Effectiveness of Cryotreatment on Steel with low Potential for Carbide Precipitation and Austenite Retention

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Abstract

The improvement of hardness and wear resistance of tool steels due to deep cryogenic treatment (DCT) is attributed to the ability of the process to transform the retained austenite. Additional contribution could be due to stimulation of carbides precipitation and the generated dislocations. All above effects act simultaneously and their assessments in the DCT process is usually done collectively. It is aimed in this study to assess the significance of applying of DCT to a tool steel which has low potential for carbide precipitation and austenite retention over conventional hardening treatment (CT). For this purpose hot worked tool steel L6 was chosen. Structure with and without retained austenite is then obtained by varying the austenitization temperature. Samples to receive DCT were deep freeze for 10 hours before tempering. Results showed that DCT had improved the hardness and wear resistance even when the structure contain no retained austenite or carbide precipitation. The improvement is even better when transformation of retained austenite is involved. XRD profiles revealed broader peaks for DCT samples comparing with conventionally treated samples which attributed more probably to the refinement of the martensitic structure rather than to the increase in dislocation density.

Key words: *deep cryogenic treatment (DCT), hardness, wear resistance, retained austenite*

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1. INTRODUCTION

Deep cryogenic treatments is one type of sub-zero treatments including cold treatment and shallow cryogenic treatment. These treatments are classified according to the lowest temperature attained during the process. Despite of the documented benefits obtained by applying of DCT for tool steels, the application of the process on the industrial scale is still limited because of the wide variation in reported results and the recommended process parameters [1]. Application of DCT for tool steels improves the hardness and wear resistance. The improvement is attributed to three metallurgical changes: (1) the enhanced precipitation of fine carbides during tempering, (2) the transformation of the retained austenite [2] and (3) the differential contraction of the matrix relative to the retained austenite and the carbides cause the generation of dislocation [3]. Increasing dislocation density per se increases the hardness and strength of the steel beside its role as favorite sites for carbon clusters and carbide precipitation. All above effects act simultaneously to improve mechanical properties and their roles in the cryotreatment process is usually assessed collectively. In this study it is attempted to judge the roles of the second and third effects mentioned above on the hardness and wear resistance in isolation of the effect of stimulated carbide precipitation. Thus, DCT will be applied to a structure without retained austenite or carbide precipitation and then comparing the results when the process is applied to a structure contain retained austenite. The improvement in both cases will be compared with results obtained by conventional hardening heat treatment.

2. Methodology

For the purpose of this study, a hot worked L6 tool steel with low potential for carbide formation was chosen and austenitized at different temperatures to get structures with and without retained austenite. Both types of structures then subjected to conventional hardening treatment and deep cryogenic treatment. Hardness and wear tests were carried out on samples tempered at low and high temperatures. Test results should reveal any improvement obtained by the DCT over the conventional treatment. X-ray diffraction was conducted to detect any profile peak broadening due to increased dislocation caused by the DCT compared with conventionally treated samples. Peak broadening comprise also contribution from instrument, but this effect can be disregarded because all profiles were obtained using the same diffractometer under the same testing condition from the same material. Therefore any relative increase in peak widths can be related either to increased dislocations or structure refinement induced by the DCT.

3. Experimental Work

The Chemical analysis for samples of the hot forged tool steel L6 was carried out using spark emission spectrometer (model JY 132 F). The composition is: 0.55 %C, 1.7 %Ni, 0.7%Cr, 0.75%Mn, 0.25%Si and balance Fe.

3.1 Hardening heat treatment

Eight groups of samples were hardened as indicated in table 1. Austenitization was carried out under nitrogen protective atmosphere then directly quenching in oil. Cryogenic treatment was carried out by deep freeze in liquid nitrogen vapor for 10 hours. Samples were placed in a basket in isolated chamber and liquid nitrogen was allowed to flow through a controlling valve down ward by gravity from a storage cylinder situated above the chamber. Electric fan is placed inside the chamber to get good vapor circulating and outlet the excess vapor. Tempering was carried out for one hour for the indicated temperatures.

