Dry Sliding Wear Analysis of D5 Tool Steel at Different Heat Treatments

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ABSTRACT

Wear test using pin-on-disc machine was used to investigate the role of different heat treatments on AISI D5 tool steel. For this purpose, the hardening (at 1020°C) for one hour, multiple tempering (at 210°C) for two hours and soft tempering(at 100°C) for two hours and soft tempering(at 100°C) for two hours treatments along with intermediate deep cryogenic treatment(at -185°C) for 36 hours selected. Microstructural characterizations of the differently treated specimens have been done by image analyses of optical micrographs software with inverted microscope. Wear tests were performed at two different loads (3.1 kg and 5.1 kg) and two different velocities (1.5 and 2.5 m/s) were applied. Whereas wear behavior has been characterized by wear rate and wear resistance. Hardness of specimens was measured by using Rockwell Hardness tester. The findings shows that the cryogenic treatment decreases the retained austenite and hence improves the wear resistance and hardness, due to more homogenized carbide distribution as well as the elimination of the retained austenite, the deep cryogenic treatment demonstrated more improvement in wear resistance and hardness compared with the conventional heat-treatment. The results indicate that, in HCT specimens there was large reduction in the wear rate and markedly enhancement in wear resistance of D5 tool steel.

KEYWORDS : AISI D5 tool steel, cryogenic treatment (CT), Wear behavior, Design of Experiment (DOE), Wear rate, Wear resistance, retain austenite, Martensite

I. INTRODUCTION

In metal forming industry tools can be exposed to very complex and surface demanding conditions, which are the result of different effects (mechanical, thermal, chemical or tribological loading) and require well defined mechanical and especially tribological properties. Wear which is one of the main cause of material wastage, is an important problem associated with industrial components. Though wear resistance is not a materials property, an understanding of the dominant wear mechanisms is very essential. The cost of wear to industry is high and the recognition of this fact lies behind the continuing development in the field of advanced materials, in order to provide a solution for mitigating tribological losses.

In tool steels, a low percentage of austenite is retained after the conventional heat-treatment named “retained austenite”. The retained austenite as a soft phase in steels could reduce the product life and, in working conditions, it can be transformed into martensite. This new martensite could cause several problems for working tools. This new martensite is very brittle and differs from the tempered one, which is used in tools. Furthermore, this martensite causes micro cracks and reduces the product life.[1] Das et. al[2] selected D2 tool steel for study, The cryogenic processing was done by uniform cooling of the samples to -196°C, and holding the samples at this temperature for different time durations. Hardness of the investigated steel samples is found to increase marginally by cryotreatment in contrast to significant increase in their wear resistance. Within the investigated range, the wear resistance of the cryotreated specimens increases with increasing holding time up to 36 h -196°C at beyond which it shows monotonic decrease with further increase in holding time. N.B. Dhokey et. Al[3] studied D3 tool steel to investigate the role of multiple tempering after cryogenic treatment of D3 tool steel. It was seen that wear rate was lowest in single tempered D3 steel, that is 93% reduction in wear rate than that of HT.
For the same hardness of HT and HCT, the wear rate is a strong function of carbide size and its distribution. Coarsening of carbides lowers wear resistance as seen in double and triple-tempered condition. Akhbarizadeh et al. [8] studied the effects of cryogenic treatment on the wear behaviour of D6 tool steel. For this purpose, two temperatures were used: -63°C as shallow cryogenic temperature and -185°C as deep cryogenic temperature. Due to more homogenized carbide distribution as well as the elimination of the retained austenite, the deep cryogenic treatment demonstrated more improvement in wear resistance and hardness compared with the shallow cryogenic treatment.

The several researchers find that, Deep Cryogenic Treatment (DCT) considerably improves the wear resistance (WR) of tool steels than those obtained either by Cold Treatment (CT) or by Conventional Heat Treatment (HT). Also it is found that the considerable reduction in wear rate (WR) and coefficient of friction (µ). It has also been reported that DCT and multiple tempering after cryogenic treatment enhances the dimensional stability and reduces the residual stresses. These favorable effects increase the service life of the components made of AISI tool steels. But past work does not clarify optimum combination of heat treatments i.e. hardening, multiple tempering, cryogenic temperature and soaking time which produce minimum wear rate and high wear resistance. This is the main focus of the present work on D5 tool steel.

