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Effect of Cryogenic Heat Treatment on Multiple Tempered D-2 Tool Steel

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Abstract: *The present day scenario in the manufacturing sector demands high productivity and the life of cutting tools plays a major role in increasing productivity. The freezing of metals has been acknowledged, for many decades, as an effective method for increasing" wear life" and decreasing residual stress in tool steels. Wear test using pin-on-disc machine was used to investigate the effect of multiple tempering after cryogenic treatment of D-2 tool steel. Conventional quenching (1010◦C) and tempering (515◦C) treatments were given along with intermediate cryogenic treatment (−196◦C). Specimens were subjected to wear tests on pin-on-disc machine in dry sliding condition for sliding distance of 6000 m at 6 kg load and for sliding speed of 3.0 m/s. Hardness data, microstructures, wear loss and Microstructure analys is analysis of worn surface throw light on the underlying metallurgical mechanism responsible in improving wear resistance property of the D-2 tool steel.*

Keywords: Tool steel, Cryogenic treatments, D-2 tool steel,dry sliding test, microstructure, Phase transitions.Multiple tempering

I. INTRODUCTION

Cryogenically treated materials find a lot of applications in engineering and allied industries. Cryogenic treatment may not always improve the performance of materials and its workability needs to be checked after some trials in designing the treatment cycle. Cryogenic treatment in tool steels causes the precipitation of finely dispersed carbides in martensite and also converts soft unstable austenite to martensite. Literature of past work does not adequately clarify the selection of tempering, cryogenic temperature and soaking time. There is a need to standardize the process for cryogenic treatment in particular tool steels and understand the underlying metallurgical mechanism responsible for improvement of wear.

II. LITERATURE REVIEW

Barron (1982) has shown that the metals, such as tool steels, which can exhibit retained austenite at room temperature, can have wear resistance significantly increased by subjecting the metal to a long soak (longer than 20 h) at temperatures of the order of 77 K. This lower temperature treatment was preferable than soaking at 189 K. For stainless steels, however, soak at 189K was satisfactory in improving the wear resistance by as much as 25%, although the lower temperature soak would improve the wear resistance by approximately the same factor. The wear resistance of plain carbon steels and cast iron was not significantly affected by the low-temperature treatment. The hardness of the materials was not influenced by the cryogenic treatment for any of the samples.L.Bourithis et.al [1] studied commercial cold work tool steels, AISI D2 and O1.Their results show that for relatively low sliding speeds, AISI O1 steel performs up to 12 times better than AISI D2 steel in adhesive wear. For higher sliding speeds, however, this order is reversed due to oxidation taking place on the surface of the AISI D2 steel. The friction coefficient of the AISI O1 tool steel against Al_2O_3 is constant regardless of the conditions applied and has a value of 0.55. The friction coefficient of AISI D2 tool steel against Al_2O_3 decreases progressively from 0.74 to 0.58 with increasing sliding speed, due to a change in the primary wear mechanism from delamination to oxidation wear.M. H. Staia et.al [13] were conducted the wear experiments at 25°C, at 300°C and at 600°C temperatures and concluded that coating the nitrided D2 tool steel substrate with TiN and TiAlN films deposited by physical vapor deposition (PVD) gives rise to an improvement in the wear behavior in comparison with the uncoated substrate. The effect of deep cryogenic treatment on the properties of AISI M2 and AISI H13 tool steels was studied by A. Molinari et.al [3]. It was observed that the deep cryogenic treatment (at -196°C) on quenched and tempered high speed steel tools increases wear resistance, toughness, hardness, reduces tool consumption and down time for the equipment set up, thus leading to cost reduction of about 50%. N. B. Dhokey et.al [8] were used pin-on-disc machine to test wear rate of D3 tool steel. Conventional quenching (at 950ºC) and tempering (at 150ºC) treatments were given along with intermediate cryogenic treatment (at -185ºC). Cryogenically treated D3 steel shows decreasing hardness from single stage to triple stage tempering whereas hardness of HT and HCT remains same. Specimens were subjected to wear tests on pin-on-disc machine in dry sliding condition for sliding distance of 6000m at 5.5kg load and for sliding speed of 3m/s. It was seen that wear rate was lowest in single tempered D3 steel that is 93% reduction in wear rate than that of HT. Subsequent tempering, i.e. double and triple tempering deteriorates wear resistance of D3 steel. D. Das, et.al [4, 5, 6, 7, 16] have done the Deep Cryogenic Treatment (DCT) on AISI D2 tool steel. Their results indicate that Deep Cryogenic Treatment (DCT) markedly enhances the wear resistance of the selected steel compared to Conventional Heat Treatment (CHT) and Cold Treatment (CT). The wear rate (WR) increases linearly with increasing normal load (FN) for all types of specimens. The degree of improvement in wear resistance (WR) by CT and DCT in comparison to CHT varies from 39% to 12% and from 88% to 34%, respectively, with increasing FN from 78.5N to 137.3N.Cord Henrik Surberg et.al [6] worked on the AISI D2 tool steel which was processed by vacuum hardening followed by multiple tempering cycles. They suggested that a deep cryogenic treatment in between the hardening and tempering processes could reduce processing time and improve the final properties and dimensional stability. Hardened blocks were then subjected to various combinations of single and multiple tempering steps (at 520°C and at 540°C) and deep cold treatments (at -90°C, at -120°C and at -150°C). The greatest dimensional stability was achieved by deep cryogenic treatments at the lowest temperature used and was independent of the deep cryogenic treatment time. The wear properties, hardness values and the micro-structural characteristics of AISI D2 tool steel cryotreated at 77K for different soaking durations (0–132Hr) were examined by D. Das et.al [7] to find out the optimized cryogenic processing time (tCP) for maximization of its wear resistance. There results indicate that the selected material exhibits the best resistance to wear (WR) when cryotreated for 36 Hr.

