Improving the Wear Resistance of D2 Tool Steel by Deep Cryogenic Treatment and Secondary Hardening

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Abstract

The wear resistance of D2 tool steel was investigated for specimens that have been subjected to conventional hardening heat treatment and Deep Cryogenic Treatment. For both treatments, tempering was carried out at temperatures 200 and 550 ºC which corresponds to the secondary hardening temperature. The increase in wear resistance due to cryogenic treatment with tempering 200 ºC and secondary hardening relative to specimens tempered at 200 ºC in conventional hardening was 1.42 and 1.25 respectively. The improvement is due to the increased secondary carbide precipitates and the transformation of retained austenite. The higher improvement achieved by cryogenic treatment is attributed to the more uniform distribution of the secondary carbide precipitates with finer size and closer interparticle spacing than in secondary hardening. Under the applied test conditions, the prevailing wear mode anticipated to be mild for all tested specimens according to the calculated wear coefficients.

Key words: secondary hardening, deep cryogenic treatment, retained austenite, wear

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1. Introduction

High carbon high chromium tool steels such as D2 are selected mainly for their high wear resistance. Higher wear resistance requires high hardness and resistance to fracture. Hardness is affected by the austenitization temperature and tempering temperature. A higher austenitization temperature increases the amount of carbide dissolution and the austenite is enriched with more carbon to produce harder martensite but the amount of retained austenite also increases which adversely affect the hardness (Jurcip 2010 pp18-20).

The as quenched parts are very brittle. For practical applications, tempering treatment is carried out to obtain a combination of strength and toughness to improve mechanical properties such as the wear resistance. In plain carbon steels, increasing the tempering temperature progressively decreases the hardness. A high wear resistance requires high hardness thus, tempering at high temperature to gain more toughness will deteriorate the wear resistance.

In alloy steels, hardness deterioration also occurs by tempering above 200 ºC but recovery in hardness starts to take place above 500 ºC

When the steel contains carbide forming elements, metal carbides are precipitated during tempering at high temperature. These carbides are more stable than iron carbide but due to the lower diffusivity of substitutional atoms compared with carbon at low temperatures, metal carbides can only form when the tempering temperature exceed 500 ºC. Formation of metal carbides in tempering is known as secondary hardening and hardness recovery occurs. In D2 tool steel secondary hardening takes place around 550ºC by precipitation of metal and iron carbides of different types. The structure of a hardened and tempered alloy steel thus contains fine carbides or secondary carbides that precipitated during tempering and coarser or primary carbides which are the undissolved carbides. Increased carbide content improves the

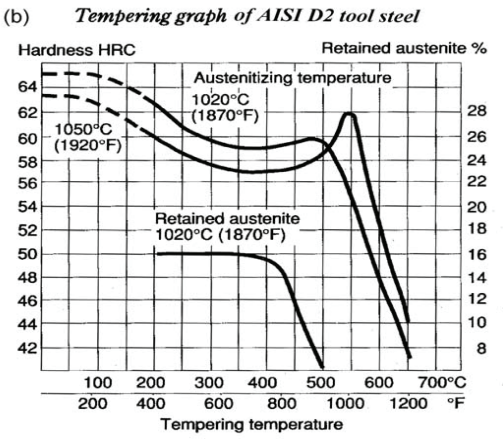


Figure 1 Variation of hardness and retained austenite content with tempering temperature in hardened D2 tool steel. Temperature correspond to secondary hardening depends on austenitizing temperature. (After Bourithis)

hardness and wear resistance properties. Figure 1 shows variation of hardness and retained austenite content with tempering temperature for D2 tool steel for two austenitization temperatures in conventional hardening heat treatment. The figure shows that secondary hardening occurs at about 550 ºC when the austenitization temperature is 1050 ºC (Bourithis 2006 pp479-489).