Table 1: Conventional and Deep cryogenic hardening heat treatments details

Heat treatment type	CT				DCT			
Austenitization temperature °C	855		880		855		880	
Deep freeze	NA		NA		10 hrs in liquid nitrogen vapor			
Tempering temperature °C	200	550	200	550	200	550	200	550

3.2 X-ray diffraction

X ray diffraction was carried out using PHILPS diffractometer type PW1800 with Cu anode, Kα2 to Kα1 ratio 0.5 over the range 2.0 to 99.9, step size 0.02 and scan step time 5 sec. The diffractometer equipped with X'Pert software for peaks search. Disc shaped samples with 20 mm diameter and 5 mm thickness were prepared for XRD and hardness test measure

3.3 Hardness test

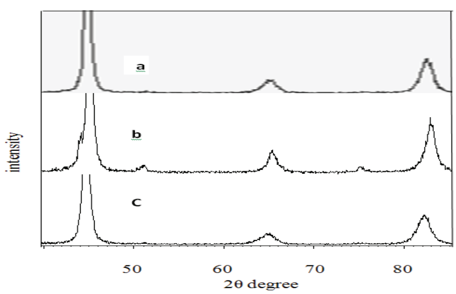
The hardness tester machine (BULUT- BMS 201-R) was used where the standard penetration was obtained with diamond cone indenter on the Rockwell C scale and the applied major load was 150 kg. Hardness measurements were taken before and after tempering and each hardness value is an average of five reading.

3.4 Sliding Wear test

The wear testing rig (Make Amsler type A-135) with a Block on Ring configuration was used. In this test the sample (the ring) is rotating against the stationary block under a specified load. The test was conducted according to ASTM G77 with applied load 100 N and 300 rpm. The test was divided into four equal intervals with a total sliding distance of 3960 m. Test samples were carefully cleaned and dried before each stage during testing and the weight was measured using a precise digital scale (KERN 572-37).

4. Results

Figure 1 (a, b and c) shows XRD profiles for un-tempered samples: a; austenitized at 855°C, b and c at 880°C before and after deep freeze respectively. The low austenitized temperature produced a complete martensitic structure while austenitization at 880°C produced structure with about 7% retained austenite as calculated by direct comparison method. The retained austenite is almost completely transformed by the deep freeze process as shown in profile in figure 1 c where the peaks correspond to the austenite phase disappeared. XRD profiles after tempering are shown in figures 2 and 3 for samples hardened at 855°C and 880°C respectively. The profiles reveal that there is no carbide formed of any type in this steel by both austenitizing temperatures. The letters C and DC denote for conventional hardening and deep cryogenic treatment respectively and the numbers 200 and 550 are the tempering temperatures



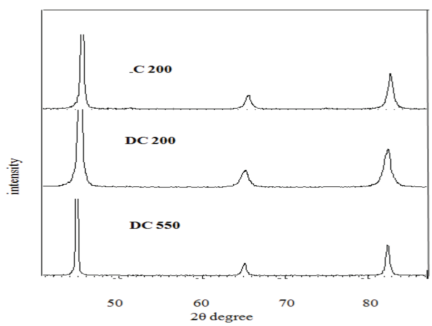


Figure 1 XRD profiles un-tempered condition a) austenitized at 855°C, b &c) austenitized at 880°C before and after deep freeze