A. Literature Gap

From the literature review, it is found that cryogenic treatment has tremendous potential for enhancing the wear resistance, hardness and toughness of tool steel materials. Substantial work has been done on the effect of cryotreatment on different tool steels to enhance their mechanical properties. Cryotreatment technology has not been widely adopted by the industries due to lack of understanding of the fundamental metallurgical mechanisms and due to the wide variation in reported research findings Literature of cryotreatment does not adequately clarify the selection of tempering temperature, tempering period and tempering cycle. So, there is a need to standardize the tempering process for particular tool steels. The underlying mechanisms behind the enhancement of wear resistance of tool/die steels by deep cryogenic treatment are still debated and yet to get crystallized. The greatest improvement in properties is obtained by selecting proper heat treatment process sequence, soaking time (cryoprocess time), stabilization, hardening temperature, heating and cooling rate Determination of appropriate level of the above parameters results in to enhance the product quality, productivity and wider acceptance in the industries. The main focus of this work is to standardizing processing cycles including austenitizing temperature, quenching temperature, soaking temperature, soaking period, tempering temperatures, tempering period and tempering cycles to optimize the properties of the material.

B. Objective of Project Work

The purpose of this work is to investigate the effect of multiple tempering after cryogenic treatment of D-2 tool steel, Characterization study with regard to micro hardness, microstructure and retained austenite to investigate the underlying mechanism. Residual stresses induced due to cryogenic treatment so to confirm whether tempering has to be done before or after cryogenic treatment or it is immaterial.

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III. MATRIALS AND METHODS

A. Material used

For the present investigation the material selected from group D is AISI D2 which is most commonly used tool steel material for dies with hardness ranging from 60-64 HRC. Generally D2 material is used where long run application (long tool life) is required. The cost of D2 tool steel is more. The pin size required to conduct wear test on Pin- on – disc tribometer is in range of dia.3 mm to 12 mm so the round bars of diameter 10 mm were selected for this experimentation. The specimens for wear test were prepared of diameter 10 mm and height 30 mm. The chemical composition of specimen is as follow,The chemical composition of specimen is as follows,

Table No. 1 Chemical Composition of D2 Tool Steel

Sr. No.	Element	Weight Percentage	
		Content as per physical test conducted	AISI Specification of D2 Tool Steel
1	C	1.5	1.40-1.60
2	Mn	0.39	0.60 Max.
3	Cr	12.19	11.0-13.0
4	Mo	1.12	0.70-1.20
5	V	0.37	1.10 Max.
6	Iron	Remaining	

B. HEAT TREATMENT

The materials selected for this study were heat treated as per procedure prescribed in ASTM A 681-08 standards and the heat treatment process combinations to specimen are shown in

Table No 2. Various kind of heat treatment employed to D2 Steel.