Deep cryogenic treatment is one of sub-zero treatments, namely: cold treatment, shallow cryogenic treatment and deep cryogenic treatment. The classification is based on the lowest temperature attained in the treatment (Das D. Sarkar 2010 pp 479-489). In Deep Cryogenic Treatment (DCT), hardened parts receive cooling in liquid nitrogen at a temperature of -169 ºC for several hours after quenching and –usually- before the tempering treatment. Deep cryogenic treatment promotes carbide precipitation during the subsequent tempering. Cryotreatment is also a practice treatment for eliminating or reducing retained austenite usually applied for dimensional stability. Therefore both secondary hardening and DCT can improve mechanical properties such as hardness and wear resistance. This study is an attempt to estimate the degree of improvement in wear resistance in D2 tool steel due to

i. The secondary hardening in conventional hardening heat treatment relative to the low tempering temperature.

ii. The application of DCT relative to the conventional hardening treatment

iii. Tempering to secondary hardening after DCT

All specimens will be tested for wear resistance at room temperature under the same conditions of applied load; sliding speed and sliding distances. Thus the obtained results can be interpreted with relation to microstructural changes that may be induced by the different heat treatments.

2. Experimental Work

Chemical analysis of the as received material was conducted using spark emission spectrometer (model JY 132 F). The composition in wt % is given in table 1

Table 1 Chemical composition of the D2 tool steel

element	C	Si	Mn	Cr	Mo	Cu	V	S	P	Fe
%wt	1.33	0.25	0.32	11.4	0.69	0.14	0.95	0.0037	0.01	balance

1.1 Material and heat treatment

Test samples were prepared to conduct charpy impact test. Disc shaped samples with 20 mm diameter and 5 mm thickness were prepared for hard-

Table 2 Conventional (CT) and deep cryogenic (DCT) hardening heat treatments details

Heat treatment type	CT		DCT	
Austenitizing temp. °C	1050		1050	
Deep freezing	NA		10 hrs in liquid nitrogen vapor	
Tempering temp. °C	200	550	200	550
Specimen labeled	CT200	CT550	DCT200	DCT550

Figure 2 Set up for deep cryogenic treatment

ness, XRD and optical and scanning electron microscopy. For wear test, the test specimens were prepared in the form of rings with inner and outer diameters of 16 mm and 30 mm respectively and width of 10 mm. Counter blocks were machined to conform to recess of the ring specimens.

The hardening heat treatments were carried out according to heat treatment regimes in table2. Austenitization was carried out at 1050 ºC under nitrogen protective atmosphere then directly quenching in oil. A high austenitization temperature was applied in order to obtain higher hardness as explained earlier. Cryogenic treatment was first attempted by direct immersion in liquid nitrogen for 24 hours but this technique was found in-effective and no increase in hardness was recorded so a set up was designed and built as shown in figure 2 to allow liquid nitrogen vapor to cool the specimens. The test specimens were placed in a basket in the insulated chamber and the liquid nitrogen was allowed to flow through a controlling valve down ward by gravity from a storage cylinder situated above the chamber as shown in figure 2. Electric fan is placed inside the chamber to attain a good vapor circulation and to outlet the excess vapor. Test specimens were deep freezed continuously for 10 hours. Tempering was carried out for one hour for the indicated temperatures. The four hardening treatments will be labeled as CT200, CT550, DCT200 and DCT550 according to the heat treatment type and tempering temperature.

2.2. *Hardness and Impact tests*

Hardness measurements were carried out on Rockwell C scale using hardness tester machine (BULUT- BMS 201-R) with applied major load of 150

kg. The average of five measurements was taken as the hardness value. The impact test was carried out on charpy samples and two samples were tested for each heat treatment. Hardness and impact tests were conducted for as quenched, as quenched and deep freezing and after tempering treatments.

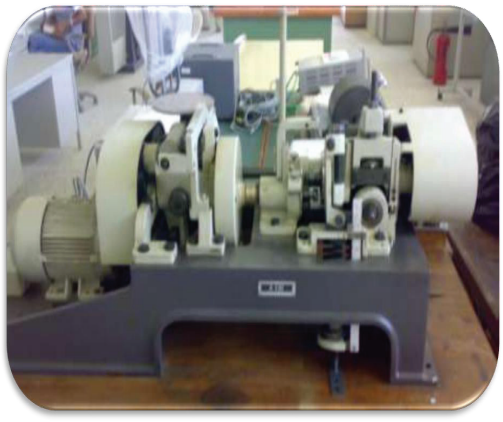
2.3. *Sliding Wear test*

The wear testing rig (Amsler type A-135) with a Block on Ring configuration was used. In this test the specimen (the ring) is rotating against the stationary block under a specified load. The test was conducted according to ASTM G77 with applied load 1000 N and 300 rpm (sliding speed 0.94 m/s). The test was divided into four equal intervals with a total sliding distance of 3960 m.