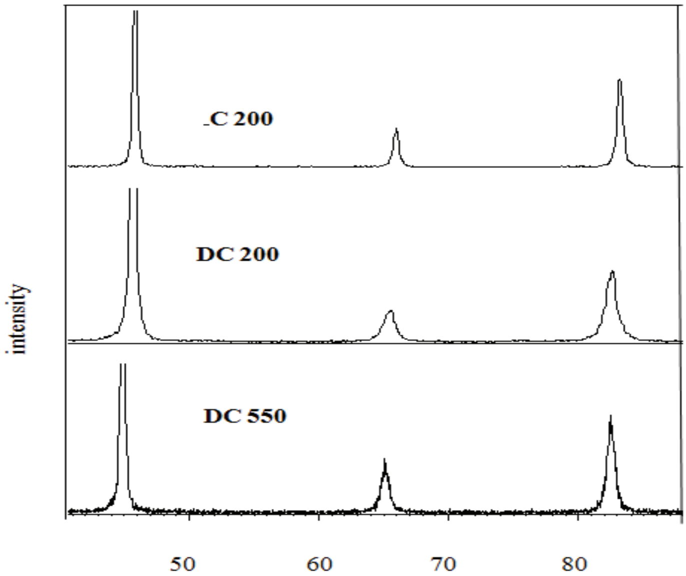


Figure 2 XRD profiles for samples austenitized at 855°C

Results of Hardness test using Rockwell C scale are presented in table 2 for both austenitization temperatures.

Table 2: Hardness (HRC) for various heat treatments

Tempering temperature. °C	Austenitzation temp.855 °C		Austenitzation temp.880 °C	
	CT	DCT	CT	DCT
0*	56	56	56	59
200	53	55	53	57
500	38	38	38.8	40
*** 0 un-tempered samples in conventional treatment or as Deep freeze and un-tempered for DCT**				

For un-tempered samples cryogenic treatment caused increase in hardness for samples austenitized at 880°C whereas samples austenitized at 855°C, the hardness remain unchanged after the cryogenic treatment. For samples tempered at 200°C, Cryogenic treatment caused increase in hardness compared with conventional treatment and the gain in hardness was higher for samples austenitized at 880°C (from 53 to 57 HRC) compared with samples austenitized at 855°C (from 53 to 55 HRC). High tempering temperature caused substantial decrease in hardness in all samples due to the weak resistance to softening of this type of steel due to the low content of the alloying elements.

Wear test results are shown in figures 4 and 5. For samples hardened at 855 °C and 880 °C respectively. All samples were tested against a block with hardness of 59 HRC obtained by

