Effects of cryogenic treatment on mechanical property and microstructure of JIS SKH51 high-speed steel

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Abstract. When SKH 51 high-speed steel was first cryogenically treated and then tempered at 540℃, the surface hardness of the SKH 51 steel increased from HRC 64 to HRC 65. This result indicates the reduction of retained austenite as well as the secondary precipitation of tiny carbides. TEM analyses indicated that cryogenic treatment can induce high-density dislocations and stacking faults within the martensite matrix. In the subsequent tempering process, conducted at 540℃, tiny secondary carbides precipitated nearby the existing dislocations and stacking faults, resulting in the hardening of SKH 51 high-speed steel. These secondary carbides mainly consisted of V-rich MC carbides and (Mo, W)-rich M2C carbides, which were identified by STEM/EDS techniques. After being cryogenically treated and then tempered at 590℃, both the surface hardness and wear resistance of SKH 51 high-speed steel were weakened below acceptable ranges. This result indicates the promotion of rapid decomposition and growth of tempered martensite. Therefore, to achieve the best surface hardness, SKH 51 high-speed steel should first be cryogenically treated, and then tempered at a temperature below 590℃.

1. Introduction
Cryogenic treatments, namely the treatment processes involving continuous cooling of materials to -196℃, have been widely used in the manufacturing of various high-precision components of precision machinery. Cryogenic treatment has been acknowledged as an effective method of microstructure refinement, residual stress relief, and dimensional stability, and shown to induce improvements in the mechanical properties of machine components, thus enhancing the service life of tools [1-9]. As a result, several companies in Taiwan have shown great interests in cryogenic treatment technology and instrumentations.

Cryogenic treatment provides some processing benefits in wear performance, thermal stability, homogenization of hardness distribution, and enhancement of tool life, among other mechanical properties. However, the effects of cryogenic treatments on the microstructure and mechanical properties of high-precision steels and components of precision machinery are not well-known. The licensed patents and previous literature that have already provided experimental data regarding the
mechanical properties of tool steels are very few. Therefore, it is benefit to the precision machinery industry to determine the actual benefits of cryogenic treatments, and verify the qualitative conclusions with experimental results. However, no previous research has provided relative experimental data to prove the advantages of cryogenic treatment, which has limited the applications of cryogenic treatment technology.

Much recent interest has focused on the effects of cryogenic treatment on the mechanical properties of SKD 11 and SKD 61 tool steels. However, little information exists concerning the effects of the sequence procedures of cryogenic treatment on SKH 51 high-speed steel. KelkarP [8] and HuangJ Y [9] have investigated the effects of cryogenic treatment on the strengthening mechanism and microstructural changes in M2 tool steels. However, further studies concerning the relationship between the mechanical properties and the phenomenon of secondary precipitation hardening are needed. Therefore, the present study examines the effects of cryogenic treatment on microstructural developments and the mechanical properties of SKH 51 high-speed steel by means of hardness testing, x-ray diffraction, scanning transmission electron microscopy and energy dispersive spectrometry.

2. Experimental procedures

JIS SKH 51 (corresponding to ASTM M2) high-speed steel was used as the raw material, the chemical composition of the steel is shown in table 1. The SKH 51 high-speed steel was preheated to 750℃ for 1 hour, and then solution heat-treated for 1 hour at 1190℃ before being rapidly quenched to room-temperature by liquid nitrogen gas. The tempering processes were carefully performed at temperatures ranging from 540℃ to 590℃. SKH 51 steel can achieve excellent surface hardness at 540℃, and displays excellent toughness at 590℃. Therefore, the tempering processes in the present study were performed at 540℃ and 590℃, respectively. The cryogenic treatment was performed at temperatures ranging from room temperature to -196℃, then cooled by liquid nitrogen at a rate of 30℃/hr. The samples were then kept at -196℃ for 8 hours.