All test specimens were tested against block pieces of D2 tool steel hardened to 66 HRC in the as deep freeze and un-tempered condition. Test specimens were carefully cleaned and dried before each stage during testing and the specimen weight was measured using precise digital scale (KERN 572-37).The test rig and configuration of the rotating test sample with the stationary block are shown in figure 3

2.4. 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 is equipped with X'Pert software for peaks search.



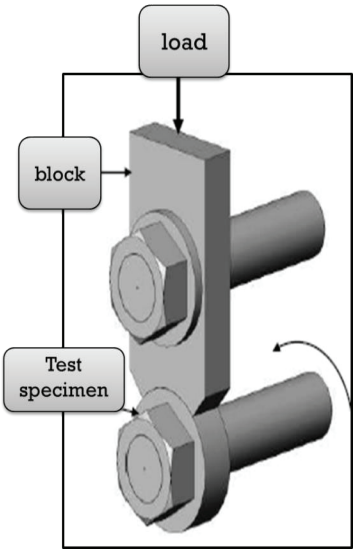


Figure 3 The wear testing rig (above) and configuration of test sample with the stationary block (right)

2.5. Optical and scanning electron microscopy

Specimens were prepared for optical and scanning electron microscopy and etched with 2% nital

3. Results

3.1 Hardness and impact test

The hardness of the as quench samples was 64 HRC and after deep freezing the hardness increased to 66 HRC. This hardness increment was accompanied with a decrease in toughness as measured by the fracture absorbed energy in impact test from 14.5 to 13.8 joule. The effect of tempering on hardness and absorbed energy are shown in figures 4 & 5 respectively

3.2 Wear test results

Wear test results are presented in figure 6 in terms of weight loss in (mg) versus the sliding distance in (m). For conventional hardening treatment, samples tempered at the secondary hardening temperature showed an improved resistance in wear over specimens tempered at 200 ºC though samples tempered at 200 ºC displayed a lower weight loss in the early part of the test or the running-in stage of the test. For samples received deep cryogenic treatment, samples tempered at 200 ºC showed better wear resistance than samples tempered at 550 ºC. The noticeable point here is that the wear resistance for specimens tempered at 550 ºC in conventional hardening or DCT were almost the same.

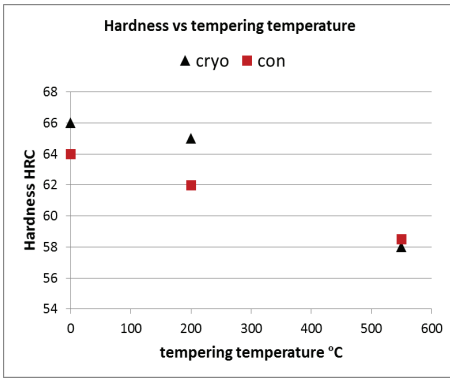


Figure 4 Variation of hardness with tempering temperature

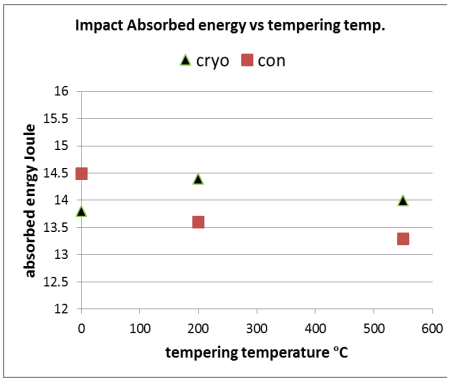


Figure 5 Variation of impact absorbed energy with tempering temperature

The test results may be presented in worn volume by converting weight to volume. For better comparison of these results, the data will be presented in terms of the wear rate (WR) in (m^3/m) and the specific wear rate (SWR) or (k) calculated using the Archard equation

