipitated from the matrix, as indicated by circles 1 - 4. **Figure 3(f)** shows secondary carbides with a higher magnification, as marked in circles 1, 2, and 3. In comparison to the micrographs of the conventionally cold treated sample, deep cryogenic treatment significantly increased the amount and density of fine secondary carbides, resulting in a more uniform dispersion of secondary carbides in the matrix of the deep cryogenically treated tool steel.

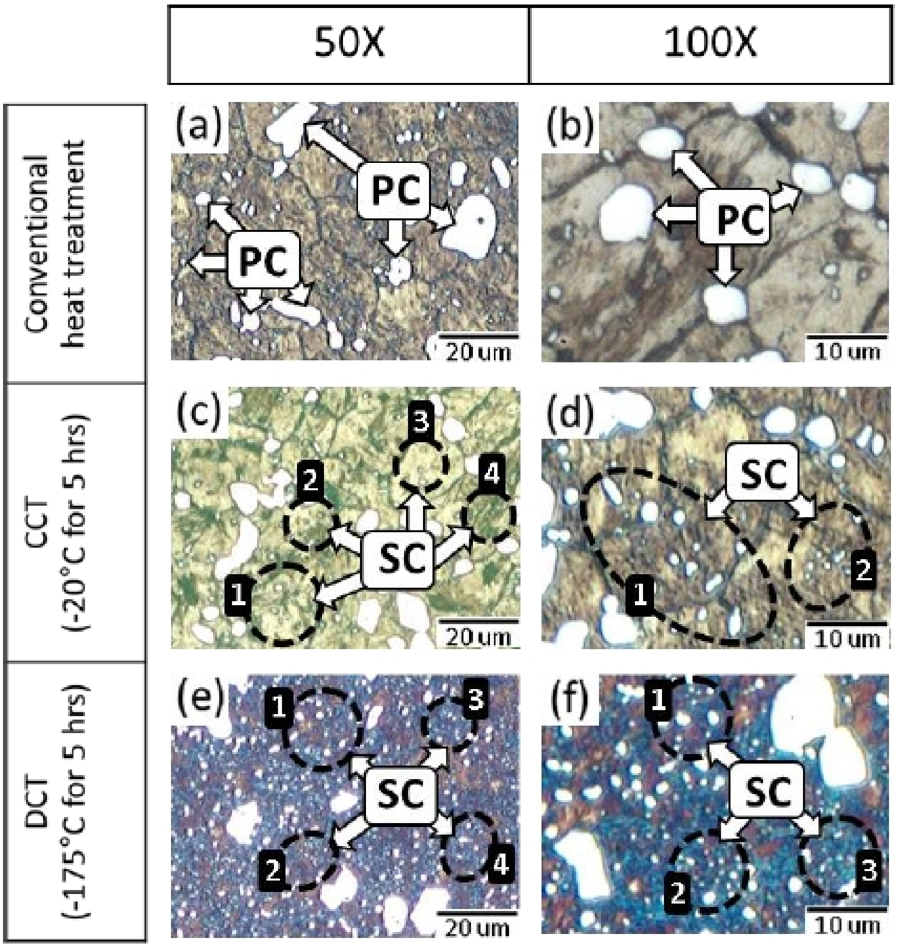

Figure 3 Micrographs of samples subjected to (a-b) HT, (c-d) CCT, and (e-f) DCT.

Figure 4 shows the evolution of the hardness of tool steel specimens subjected to three kinds of treatments. It is obvious from **Figure 4** that conventional cold treatment and deep cryogenic treatment improved the hardness of tool steel with respect to conventional heat treatment.

Figure 5 exhibits the wear behavior of specimens with three different treatment conditions. It is obviously found from **Figure 5(a)** that conventional heat treatment showed the highest wear weight loss, followed by cold treatment and deep cryogenic treatment. Conversely, deep cryogenic treatment exhibited the highest wear resistance, followed by conventional cold treatment and conventional heat treatment. These results mean that the wear resistance of SKD 11 tool steel can be significantly improved. Usually, the precipitation of fine secondary carbides and their density results in an increased dispersion hardening effect. Transformation of retained austenite to martensite also provides a hardening effect. Therefore, the improvement in the hardness and wear resistance performance of SKD 11 tool steel specimens treated by cryogenic treatment can be attributed to the presence of precipitated secondary carbides and the increased amount of martensite transformed from the retained austenite during the cryogenic treatment. In addition, the effect of cryogenic treatment on the microstructure modification of this tool steel was found to be more pronounced in deep cryogenically treated tool steel than in cold treated tool steel. This finding can be attributed to the significant increase in the density of fine secondary carbides and their uniform distribution in deep cryogenically treated tool steel.