Table 1. Chemical compositions of the studied JIS SKH 51 steel by GDS analysis (wt%).

|       | C   | Cr  | Mo  | W   | V   | Co  | Fe  |
|-------|-----|-----|-----|-----|-----|-----|-----|
| SKH 51| 0.86| 3.95| 4.75| 5.83| 1.82| 0.56| Bal.|

Specimens for transmission electron microscopy were prepared by using a double-jet electropolisher with an electrolyte consisting of 10% perchloric acid, 30% acetic acid and 60% ethanol. The polishing temperature ranged from -5℃ to -15℃, and the current density ranged from 3.5 to 4.0×10^4A/m^2. Electron microscopy was performed on a Philips FEG-20 scanning transmission electron microscope (STEM) operating at 200 kV.

3. Results and discussion

3.1. Effects of cryogenic treatment on the mechanical properties of SKH 51 high-speed steel

Figures 1(a) and 1(b) depict the hardness and wear mass loss properties of SKH 51 high-speed steel after being quenched, cryogenically treated and tempered at 540℃, respectively. The hardness of the quenched and then cryogenically treated SKH 51 high-speed steel was HRC66, which is similar to that of the quenched specimen that did not undergo cryogenic treatment. It indicates that the influence of cryogenic treatment on the as-quenched SKH 51 high-speed steel is not apparent in relation to surface hardness. However, the hardness of the SKH 51 high-speed steel after being quenched, cryogenically treated and then tempered at 540℃ is approximately HRC65, which is excellent compared to the specimen tempered at 540℃ without cryogenic treatment. Our experimental results indicates that the SKH 51 high-speed steel which was cryogenically before being tempered at 540℃ demonstrates excellent surface hardness and wear resistance. This necessitates the investigation of the microstructural changes in SKH 51 high-speed steel caused by cryogenic treatment described in the
following section.

**Figure 1.** Effects of cryogenic and tempering treatments at 540°C on the mechanical properties of the studied SKH 51 steel. (a) the surface hardness; (b) the wear resistance. (Q: quenched; C: cryogenic).

**Figure 2.** Effects of cryogenic and tempering treatments at 590°C on the mechanical properties of the studied SKH 51 steel. (a) the surface hardness; (b) the wear resistance.

Figures 2(a) and 2(b) demonstrate the surface hardness and wear mass loss properties of SKH 51 high-speed steel under as-quenched condition, after cryogenic treatment and after being tempered at 590°C, respectively. After being cryogenically treated and then tempered at 590°C, both the surface hardness and wear resistance of the SKH 51 high-speed steel are compromised. The surface hardness of SKH 51 high-speed steel after being quenched and then tempered at 590°C is HRC 60.5, which is relatively similar to that measured from the specimen that was cryogenically treated and then tempered at 590°C. The influence of cryogenic treatment was not obvious for SKH 51 high-speed steel after being tempered at 590°C. Therefore, to achieve better surface hardness and wear resistance at 590°C, the SKH 51 high-speed steel should be tempered at 590°C before undergoing cryogenic treatment. It was not necessary to perform the cryogenic treatment before the tempering processes. The above heat treatment procedure was also convenient and suitable for the industrial production process.

### 3.2 Effects of cryogenic treatment on the microstructure of SKH 51 high-speed steel

Figure 3(a) represents an optical micrograph of SKH 51 high-speed steel in the as-quenched and then cryogenically treated condition, clearly showing the finer prior austenite grains as well as particle-like carbides. The microstructure is similar to that observed in the as-quenched specimen without cryogenic treatment, as shown in figure 3(b). It was difficult to visually distinguish the martensite and retained austenite phases in the optical micrograph. Figures 4(a) and 4(b) show x-ray diffraction patterns of the SKH 51 high-speed steel under different heat treatment conditions. Figure 4(a) displays an x-ray
diffraction (XRD) profile of the as-quenched specimen without cryogenic treatment, demonstrating the presence of martensite, carbides and retained austenite. Figure 4(b) shows an XRD profile of the specimen after being quenched and then cryogenically treated, showing the presence of martensite and carbides. This result indicated that the amount of retained austenite decreased significantly with cryogenic treatment.