Sr. No.	Heat Treatment Sequence	Heat Treatment Sequence Details
B1	AQT	Austenitizing + Quenching + tempering.
B2	AQC	Austenitizing + Quenching + Cryogenic Treatment.
B3	AQCT	Austenitizing + Quenching + Cryogenic + tempering.
B4	AQCTT	Austenitizing + Quenching+ Cryogenic + double tempering.
B5	AQCTTT	Austenitizing + Quenching + Cryogenic +Triple tempering.

1. Hardening (Austenitizing)

The first step in the heat treatment of AISI D2 tool steel was hardening. The purpose of hardening was to harden steel to increase the wear resistance, cutting ability. Hardening of tool steel was done in electrically heated protected atmosphere type furnace. The Pre heating was done at temperature650^0C and hardening at a temperature of 1010°C for 15 min. to achieve the austenitization point & grain uniformity (Structural balance).

2. Quenching

Harden tool steel followed cooling which provides great benefit of minimizing distortion and dimensional changes. Quenching is done at 540^0C in cold oil (Hiquench M-Hard castle petrofer make) for 20 min. with continuous agitation, to avoid thermal shocks.

3. Tempering

Tempering of tool steel was done in electrically heated protected atmosphere type furnace to relieve the internal stresses developed due to rapid cooling of steels after hardening process and to reduce brittleness due to volumechanges occurring in the austenite to martensite transformation.

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4. Cryogenic Treatment

The improvement in mechanical properties of tool steels subjected to cryogenic treatment depends on to the combined effect of transformation of retained austenite to martensite and precipitation of fine carbides. From the literature review better improvement for cold work tool steel is obtained by DCT at temperature range of -185°C to -196°C and the soaking period of range 36 h.So, in the present work, the specimens were cryotreated at -196°C and the soaking time selected is 36 hr.bath was allowed to cool down slowly (3-4°C/ min) to avoid thermal shocks. Once the cryogenic treatment was over, all the specimens were allowed to warm up in an insulated thermocol box which takes normally 16-24 hr to reach the room temperature depending on the treatment given.

5. Micro-structural Examination

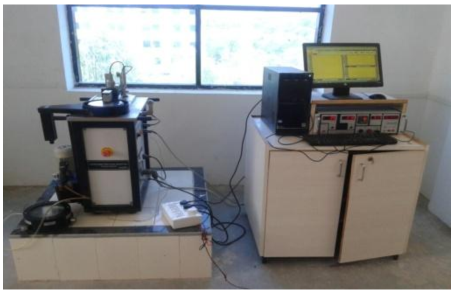
Microstructure analysis was carried by inverted optical microscope (Make-CARL ZEISS Germany, Model-AL350) at magnification 450X. Bakelite moulds are prepared and moulds are first surface leveled on endless emery belt (80/0) paper. Further samples were subjected to separately polishing on emery paper (240, 400, 600, 800 and 1000) so as to make surface free from scratches. Final polishing was done on velvet cloth polishing machine with intermittent application of fine suspensions of alumina to get better finish on polished surface. The polished specimens were etched using 3% Nital and 5% picral solution for microstructural examination. The freshly prepared etchant 'Nital', of composition approximately 3 ml Nitric acid with 100 ml ethyl alcohol and Picral of composition 5 g picric acid in 100 ml ethanol, was used for revealing micro constituents of tool steel. Microstructures were then recorded by image analyzer system. Different phases like retained austenite, untemperedmartensite, tempered martensite were checked. Also carbide sizes and precipitation of fine carbide were observed. Carbides are divided in two categories viz. Primary carbides (size > 5 microns) and secondary carbides (within 1 micron to 5 microns).