$$SN = V/K$$

Where S is the sliding distance (m), N is the applied load (N) and V is the worn volume (m^3) obtained by converting the weight loss to volume. The specific wear rate (k) in (m^2/N) is calculated by plotting the worn volume versus the product SN and the slope of the line is the value of k. Figure 7 shows the method of calculation as applied to CT200 and DCT200

specimens. The improvement in wear resistance obtained by DCT and secondary hardening is assessed relative to the specimen conventionally hardened and tempered at 200 ºC (specimen CT200)

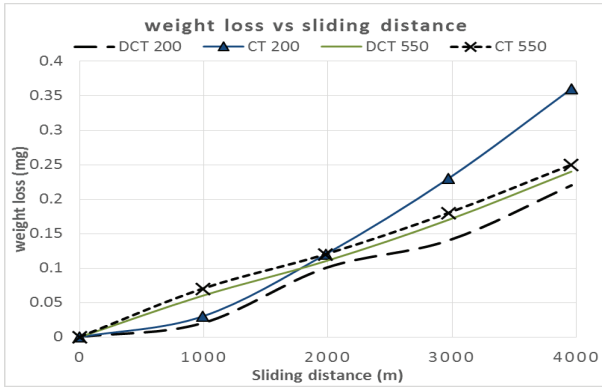


Figure6 Wear test results for all test specimens in terms of weight (mg) loss versus the sliding distance (m)

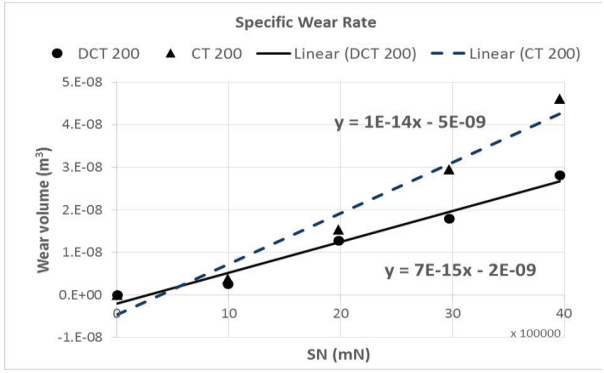


Figure.7 Shows calculating of the specific wear rate

Table 3 Wear test results

Heat treatment	Wear rate W_R (m^3/m)	SWR (m^2/N)	β	Wear coeff. (K)
CT 200	1.0×10^{-11}	1.0×10^{-14}	----	7.5×10^{-5}
CT 550	8.0×10^{-12}	8.0×10^{-15}	1.25	4.8×10^{-5}
DCT 200	7.0×10^{-12}	7.0×10^{-15}	1.43	5.8×10^{-5}
DCT 550	8.0×10^{-12}	8.0×10^{-15}	1.25	5.2×10^{-5}

by taken the ratios (β) of the wear rates as a ameasure of the improvement. For example for the specimen received deep cryogenic treatment and 200 ºC tempering (specimen DCT200) the improvement is calculated as

(W_R of CT200/W_R DCT200).

To compare the wear severity for tested specimens the wear coefficient (K) is calculated according to the following equation

$$K = \frac{W_R}{N} \times H_v$$

Where W_R is the wear rate in (m^3/m), N is the applied load in (N) and H_v is the hardness in (N/m^2) (Das D. ,Dutta 2009 pp1249-1257).