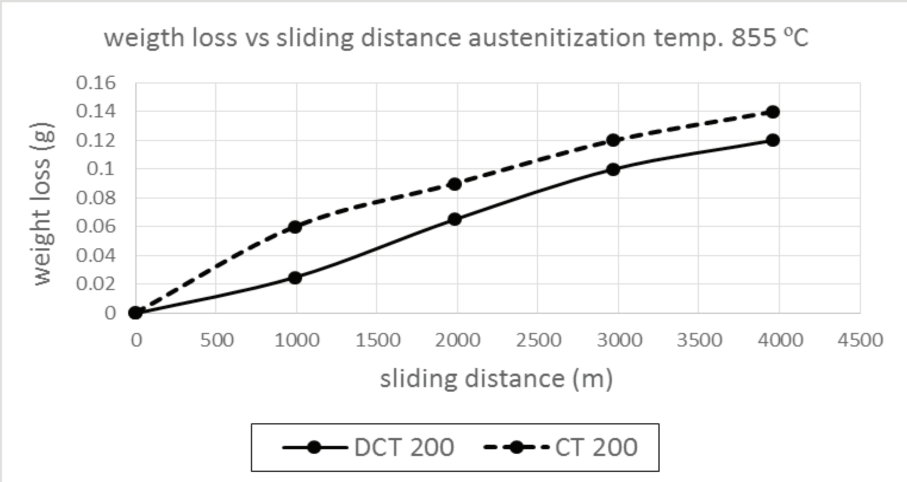


Figure 4 wear test results for samples hardened at 855°C

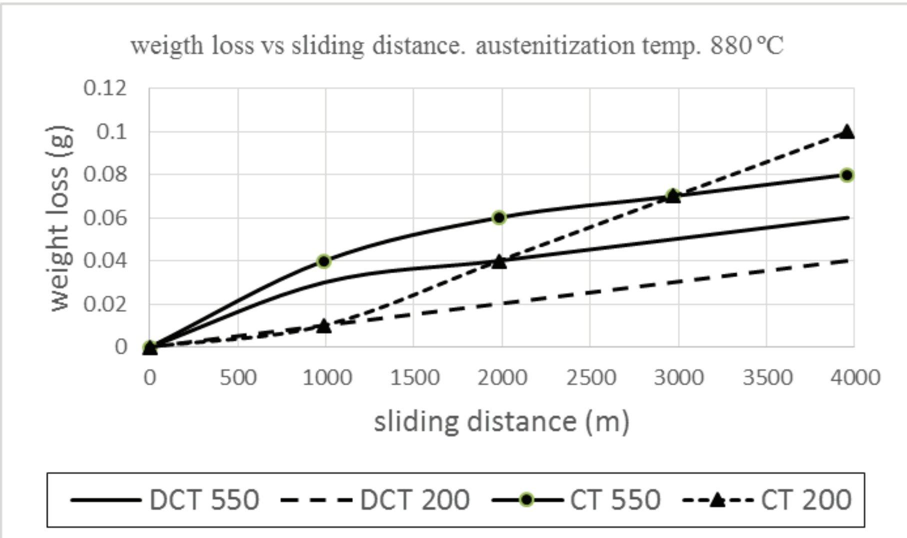


Figure 5 wear test results for samples hardened at 880°C austenitizing at 880 °C then deep freeze for 10 hours but un-tempered. Cryogenic treatment improved the wear resistance for both austenitization temperatures with the samples austenitized at the high temperature showed even better wear resistance.

The specific wear rate (SWR) known as k was calculated according to Archard equation

SN=V/k

Where S is the sliding distance (m) N is the applied load (N) and V is the worn volume obtained by converting the weight loss. The specific wear rate (k) in (m^2/N) is calculated by plotting the wear volume versus the product SN and the slope of the line is the value of K.

The SWR for DCT and CT samples hardened at 880 °C and tempered at 200 °C were 1.3 $\times 10^{-14}$ m^2/N and 3.9×10^{-14} m^2/N respectively. This result corresponds to an improve in wear resistance of deep cryogenically treated samples over conventionally treated samples by 66%

5. Discussion

X-ray diffraction was conducted to detect phases and their relative amounts and to monitor changes in profile line shape which reflect changes induced on the microstructure by the cryogenic treatment.

The high austenitization temperature produced structure with retained austenite due to the more enrichment of the austenite with carbon during austenitization which increased the stability of the austenite phase and lowers the Ms temperature.

Values of Peaks width (FWHM) from profiles in figures 2 and 3 for the corresponding diffracting planes -as measured by the built in software- are indicated in tables 3. From this table, comparing peaks widths for samples tempered at 200°C clearly showed that cryotreament caused increase in peaks width compared with conventionally treated samples. Peak broadening can be caused-in currently investigated samples- by structure refining or due to increased dislocation density.

Table 3: Values of (FWHM) for samples hardened at 855°C and 880°C

	samples hardened at 855°C				samples hardened at 880°C			
Diffraction. plane	as-quench	CT 200	DCT 200	DCT 550	as quench & deep freeze	CT 200	DCT 200	DCT 550
110	0.512	0.197	0.256	0.256	0.708	0.295	0.295	0.157
200	0.629	0.236	0.629	0.472	0.523	0.177	0.63	0.276
211	0.576	0.240	0.432	0.264	0.768	0.336	0.384	0.354

A number of researchers found that cryogenic treatment cause structure refinement. Ning Xu et.al in their investigation on low carbon steel found that the martensite phase becomes more homogenous by cryogenic treatment [4]. Leskovsek et.al. and Jelenkowski et. al. claimed that deep cryotreatment resulted in finer needlelike martensitic [5,6].while other researchers detect increase in dislocation density due to cryotreatment. Kelkar et al in their study on effect of cryotreatment on M2 tool steel proved increase in dislocations due to cryotreatment [3]. On the other hand others consider increased dislocation density in martensitic structure is unlikely. Dong Yun et al reported that the defect density in martensite is generally so high that it is practically difficult to directly observe whether more dislocations or twins are generated by the cryogenic treatment process [7]. The explanation set by those who found increase in dislocations is that differential contraction between the matrix and the retained austenite cause the generation of dislocation [3]. Therefore, the peak broadening detected in this work is more likely to be attributed to the refining of the martensite when the structure contain no retained austenite while for structure contain retained austenite, the peak broadening could be attributed to both structure refinement and increased dislocation density.