6. Hardness Measurement

The hardness test was carried out by using Vickers Hardness test method. The samples were taken from each group and subjected to the hardness test. The Vickers hardness test carried out in such a way that three indentation were made in each test sample. The hardness number was determined based on the formation of the indentation due to the applied force. The flat surface was prepared by polishing paper on 1/0. For Hardness measurement the load applied was 10 kgf for a dwell time 10s. Three readings are taken for each type of treatment and the arithmetic mean is used as Vickers hardness number. The average Vickers hardness number and its equivalent Rockwell hardness number are also calculating for discussion.

7. Experimental Test Rig

In order to investigate the wear resistance of the heat treated AISI D2 tool steels to adhesion wear, the pin on disc method was used. Dry sliding wear tests were carried out on computerized pin-on-disc "Wear and Friction monitor" (DUCOM: TR- 20LE-PHM-400) .The photograph of experimental set up is shown in fig.



Photograph of experimental set up (Tribometer TR-20LE).

7.1 Specifications of PIN and Disc Tribometer (TR-20LE)

The specifications of Pin-on-Disc Wear and Friction monitor are shown in Table No. 5.4.

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Table No.3 Specifications of pin on disc Tribometer (TR-20LE)

Make	Ducom Ltd, Bangalore.
Pin Size	3 to 12 mm diagonal
Disc Size	165 mm dia. X 8 mm thick
Wear Track Diameter (Mean)	10 mm to 140 mm
Sliding Speed Range	0.26 m/sec. to 10 m/sec.
Disc Rotation Speed	100-2000 RPM
Normal Load	5N to 200 N Maximum
Friction Force	0-200 N, digital readout, recorder output
Wear Measurement Range	$\pm$2 mm, LC 0.1μm digital readout, and recorder output
Power	Kw , 230V, 15A, 1 Phase, 50 Hz

7.2 Test Conditions

Sliding wear tests were performed according to ASTM G99-05 standard using pin on disc wear testing machine (DUCOM: TR 20LE). From the literature review the experimental parameter such as sliding velocity, load, sliding time and lubrication condition for wear test were decided. From literature the sliding velocity for selected material is in the range of 3 m/s and load applied in the range of 6. As per literature the wear tests were continued for sliding duration 60 minute because during this duration cumulative height loss of pin specimen were approximately 2 mm or equivalalent to 25 mm^3 cumulative volume loss. The experimental parameter for AISI D2 is as shown in Table 5.5.

Table No. 4 Experimental parameter

Sr. No.	Parameters	Values
1	Velocity	3.0 m/s
2	Load	6 kg
3	Sliding time	60 min
4	Lubricant Condition	Dry

The test samples were made as the Pin. During testing pin of 10mm diameter and 30mm length was clamped in a holder and held against the rotating counter disc made of WC - coated EN-35 with the roughness value, $R_a < 0.5\mu m$. The faces of the pin specimens were polished and cleaned in acetone in an ultrasonic cleaner. The wear tests were carried out at three different normal loads (F_N): 58.86 N (6 kg) at a constant linear sliding velocity (V) of 3 m/s in dry condition at the room temperature. The values estimated nominal contact pressure in the pin specimens are 1.7486 Mpa corresponding to F_N of 6 kg, respectively. The wear tests were carried for 60 minutes duration. The surface of each specimen was cleaned at each time before each test. Every time new track radius of rotating disc was used so that pin was exposed to fresh surface of counter face. Wear and frictional force was measured continuously through a load cell measuring the tangential force.

IV. RESULT AND DISCUSSION

A. Hardness Study

The results obtained from the Vickers hardness test of the AISI D2 tool steel for different heat treatments sequence are tabulated in Table 5 The result shows cryogenically treated samples had an increase in the hardness level compared to conventional heat-treated samples. The hardness of the cryotreated samples of AISI D2 tool steel showed an improvement of 2% to 15 % over the CHT samples the increment in hardness may be attributed to the complete transformation of the austenite to martensite.