1.3 X ray diffraction

XRD profiles (intensity vs 2θ) for the four specimens are shown in figures 8 and 9. The profile for the CT200 specimen revealed retained austenite phase in the structure (peaks marked with γ in the profile). The amount of retained austenite was about 12 % (calculated by direct comparison method) which almost completely had transformed by the cryogenic treatment for 10 hours while tempering at 550 ºC had completely eliminate any retained

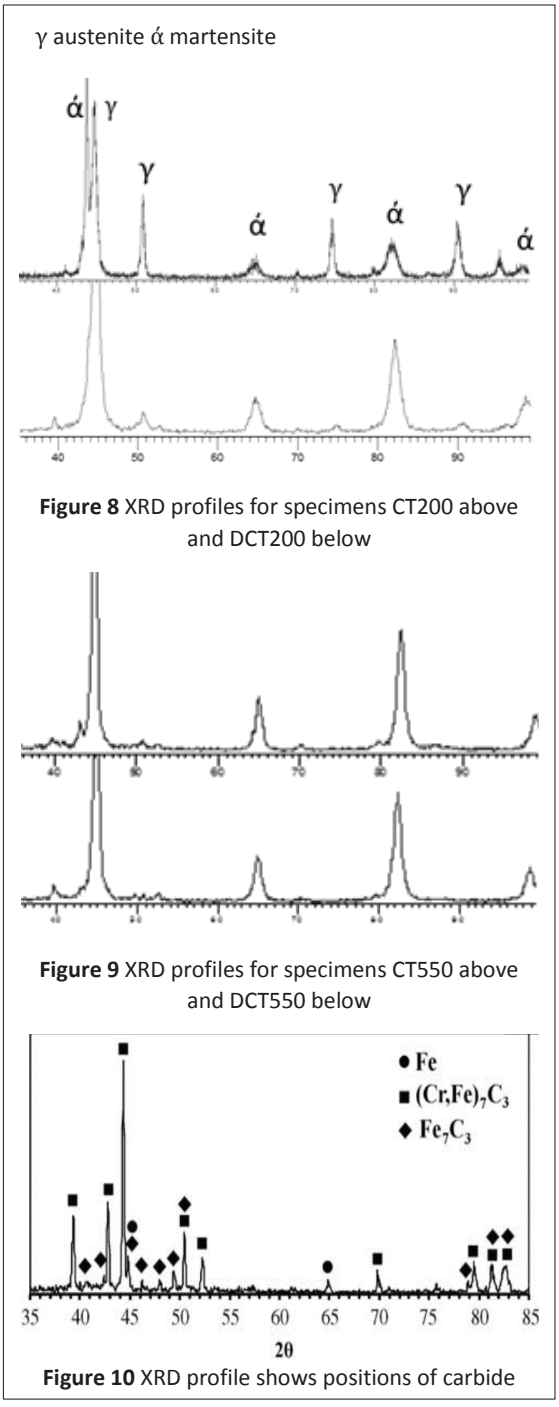


Figure 8 XRD profiles for specimens CT200 above and DCT200 below

Figure 9 XRD profiles for specimens CT550 above and DCT550 below

Figure 10 XRD profile shows positions of carbide

austenite. Both the cryogenic treatment and secondary hardening resulted in precipitation of carbides as can be noticed by intensification of peaks correspond to carbide phases such as peaks at (2θ) 39º or 70º for example. The comparison is related to figure 10 which shows the positions of carbide peaks when using an X ray with Cu Kα radiation.

1.4. Metallographic examination

Figures 11 and 12 show optical micrographs for the four specimens. The micrographs show the presence of primary and secondary carbides with different sizes in all samples. The remarkable observation is the enormous fine carbide precipitates in specimen DCT200 compared with other specimens. Carbides larger than 5 μm are classified as primary (coarse) carbides which are undissolved carbides or as secondary (fine) if their size is less than 5 μm which are precipitated during tempering. The magnification obtainable by optical microscopy is not sufficient to precisely

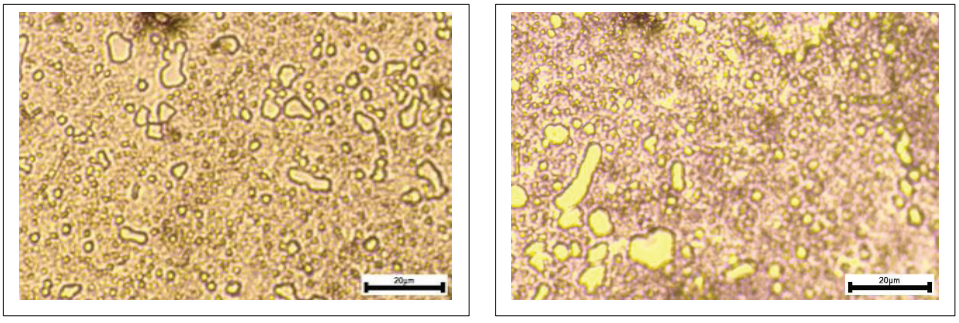


Figure11 Microstructure of conventionally treated (left) and cryogenically treated specimens (right) tempering temperature 200 ºC