II. EXPERIMENTAL DETAILS

The present investigation has been conducted with samples of AISI D5 tool steel. The chemical composition of specimen (diameter 9.15 mm; height 30 mm) as analysed by Optical Emission Spectrometer (ASTM-E-1066-2008) is given in Table no 1.

Table no 1. Chemical Composition of D5 Tool Steel

<table>
<thead>
<tr>
<th>Sr. No.</th>
<th>Element</th>
<th>Weight Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>C</td>
<td>1.52</td>
</tr>
<tr>
<td>2</td>
<td>Mn</td>
<td>0.43</td>
</tr>
<tr>
<td>3</td>
<td>Cr</td>
<td>11.57</td>
</tr>
<tr>
<td>4</td>
<td>Mo</td>
<td>0.88</td>
</tr>
<tr>
<td>5</td>
<td>Co</td>
<td>2.99</td>
</tr>
<tr>
<td>6</td>
<td>V</td>
<td>0.21</td>
</tr>
<tr>
<td>7</td>
<td>Si</td>
<td>0.36</td>
</tr>
<tr>
<td>8</td>
<td>S</td>
<td>0.012</td>
</tr>
<tr>
<td>9</td>
<td>P</td>
<td>0.016</td>
</tr>
<tr>
<td>10</td>
<td>Fr</td>
<td>Balance</td>
</tr>
</tbody>
</table>

2.1 Treatments

The material chosen in this work was given various treatments and treatment cycles indicated in Table no

Hardening

The first step in the heat treatment of AISI D5 tool steel was hardening. The purpose of hardening was to harden steel to increase the wear resistance, cutting ability. Hardening of AISI D5 tool steel was done in the tubular furnace (3.5KW, 230V AC, 15A, 1200±10°C) at a temperature of 1020°C [1, 6] for 1 Hour. During hardening process, inert gas Argon was supplied in tubular furnace to avoid oxidation. Harden AISI D5 tool steel followed air cooling which provides great benefit of minimizing distortion and dimensional changes [11]. Tempering The process which consists of heating the hardened components to a temperature between 100°C and 700°C, holding at this temperature for specific period and cooling to room temperature, usually by air [12]. Tempering of D5 tool steel was done in the muffle furnace (3.8KW, 230V AC, 600±10°C). Samples of treatment HT, HTT, HTTT, HCT, HCTT, HTC, HTTC and HTTTC after H and HC were immediately subjected to tempering at temperature of 210°C with 2 Hr soaking time, followed by air cooling to room temperature, that is,
single tempering. After air cooling the remaining samples were subjected to double tempering for the same temperature and time and then air cooled. Similar procedure is again followed for triple tempering on the remaining samples from double tempering. The detailed heat treatments are indicated in Table no.

Table no 2. Different Heat Treatments Employed to AISI D5 tool steel

<table>
<thead>
<tr>
<th>Sr.No</th>
<th>Nomenclature</th>
<th>Particulars of Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>HT</td>
<td>Hardening (1020°C for 1 Hr), Single Tempering (210°C for 2 Hr)</td>
</tr>
<tr>
<td>2</td>
<td>HTT</td>
<td>Hardening (1020°C for 1 Hr), Double Tempering (210°C for 2 Hr)</td>
</tr>
<tr>
<td>3</td>
<td>HTTT</td>
<td>Hardening (1020°C for 1 Hr), Triple Tempering (210°C for 2 Hr)</td>
</tr>
<tr>
<td>4</td>
<td>HCT</td>
<td>Hardening (1020°C for 1 Hr), Cryotreated (-185°C for 36 Hr), Single Tempering (210°C for 2 Hr)</td>
</tr>
<tr>
<td>5</td>
<td>HCTT</td>
<td>Hardening (1020°C for 1 Hr), Cryotreated (-185°C for 36 Hr), Double Tempering (210°C for 2 Hr)</td>
</tr>
<tr>
<td>6</td>
<td>HCTTT</td>
<td>Hardening (1020°C for 1 Hr), Cryotreated (-185°C for 36 Hr), Triple Tempering (210°C for 2 Hr)</td>
</tr>
<tr>
<td>7</td>
<td>HCST</td>
<td>Hardening (1020°C for 1 Hr), Cryotreated (-185°C for 36 Hr), Soft Tempering (100°C for 1 Hr)</td>
</tr>
<tr>
<td>8</td>
<td>HTCST</td>
<td>Hardening (1020°C for 1 Hr), Single Tempering (210°C for 2 Hr), Cryotreated (-185°C for 36 Hr), Soft Tempering (100°C for 1 Hr)</td>
</tr>
<tr>
<td>9</td>
<td>HTTCST</td>
<td>Hardening (1020°C for 1 Hr), Double Tempering (210°C for 2Hr), Cryotreated (-185°C for 36 Hr), Soft Tempering (100°C for 1 Hr)</td>
</tr>
<tr>
<td>10</td>
<td>HTTTCST</td>
<td>Hardening (1020°C for 1 Hr), Triple Tempering (210°C for 2 Hr), Cryotreated (-185°C for 36 Hr), Soft Tempering (100°C for 1 Hr)</td>
</tr>
</tbody>
</table>