Table No. 5 Percentage increase in hardness as compare to CHT Specimens

Sr. No.	Heat Treatment Sequence	D2		
		HV	HRC	% increase as compare to CHT
B1	CHT	805	64	0
B2	AQC	927	68	15.15

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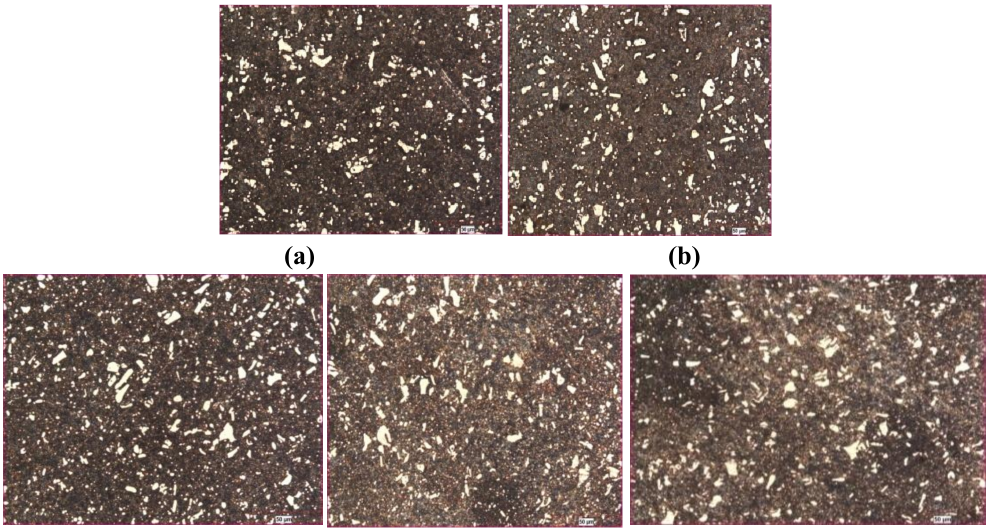
B3	AQCT	916	67	13.79
B4	AQCTT	841	65	4.47
B5	AQCTTT	828.7	65	2.94

The hardness of AQC samples of D2 tool steel is highest. Because the untempered structure has the highest hardness but the material is more brittle due to presence of more untempered martensite which is seen in the microstructure. Martensite is a highly supersaturated solid solution of carbon in iron. Hence, tempering should be done to reduce the brittleness by scarifying some hardness and to relieve internal stresses and to increase toughness and ductility. During tempering, martensite rejects carbon in the form of finely divided carbide phases. The end result of tempering is a fine dispersion of carbides in the α-iron matrix.

Cryogenic treatment is more effective in reducing the amount of austenite and can make a larger number of fine secondary carbides precipitate, which can increase the dispersion strengthening effect; both are beneficial for increased hardness. Also, the decrease in grain size can improve the hardness. The grain size of martensite is smaller than that of austenite; cryogenic treatment can produce more martensite, which implies a fine grain strengthening effect. So, this can also be the fact that the hardness of the alloy in cryogenic treatment is higher than that in the CHT samples.

B. Microstructure analysis of D2 tool steel

Microstructure of various combinations of treatment for the specimen of D2 steel is shown in fig.4.1 (a-e). Fig (a) shows microstructure of CHT specimen in which 50% retained austenite is present after tempering and more large size globular shape (9μ) and nodular shape (11x4 μ) size carbides are present. Untemperedmartensite observed is more up to 10%. Fig (b) shows microstructure of AQC specimen in which Massive carbides not seen and 95% unstable austenitic structure seen. The structure consists of 7 % large size secondary carbides. More untemperedmartensite observed. Fig (c) shows microstructure of AQCT specimen in whichRetained austenite is present up to 20% under tempered structure and small size carbide present up to 10%. Untemperedmartensite observed below 2 %. Fig (d) shows microstructure of AQCTT specimen in which 50% retained austenite present under tempered structure. Small size carbide is observed up to 12% in matrix of tempered martensite. Untemperedmartensite observed is more up to 5 %.