Figure 3. Optical micrographs of the SKH 51 high-speed steel. (a) solution-treated at 1190°C and then cryogenically treated; (b) solution-treated at 1190°C without cryogenic treatment.

Figure 4. XRD patterns of the SKH 51 steel under different heat treatment conditions. (a) The as-quenched SKH 51 steel without cryogenic treatment; (b) The as-quenched and then cryogenically treated steel. (M: martensite; RA: retained austenite).

Figure 5(a) shows a bright-field (BF) electron micrograph of the as-quenched SKH 51 steel, demonstrating the presence of dispersed coarse carbides within the martensite matrix. Figures 5(b) and 5(c) show two selected area diffraction patterns (SADPs) of the dispersed carbides. Diffraction analysis demonstrates that the carbides consisted primarily of MC and M6C carbides within the martensite matrix. Figures 5(d) and 5(e) show two EDS spectra of the particle-like M6C carbides and the surrounding martensite matrix, respectively, from the as-quenched SKH 51 high-speed steel. The primary differences between these two EDS spectra are the tungsten and molybdenum contents. It is clear that the particle-like carbides within the martensite matrix are tungsten-rich and molybdenum-rich M6C carbides, as confirmed by electron diffraction analysis. Figures 6(a) to 6(c) depict electron micrographs of the SKH 51 high-speed steel after being quenched and then cryogenically treated. Besides the particle-like carbides and the martensite matrix, the high density of plate-like twin faults and dislocations can be observed within the martensite matrix.

Figure 7(b) displays an optical micrograph of the SKH 51 high-speed steel being solution-treated, cryogenically treated, and then tempered at 540°C, clearly displaying the finer tempered martensitegrain as well as the presence of particle-like carbides. The microstructure was similar to that observed in the as-quenched specimen without cryogenic treatment, as shown in figure 7(a). However, the cryogenically-treated SKH 51 steel displays better surface hardness and wear resistance. Based on
Figure 5. TEM electron micrographs of the SKH 51 steel under as-quenched conditions. (a) BF electron micrograph showing the carbides dispersed within the martensite matrix. (b) and (c) show SADPs of the dispersed carbides in (a); (d) and (e) show EDS spectra obtained from the $M_6C$ carbide and its surrounding martensite matrix, respectively.

Figure 6. TEM electron micrographs of the SKH 51 high-speed steel after undergoing solution treatment at 1190°C and then cryogenic treatment. (a) BF electron micrograph showing the presence of carbides dispersed within tiny laths structure of the martensite matrix; (b) BF electron micrograph, showing twins and dislocations within the martensite matrix; (c) DF electron micrograph taken from the same martensite region shown in (b).
was also suitable for industrial production.

Figures 11(a) and 11(b) shows two x-ray diffractions of SKH 51 high-speed steel under different heat treatment conditions. Figure 11(a) shows an XRD profile taken from the as-quenched and tempered at 590°C specimen without cryogenic treatment, showing the presence of martensite and...
Figure 9. EDS spectra obtained from the SKH 51 steel which was cryogenically treated and then tempered at 540°C. (a) EDS spectrum taken from the particle-like MC carbide marked as "A" in figure 8(c); (b) EDS spectrum taken from the particle-like M2C carbide marked as "B" in figure 8(c).

Figure 10. TEM BF micrographs of the SKH 51 steel. (a) solution-treated and then tempered at 590°C without cryogenic treatment; (b) solution-treated, then cryogenically treated and finally tempered at 590°C.

Figure 11. XRD patterns of the SKH 51 steel under different heat treatment conditions. (a) solution treated and then tempered at 590°C without cryogenic treatment; (b) solution treated, then cryogenically treated and finally tempered at 590°C.

carbides. Figure 11(b) shows an XRD profile of the specimen after undergoing cryogenic treatment and then being tempered at 590°C, which is similar to that observed from the specimen which was quenched and then tempered at 590°C. However, the higher peak relating to MC carbides was derived from the cryogenically treated specimen. The amount of MC carbides was greater than that observed in SKH 51 steel that was only tempered at 590°C without cryogenic treatment. This result indicated that cryogenic treatment enhanced the secondary precipitation of tiny MC carbides during the tempering process, which was consistent with the visual observation of carbides as shown in figure 10(b).