The effect of transformation of retained austenite on peak width can be noticed by comparing values of peak widths of as quenched (un-tempered) samples from both austenitization heat treatment to obtain complete martensitic structure. Samples austenitized at 880°C contain about 7 % retained austenite, after deep freeze the peaks were relatively boarder than that of as quenched samples austenitized at 855°C which originally do not contain retained austenite.

It is also noted that peak width does not increase linearly with 2θ this is because distribution of dislocation also affect the peak width not only its density [8].

The preceding explanation on microstructural changes assist in interpreting the resultant increase in hardness and wear resistance obtained by the DCT. For as-quenched samples there is no increase in hardness by deep freeze when the structure is originally fully martensitic despite the thought of the structure refinement but when transformation of retained austenite is involved there is an increase in hardness by 3.0 HRC. Tempering at 200°C result in decrease in hardness compared with un-tempered condition. However the decrease is more evident for conventionally treated samples compared with cryotreated samples. The decrease in hardness is due to recovery and re-arrangement of dislocations and this effect is extended at higher tempering temperature to causes further decrease in hardness combined with the low resistance to softening due to the low content of alloying elements in this alloy.

Results of wear tests showed increase in wear resistance obtained by applying of DCT over conventional treatments for structures with or without retained austenite. Higher wear resistance is obtained when there is increase in hardness and toughness as well. Therefore the suggestion of structure refinement induced by the DCT will support the wear resistance improvement because structure refinement will increase the toughness. Cryotreated samples austenitized at 880°C showed better wear resistance over samples austenitized at 855°C because of the higher hardness. Figure 5 showed decrease in wear resistance of samples tempered at 550°C even for DCT samples, clearly this is due to the decline in hardness.

6. Conclusions

The achieved improvement in hardness and toughness obtained by cryotreatment is to be more likely due the refinement of the martensitic structure rather than the increased in dislocation density. Accepting the first mechanism is supported by the increase in wear resistance which require increase in hardness and toughness as well, but increase in dislocation density, although it causes increase in hardness it lowers the toughness. However structure refinement and increased dislocations both manifested in broadening of XRD profiles, so more investigations should be undertaken to firmly resolve the more likely mechanism. Additional increase in hardness and wear resistance is obtained by conversion of retained austenite by applying of DCT.

References

[1] Patil P. I., Tated R. G., International Conference in Computational Intelligence (ICCIA) Proceedings published in International Journal of Computer Applications (IJCA) (2012) 10-29

[2] Das D., Sarkar R., Dutta A.K., Ray K.K., Mat Sci Eng A-Struct., 528 (2010) 589–603-doi:10.1016/j.msea.2010.09.057

[3] Kelkar R., Nash P., Zhu Y., Heat Treating Progress, 8 (4) (2007) 57-60
[4] Xu N., Cavallaro G.P., Gerson A.R., J. Mater. Res., 27 (16) (2012) 2122-2130, doi: 10.1557/jmr.2012.135
[5] Leskovsek P. V., Vizintin J., Mater Manuf Process, 24 (2009) 734–738
[6] Jeleńkowski J., Ciski A., Babul T., Journal of Achievements in Materials and Manufacturing Engineering, 43 (1) (2010) 80-87
[7] Yun D., Xiaoping L, Hongshen X., Heat Treatment of Metals, .3 (1998) .55-59
[8]. Bor T.C, Cleveringa H.H.M., Delhez R., Van der Giessen E., Mat Sci Eng A-Struct., 309–310 (2001) 505–509