(a) (b)

(c) (d) (e)

Fig. 4.1 Microstructure of various combinations of treatments for the specimen of D2 Tool steel
a) CHT b) AQC c) AQCT d) AQCTT e)AQCTT

Fig(e)shows microstructure of AQCTTT specimen Microstructures of all combination treatment shows carbides in globular and nodular shape and is uniformly distributed in the matrix of tempered martesites.

The microstructure of the AQCT, AQCTT samples of D2 tool steel shows that most of the retained austenite was converted into martensite, thereby increasing the hardness and an improvement in its wear resistance compared to the CHT sample. In addition to the conversion of the retained austenite into martensite, the microstructure of the AQCT,

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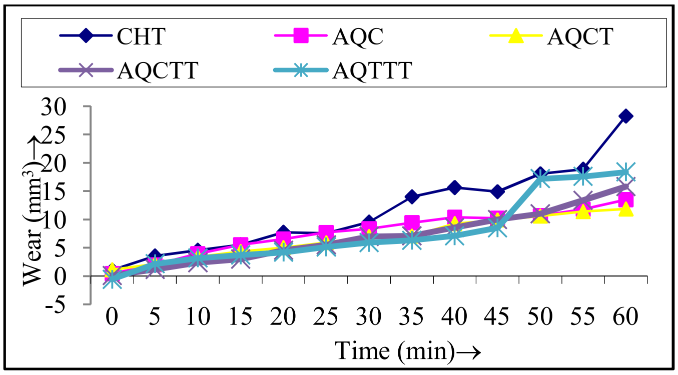
AQCTT and AQCTTT samples showed a marked increase in carbide precipitation. This indicates that tempering subsequent to cryogenic treatment is essential to cause carbide precipitation in the martensitic matrix. These are also responsible for the improvement in the wear resistance and hardness.

After cryogenic treatment, there was less retained austenite in the heat treated samples of AISI D2 tool steel because the retained austenite is more unstable at lower temperatures and likely to transform into martensite. It can be seen from the micrograph that a large amount of fine carbides of micrometer size were precipitated throughout the structure. The fine carbides precipitated through the cryogenic treatment tie up with certain elements and restrict the promotion of instability during service. It is noted that holding martensite at a lower temperature increases its lattice distortion and thermodynamic instability, both of which drive carbon and alloying atoms to segregate at the nearby crystal defects. These segregated regions act as sites for nucleation of fine carbides.

C. WEAR MESUREMENT

1. Wear (mm^3)

It is observed that both normal load and sliding velocity affect the wear volume of the specimens. The wear volume increased with increasing normal load and sliding speed. It was observed that the cryogenically treated specimens have less wear as compared to conventional heat treatment specimens. Apparently there is no straight correlation with hardness. Even though difference of hardness of conventional heat treatment and cryotreated specimens is not much more, but there is a dramatic drop in wear volume in wear test. There is an increasing wear volume reflected in multiple tempering specimens.



Graph No.2 Wear of D2 material for 6Kg load and 3m/s velocity for different heat treatment.

From wear test report at different load and speed it is observed that for D2 tool steel the wear was increased in the order of process sequence AQCT, AQCTT, AQCTTT, AQC, CHT specimens. In the case of AQCT Specimens, the large reduction in wear could be mainly due to complete transformation of the retained austenite to martensite and the precipitation of more amounts of fine carbides during cryogenic treatment and subsequent tempering as compared to other heat treated specimens.

2. WEAR RESISATANCE (WR)

Wear behavior can be conveniently expressed in terms of dimensionless wear coefficient (k). The inverse of dimensionless wear coefficient in known as wear resistance (WR).The calculated wear resistance of different heat treated specimens are depicted in graph