Cryogenic Treatment
The block diagram of cryoprocessor is as shown in Fig 1. As soon as liquid nitrogen enters into Cryoprocessor, it gasifies immediately through multi hole slitter and thus cooling of the specimen takes place. Temperature of the bath is sensed using Resistance Temperature Detector, which provides online feedback of temperature of the bath to regulate the flow of liquid nitrogen.

![Fig 1. Block Diagram of Cryoprocessor](image)

In the present work, the specimens were cryotreated at -185°C, the soaking time was selected 36 Hr [7]. The bath was allowed to cool down slowly (3-4°C/ min) to avoid thermal shocks.
Once the subzero 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. A computer generated thermal profile of the processor is shown in Fig.2. Blue line stands for instantaneous process temperature and red line stands for set point which was maintained manually. It must be noted that the actual process temperature follows the set points.

After cryogenic treatment the specimen of HTCST, HTTCST, HTTTCST and HCST are followed by soft tempering. Soft tempering was done in muffle furnace (3.8KW, 230V AC, 600±10°C) at 100°C for one Hr.

![Computer Generated Thermal Profile of Cryoprocessor](image)

2.2 Metalography

Microstructure analysis was carried by image analyzer software; with inverted microscope (Make-CARL ZEISS Germany, Model-Axiovert 40Mat). Carefully prepared samples were 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. A freshly prepared etchant “Nital”, of composition approximately 5 ml Nitric acid with 100 ml ethyl alcohol (i.e. approximately 5%), was used for revealing micro constituents of AISI D5 tool steel. Microstructures were then recorded by image analyzer system as shown in Fig 3.
2.3 Hardness Measurement
The hardness of polished AISI D5 tool steel specimens was measured on a Rockwell hardness testing machine. The samples prepared for metallography, are used for hardness measurement. The hardness is measured on “C” scale with 10Kg minor load and 150Kg major load applied. A minimum of 5 readings have been taken to estimate the average value of hardness of the specimen.

2.4 Evaluation of wear behaviour

In order to investigate the resistance of the heat treated AISI D5 tool steels to adhesion wear the computerized pin-on-disc tribometer as shown in Fig. 4 was used. Dry sliding wear tests were carried out on this computerized pin-on-disc wear testing machine (DUCOM: TR 20LE, India). During testing, pin of D5 steel specimen was kept stationary while the circular disc was rotated. The apparatus consisted of an EN-31(68 HRC maintained) steel disc of diameter 160 mm used as counter face. The test sample was clamped in a holder and held against the rotating steel disc as shown in Fig. schematically. Specimens of D5 steel were subjected to
wear test as per experimental plan indicated in Table 1. Wear behaviour can be conveniently expressed by Wear Volume (W), wear rates \((W_r)\) and dimensionless wear coefficient \((k)\).

The wear rates \((W_r)\) have been estimated as wear volume loss \((\text{mm}^3)\) per unit sliding distance \((m)\). The Archard’s Law of Wear expressed as,

\[
W_r = k \frac{F_N}{H} \frac{L}{W}
\]

Where, \(W_r\) = wear rate \((\text{mm}^3/m)\), \(W\) = wear volume \((\text{mm}^3)\), \(L\) = sliding distance \((m)\), \(k\) = wear coefficient, \(F_N\) = normal load \((\text{Kg})\), \(H\) = hardness \((\text{kg/mm}^2)\).