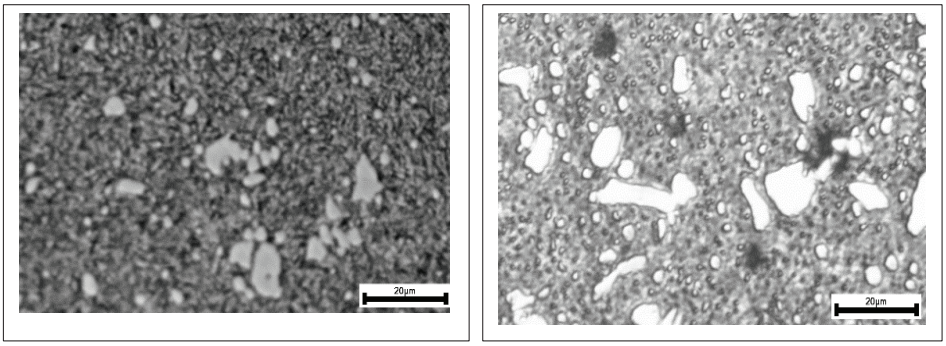


Figure12 Microstructure of conventionally treated (left) and cryogenically treated specimens (right) tempering temperature 550 ºC

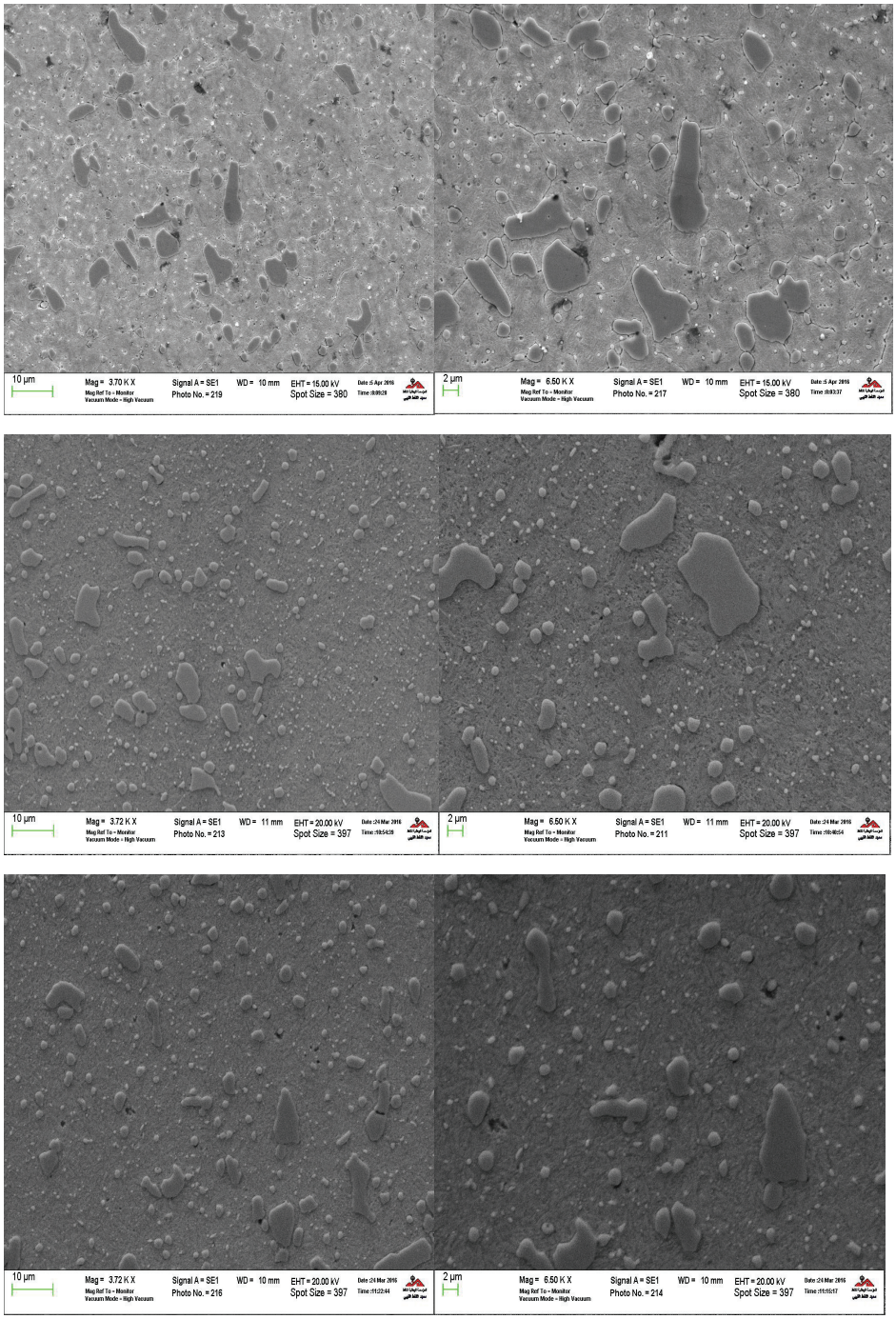


Figure 13 SEM micrographs for specimens CT200 (upper), DCT200 (middle) and CT550 (lower) micrographs on the right column at higher magnification

resolve the carbide size of the fine carbides. SEM micrographs for CT200, DCT200 and CT550 at two levels of magnifications are shown in figures 13. The high magnification in SEM micrographs revealed a substantial amount of very fine carbide precipitates in DCT200 specimen whereas secondary carbides in CT550 specimen are coarser in size and less in number than in specimen DCT200.

4. Discussion

Results of the wear test are presented in table 3 for all test specimens. The amount of improvement in wear resistance as indicated by the ratio (β) showed that cryogenic treatment resulted in a higher increase in wear resistance than obtained by secondary hardening. The values of wear coefficient for all test specimens were in the same order of magnitude which mean that all specimens undergone the same wear mode. One way to determine the wear mode is by calculating the wear coefficient. The values obtained in the current tests indicate that the wear mode is mild oxidative for all test specimens according to the wear coefficient values. (Das D. ,Dutta 2009 pp1249-1257). Investigation carried out by Das *et al* found that for deep cryogenically treated specimens the wear mode changed from severe to mild and the mechanism from delamination to oxidative when the sliding speed is decreased from 1.5 m/s to 1 m/s (Das D. Sarkar 2010 pp479-489) In the current