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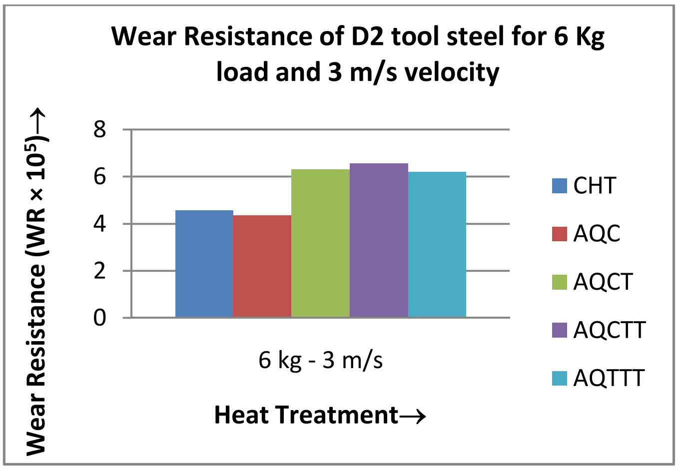
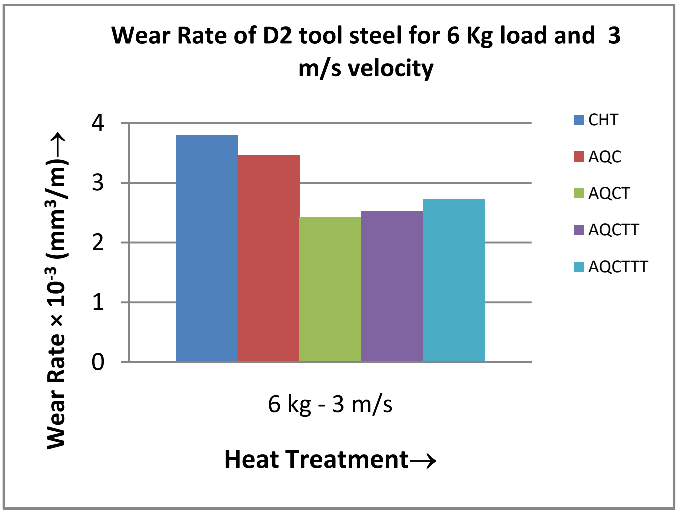


Graph. 3 Wear Resistance (WR) of D2 tool steel for different heat treatment sequence

3. Wear Rate (WR)

From wear test report it is observed that for D2 tool steel the wear rate was increased in the order of heat treatment process sequence AQCT, AQCTT, AQCTTT, AQC and CHT specimens.



Graph. 4 Wear Rate (WR) of D2 tool steel for different heat treatment sequence

V. CONCLUSION

The present investigation based on the effect of cryotreatment on friction and wear behaviour of cold work tool steel,after conducting the experimental work, following conclusions can be drawn from the results

- Cryogenic treatment is an add on process to conventional heat treatment process of tool steel.
- Cryogenic treatment improves microstructure of metal i.e. controlled transformation of retained austenite into martensite.
- The hardness of the cryotreated samples of AISI D2 tool steel showed an improvement of 2% to 15 % over the CHT samples. The increment in hardness may be attributed to the complete transformation of the austenite to martensite.
- The hardness of AQC samples of D2 tool steel are highest but having less wear resistance because the untempered structure has the highest hardness but the material is more brittle due to presence of more untempered martensite which is seen in the microstructure. Hence, tempering should be done to reduce the brittleness by scarifying some hardness and to relieve internal stresses and to increase toughness and ductility.

- The microstructure of D2 tool steel shows that upto maximum 80% the retained austenite was converted into martensite for same heat treatment process cycle.
- The comparison of microstructure of AQC specimen and AQCT, AQCTTT, AQCTT, CHT specimen of tool steel indicates that tempering subsequent to cryogenic treatment is essential to cause carbide precipitation in the martensitic matrix. These are also responsible for the improvement in the wear resistance.
- The wear volume, wear rate (W_R) increases linearly with increasing normal load for all type of samples. Also it was observed that at higher velocities the wear rate is enhanced.
- The lowest wear volume, and wear rate was observed in AQCT specimens of AISI D2 tool steel.
- Wear volume, coefficient of friction, wear rate and wear resistance of the materials depends upon the combination of heat treatments.
- Therefore, among different heat treatment sequence AQCT heat treatment is observed to be optimum for AISI D2 tool steel.

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