III. RESULT AND DISCUSSION

3.1 Hardness Study

The result shows that cryogenic treatment increases hardness as shown in Fig.5. It is observed that for D5 tool steel the hardness of HCT specimens improves approximately 5-10% as compared to HT specimens. It is also observed that the hardness value for multiple tempering before and after cryotreatment were decreases. The hardness values for specimens subjected to different heat treatment can be directly related to the magnitude of reduction of soft retained austenite with associated improvement in the amount of hard secondary carbides and tough tempered martensite.

3.2 Microstructure Evaluation

Microstructure in as-received state, indicated in Fig.3(a), shows heavy segregation of carbides that are bit massive in size. Fig. depicts microstructure of D5 tool steel for HT, HTT, HTTT, HCST, HTCST, HTTCST and HTTTTCST treatments specimen exhibit non-uniform distribution of large, elongated, white regions of primary chromium carbides and uniform distribution of nearly spherical secondary chromium carbides. Such high wear rate could be attributed to primarily coarse carbides. But for HCT, HCTT and HCTTT specimens there is uniform distribution of white regions of primary chromium carbides and nearly spherical secondary chromium carbides.

This as-received steel was subjected to hardening in order to dissolve carbides and subsequently followed by sequential treatments (e.g. tempering, cryotreatment, multiple tempering). It is clearly seen that there is a definite reduction in carbide size as observed in Fig.3 (d) It is seen that multiple tempering has deteriorated the performance as evident from hardness data and effect of multiple tempering has reflected in coarsening of carbides. Coarsening of carbides might have been caused by Oswald ripening. Hence it was necessary to evaluate performance of material using dry sliding wear test.

3.3 Wear Mechanism

It is observed that both normal load and sliding velocity affect the wear volume, which attains values between 1.596 \(\text{mm}^3\) to 11.365 \(\text{mm}^3\). The wear volume increases with increasing normal load, but wear volume of the D5 tool steel observed to be fluctuating with increase in 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 HT and HCT specimens is not much more, there is a dramatic drop in wear volume in wear test. There is an increasing wear volume reflected in multiple tempering specimens. From wear test report it is observed that the wear volume was increased in the order of HCT, HCTT, HCTTT, HCST, HTCST, HTTCST, HTTT, HTT and HT specimens. In the case of HCT Specimens, the large reduction in wear could be mainly due to the retained austenite elimination and the homogenized carbide distribution as well as more chromium carbide as compared to other heat treated specimens.

Wear test report reflect that the coefficient of friction of D5 tool steel increases due to multiple tempering before and after the cryogenic treatment. For HCT specimens the value of coefficient of friction is very less due to largest hardness as compared to other type of specimens. The coefficient of friction of D5 tool steel observed to be fluctuating with increase in sliding speed. The results in these figures show that the wear rate of HCT specimens is very less as compared to other type of heat treated samples; Such high wear rate could be attributed to primarily coarse carbides. In the case of HCT specimens, the large reduction in wear rate could be mainly due to the homogenized carbide distribution, additional amount of fine carbides nucleated during cryogenic treatment and reduction in carbide size as compared to other heat treated specimens i.e. wear behavior is highly influenced by microstructural parameter like carbide size and its distribution. It is also observed that the wear rate increase linearly with increasing normal load for all types of specimens.

High wear rate in case of HT, HTT, HTTT, HCTT, HCTTT, HCST, HTCST, HTTCST, and HTTTCST specimens as compared to HCT specimens could be attributed to primary coarse carbides. The lowest wear rate is shown by HCT specimens as a result of additional amount of the fine carbides nucleated during cryogenic treatment.

![Graph showing wear rate of D5 tool steel for 3.1Kg load and 1.5m/s velocity](image1)

Fig. 6 Wear Rate of D5 tool steel for 3.1Kg load and 1.5m/s velocity for different heat treatment

![Graph showing wear rate of D5 tool steel for 3.1Kg load and 2.5m/s velocity](image2)

Fig. 7 Wear Rate of D5 tool steel for 3.1Kg load and 2.5m/s velocity for different heat treatment
Wear behavior can be conveniently expressed in terms of dimensionless wear coefficient (k). The inverse of dimensionless wear coefficient is known as wear resistance (WR). It has reported that the improvement in wear resistance could be attributed to \( \eta \)-carbide of nanosize. This is reflected in HCT, whereas wear resistance property deteriorate with double and triple tempering and it is clearly seen that coarsening of carbide is observed. In the double- and triple tempered D5 steel, the carbides coarses and grow in size as evident from (e and f).