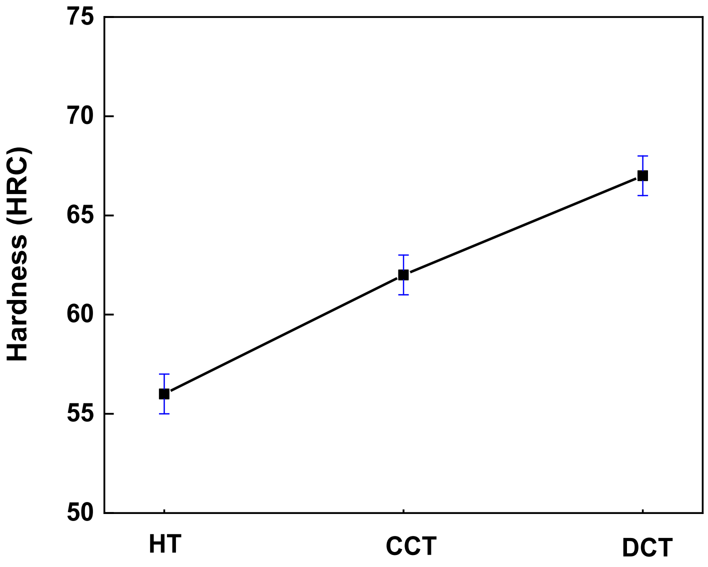

Figure 4 Measured hardness of samples subjected to HT, CCT, and DCT.

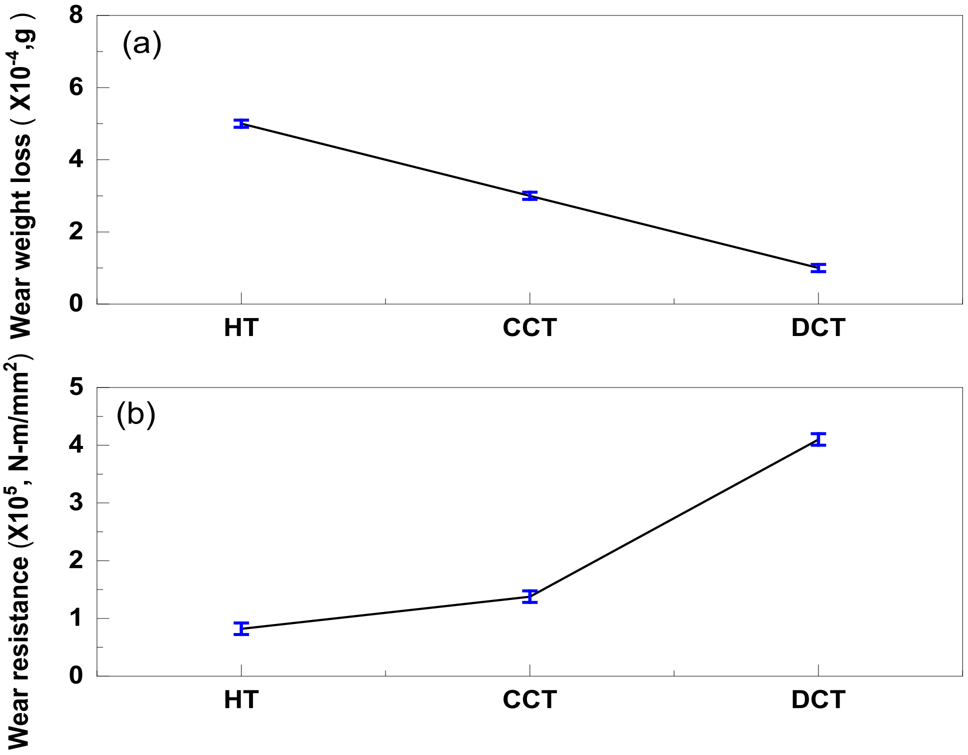

Figure 5 Measured hardness of specimens subjected to HT, CCT, and DCT.

5. Conclusions

The cryogenic treatment of tool steels has been already reviewed, particularly in terms of the process of cryogenic treatment and the modification of microstructures and mechanical properties. The case study of two cryogenic treatments, conventional cold treatment and deep cryogenic treatment, with respect to the conventional heat treatment, has also been presented. From this present review, the following can be concluded.

1) A wide range of studies indicated that cryogenic treatment can improve the hardness and wear resistance of tool steel. This supplemental sub-zero process for conventional heat treatment can help improve the quality of tool steels.

2) Principally, cryogenic treatment resulted in the increased hardness of tool steels by two main mechanisms: the transformation of retained austenite into martensite, and the precipitation of fine secondary carbides.

3) Deep cryogenic treatment provided a significant increase in the density of fine secondary carbides and promoted a uniform distribution of secondary carbides, leading to significant improvement in the hardness of tool steels.

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