experiments the sliding speed was about 1 m/s which confirm the anticipation for the wear mode. The importance of determining the wear mode in this investigation is that if the wear mode is not severe then the microstructural parameters will determine the wear performance and not the high temperature characteristics of the material (Fontalvo 2006 pp 1028-1034). If different wear modes were operating for different tests then effect of test conditions in addition to the metallurgical factors should be considered to assess the results.

XRD profiles showed that cryogenic treatment and secondary hardening had produced similar microstructural changes which are transformation of retained austenite and increased carbide precipitation. These two metallurgical changes are responsible for the increase in the wear resistance. Many authors confirm the effect of reducing the retained austenite content on improving the wear resistance (Das D. Ray 2009 pp1361-1370). With more carbides precipitated out of the martensitic structure it becomes more stress relived by rejecting more carbon atoms. Thus the internal tension of the martensite matrix is progressively reduced with increasing precipitation of carbides and the microcracks susceptibility is reduced (Paulin 1993 pp 26-28). This ef-

fect may explain the increased resistance to delamination for cryogenically treated specimens.

Optical microscopy revealed the increased density of secondary carbides precipitated due to secondary hardening and cryogenic treatment. The remarkable difference produced by the cryogenic treatment over secondary hardening can be observed by comparing distribution of secondary carbide precipitates as seen in the SEM micrographs.