### 3.4 Improvement in wear resistance (\( \alpha \ % \))

In order to quantity the magnitude of improvement of WR by HCT over that of HT treatment specimens, a parameter has been considered here, which is defined as follows,

\[
\alpha \ % = \left[ \frac{WR_{HCT}}{WR_{HT}} - 1 \right] \times 100
\]

Where, WR is wear resistance; HCT and HT denote the type of the specimen. The value of have been calculated for different types of specimens under different test conditions. As compared to other type of samples, there is greatest improvement in wear resistance in case of HCT samples. The results are shown for 3.1 kg and 5.1Kg normal load, at 1.5m/s and at 2.5m/s velocity, which illustrates that in comparison to HT, HCT treatment enhance the WR of D5 tool steel by 126.8427 % to 174.6987%.

This experimental work shows that the combined effect of heat treatment and cryogenic treatment can assist in improving wear resistance in single tempering, whereas wear resistance deteriorate in subsequent double and triple tempering.
Correlation of wear properties (Fig.10) with the results of microstructural reveals that the improvement in WR is dependent on the microstructures generated by different heat treatments apart from its dependence on the test conditions. It can be reiterated at this stage that the reduction of retain austenite content and modifications in the size and distribution of SCs are the primary factors responsible for the improvement in WR by HCT over that obtained by the other.

The dependence of α, on F_N is related to the non-linear variation of W_R. Therefore, WR of a material is expected to be dependent on the dynamical changes related to microstructure and properties. The obtained microstructural characteristics of the steel specimens subjected to different heat treatments reveal that the amount of soft υR decreases and concurrently the amount of hard SCs increases (Fig. 3. d), together with their size refinement reduction of inter-particle spacing, and increased population density in the HCT.

3.5 Regression Result

In regression analysis, with the help of regression coefficients we can calculate the correlation coefficients. The square of coefficients, called coefficients of determination R^2(R-Sq), measures the degree of association of correlation that exists between the variables. In general, greater the value of R^2(R-Sq) better is the fit and more useful the regression equation is as predictive device.

For regression analysis Minitab 16 Software was used. Wear Rate versus Load and Speed regression equations has been developed to predict the wear rate for all types of D5 tool steel specimens. The regression equation is

\[\text{Wear rate} = -0.320 + 0.119 \text{ Speed} + 0.175 \text{ Load}\]

\[S = 0.0091315 \quad R-Sq = 100\% \quad R-Sq(adj) = 100\% \]

From regression result, it is observed that the value of R^2(R-Sq) for HCT specimen has greater the value i.e. 100%. Thus better is the fit and more useful the regression equation is as predictive device.

IV. CONCLUSION

[1] Cryogenically treated specimen shows decreasing hardness from single stage to triple stage tempering and Rockwell hardness number (HRC) of HCT specimens improves approximately 5-10 % by HT specimens.
[2] The wear rate (WR) and wear volume increases linearly with increasing normal load for all type of samples. Coefficient of friction decreases with increase in normal load.
[3] It is observed that at higher velocities, wear rate is enhanced, but the coefficient of friction observed to be fluctuating.
[5] It is observed that the largest improvement in wear resistance (WR) is observed in HCT specimens, which is 126.8427 % to 174.6987% that of the HT specimens. Subsequent tempering, i.e. double and triple tempering deteriorates wear resistance. The improvement in WR decreases with increasing severity of wear test conditions, i.e. increasing normal load.
[6] Among different heat treatment combinations, the lowest wear volume, coefficient of friction and wear rate is observed in HCT specimens.
In comparison with conventional heat treatments the deep cryogenic treatment shows significant improvement in wear resistance. As deep cryogenic treatment gives more homogenous carbide distribution, elimination of retain austenite.

V. ACKNOWLEDGEMENTS

And finally this day has come. I am presenting the paper with great pride. There are too much efforts of gardener to yield the beautiful flowers. So we should not forget him while praising flower. It is a matter of gratification for me to pay my respects and acknowledgements to all those who have imparted knowledge and helped me to complete my report.

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Biographies and Photographs
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