Based on the above observations, the surface hardness of the as-quenched SKH 51 steel generally decreased during the tempering processes. However, the SKH 51 steel which was cryogenically
treated and then tempered at 540 °C demonstrating excellent surface hardness as well as wear resistance. It was observed that cryogenic treatment can reduce the amount of retained austenite, as well as induce a high density of dislocations and stacking faults within the martensite matrix. During the subsequent tempering at 540 °C, tiny secondary MC and M2C carbides precipitated near the existing dislocations and stacking faults, which resulted in the secondary precipitation hardening of the SKH 51 high-speed steel. When the tempering processes was performed at 590 °C, the secondary precipitation of MC carbides was observed. However, both the surface hardness and wear resistance of the SKH 51 high-speed steel became compromised. This result may have been caused by the promotion of rapid decomposition and growth of tempered martensite. Further investigation of the effects of cryogenic treatment on the toughness of SKH 51 high-speed steel is necessary to further understand the industrial applications of cryogenic treatment.

4. Conclusions
When the SKH 51 high-speed steel was cryogenically treated and then tempered at 540 °C, the surface hardness of the SKH 51 steel increased from HRC 64 to HRC 65. This result indicates the reduction of retained austenite as well as the secondary precipitation hardening of tiny carbides.

TEM analysis indicated that cryogenic treatment can induce high density dislocations and stacking faults within the martensite matrix after cryogenic treatment. During the subsequent tempering at 540 °C, tiny secondary carbides precipitated near the existing dislocations and stacking faults, which resulted in the secondary precipitation hardening of the SKH 51 high-speed steel. These secondary carbides consisted primarily of Vanadium-rich MC carbides as well as (Mo, W)-rich M2C carbides, as identified by STEM/EDS techniques.

After being cryogenically treated and then tempered at 590 °C, both the surface hardness and wear resistance of the SKH 51 high-speed steel became compromised. This result indicates the promotion of rapid decomposition and growth of tempered martensite. The influence of cryogenic treatment was not obvious for SKH 51 high-speed steel after being tempered at 590 °C.

Therefore, to achieve the best surface hardness, the SKH 51 high-speed steel should be cryogenically-treated first and then tempered at 540 °C.

References
[1] Barron R F 1982 Cryogenic treatment of metal to improve wear resistance Cryogenics 22 409-13
[2] de Silva F J, Franco S D, Machado A R, Ezugwu E O and Souza Jr A M 2006 Performance of cryogenically treated HSS tools Wear 261 674-85
[3] Das D, Dutta A K and Ray K K 2009 Correlation of microstructure with wear behavior of deep cryogenically treated AISI D2 steel Wear 267 1371-80
[4] Zhirafar S, Rezaein Aand Pugh M 2007 Effect of cryogenic treatment on the mechanical properties of 4340 steel J. Mater. Proc. Tech. 186 298-303
[5] Leskovsek V, Kalin Mand Vizintin J 2006 Influence of deep-cryogenic treatment on wear resistance of vacuum heat-treated HSS Vacuum 80 507-18
[6] Lal D M, Renganarayanan Sand Kalanidhi A 2001 Cryogenic treatment to argument wear resistance of tool and die steels Cryogenics 41 149-55
[7] Das D, Dutta A K, Toppo Vand Ray K K 2007 The effect of cryogenic treatment on the carbide precipitation and tribological behavior of D2 steel Materials and Manufacturing Processes 22 474-80
[8] Kelkar P, Nash Pand Zhu Y T 2003 The mechanism of property enhancement in M2 tool steel by cryogenic treatment the 45th MWSP Conference Proceedings Chicago, Illinois, November 9 - 12, 2003 pp 13-9
[9] Huang J Y, Zhu Y T, Liao X Z, Beyerlein I J, Bourke M A and Mitchell E 2003 Microstructure of cryogenic treated M2 tool steel Materials Science and Engineering A339 541-4