Metal carbides such as $(Fe,Cr)_7Cr_3$ or $Cr_{23}C_7$ are more stable than cementite but due to the low diffusivity of substitutional elements at low temperatures, metal carbides are formed at high tempering temperature when the diffusivity of substitutional elements increases. Irrespective of the type of the carbide formed the wear resistance depends on the size of carbides and distribution rather than the carbide type (Homark 1975 pp39-61). As the amount of carbides increased the interparticle spacing decreases which improves the adhesive wear resistance of the tool steel. Fontalvo *et al* found that increasing carbide content in tool steel increases the adhesive wear resistance providing the inter-particle spacing become closer (Fontalvo 2006 pp 1028-1034). In other words finer carbides are more efficient to improve the wear resistance. On this basis, because cryogenic treatment produced ultrafine precipitation less than 1 μm in size with very small interparticle spacing as shown in figure 13 in DCT200 specimen their effect to improve the wear resistance will be more pronounced than carbides produced by secondary hardening treatment which are larger in size and more widely spaced.

The mechanism of carbide precipitation due to cryogenic treatment is explained by many authors to be due to the increased dislocation density by the deep cooling (Kelkar 2007 pp 57-60). Other researchers 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 during the cryogenic treatment process (Yun D. 1998 pp55-59).

Kelkar *et al* attributed the increased dislocation density to two factors; i) the differential contraction of the retained austenite phase (before transformation) and ii) the 4% volume expansion from austenite transformation to martensite. Diffusion of carbon atoms to dislocations reduces the overall energy of the system, and can occur to a limited extent at such a low temperature. However, significant segregation of carbon to dislocations occurs during the subsequent tempering and the produced precipitates are homogeneous and

very fine (Kelkar 2007 pp 57-60).

Tempering to secondary hardening in conventional hardening and after deep cryogenic treatment resulted in almost the same wear resistance with slightly less weight loss for cryogenically treated specimen. The similarity in wear behavior is obviously due to the similarity in the microstructures and carbide distribution produced by the two routes of hardening. Therefore cryogenic treatment must be followed by a low tempering temperature in order to obtain maximum improvement in wear resistance.

5. Conclusions

Both cryogenic treatment and secondary hardening produced similar microstructural changes namely: conversion of retained austenite and precipitation of secondary carbides. These two microstructural changes are the reason for the increase in wear resistance in both hardening treatments. The superiority achieved by cryogenic treatment over secondary hardening is attributed to the distribution of the carbide precipitation. In cryogenically treated specimen carbide precipitates were finer in size with closer interparticle spacing.

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تحسين خواص البلى لصلب العدة D2 بإستخدام التبريد العميق والتصليد الثانوي

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الملخص

في هذا ألبحث تمت دراسة مقاومة البلى لصلب العدة (D2 tool steel) لعينات خضعت لمعالجة تصليد تقليديه وأخرى للمعالجه بالتبريد ألعميق. لكلا المعالجتين أجريت عمليات ألمراجعه عند درجة حرارة 200 °م.و درجة حرارة التصليد الثانوي أي 550 °م. بينت ألنتائج تحسن مقاومة البلى بعد عملية التبريد العميق مع المراجعه عند 200 °م وعملية التصليد الثانوي بمقدار1.42 و 1.25على التوالي مقارنة بعملية التصليد التقليديه مع المراجعه عند 200 °م. عزي التحسن إلى الزياده في ترسبات الكربيدات ألثانويه ولتحول طور ألأوستنيت المتبقي. ألتحسن ألأعلى الناتج من عملية المعاجه بالتبريد ألعميق بسبب أنَ الكربيدات الثانويه كانت أكثر إنتضاماً في التوزيع حيث أنها أصغر حجما وأكثر تقاربا مقارنة بعملية التصليد الثانوي. تحت ضروف ألأختبار المطَبقه صنف البلى في جميع ألأختبارات من النوع المعتدل وفقاً لحسابات معامل البلى

الكلمات المفتاحيه: معالجة التصليد الثانوي، معالجه التبريد العميق، طور الأوستنيت المتبقي، البلى

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