



# “Effects of Deep Cryogenic Treatment on Wear Behavior of D6 Tool Steel”

Prof. R.H.Naravade<sup>1</sup>, Prof. R.K.Belkar<sup>2</sup>, Prof. D.S.Bajaj<sup>3</sup>

Lecturer, Department of Mechanical Engineering, P.Dr.V.V.P. Polytechnic, Loni<sup>1</sup>

Head of Mechanical Engineering Department, P.Dr.V.V.P. Polytechnic, Loni<sup>2</sup>

Associate Professor, Department of Mechanical Engineering, Amrutvahini College of Engineering, Sangamner<sup>3</sup>

**Abstract:** This experimental work intended to investigate the effects of cryogenic treatment on the wear behavior of D6 tool steel. For this purpose, the temperature was used  $-185^{\circ}\text{C}$  as deep cryogenic temperature. The effects of cryogenic temperature (deep), cryogenic time (kept at cryogenic temperature for 36 hr) on the wear behavior of D6 tool steel were studied. The findings showed 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. By increasing the keeping time at cryogenic temperatures, more retained austenite was transformed into martensite; thus, the wear resistance was improved and further hardness were observed. This experimental work also intended to investigate the role of multiple tempering before and after cryogenic treatment on friction and wear behaviour of D6 tool steel as classified by American Iron and Steel Institute (AISI). D6 tool steel is used for measuring tools, blanking dies, forming dies, coining dies, long punches, forming rolls, edging rolls, master tools, extrusion dies, drawing dies, moulds for pressing abrasive powders etc. The different combination of heat treatments like hardening (at  $1020^{\circ}\text{C}$ ) for one hour, tempering (at  $210^{\circ}\text{C}$ ) for two hours and deep cryogenic treatment (at  $-185^{\circ}\text{C}$ ) for 36 hours was done on D6 tool steel. Wear test were performed using pin-on-disc wear tester to which two different normal loads (3.1Kg and 5.1Kg) and two different velocities (1.5m/s and 2.5m/s) were applied. Hardness of specimens was measured by using Rockwell Hardness tester. Microstructural characterizations of the differently heat treated specimens have been done by image analyzer software with inverted microscope. The findings show that the cryogenic treatment improves the wear resistance and hardness of D6 tool steel. The results indicate that, in HCT specimens there was large reduction in the wear rate and markedly enhancement in wear resistance of the D6 tool steel.

**Keywords:** AISI D6 tool steel, cryogenic treatment, wear rate and wear resistance.

## I. INTRODUCTION

To understand the effects of cryogenic processing it is essential that one can be acquainted with the heat treating of metals. The primary reason for heat treating steel is to improve its wear resistance through hardening. Gears, bearings, and tooling for example are hardened because they need excellent wear resistance for extended reliability and performance [1]. Cryogenic processing (CP) is presently employed in the fields like aerospace and manufacturing industries, sports and music instruments, firearms etc. for performance enhancement of various components. In the last decade, a good number of investigations have been directed to improve the tribological properties like wear resistance of tool/die steels by cryotreatment. Cryogenic treatment of tooling steels is a proven technology to increase wear resistance and extend intervals between component replacements for dies, punches, drill bits, end mill cutters, bearings, cams, crankshafts, blocks, pistons, blades etc. Controlled CP is commonly appended in-between conventional hardening and tempering treatments for tool/die steels [2]. The execution of the deep cryogenic treatment on quenched and tempered high speed steel tools increases hardness, reduces tool consumption and down time for the equipment set up, thus leading to cost reductions of about 50% [3]. In last few years it is found that the use of combination of different heat treatment like hardening, multiple tempering and cryogenic treatments effect on the friction and wear behaviour of AISI D6 tool steel [4].

## II. AISI D6 TOOL STEEL

Tool steels were developed to resist wear at temperatures of forming and cutting applications. Tool steel is used on a wide variety of applications where resistance to wear, strength, toughness and other properties are selected for optimum performance. They are broadly divided into six categories: cold work, shock resisting, hot work, high speed, mold and special-purpose tool steels [1]. Generally speaking, many tool steels fulfill the requirements for a given application, so that final selection is guided by considering the tool life as well as the



cost of material and fabrication. First reasonable factor affecting the tool life is adhesion wear [1]. The high-carbon, high-chromium cold work tool steel, designated as group D (Deep Hardening) steel in the AISI classification system, are the most highly alloyed cold-work steel. The high-carbon, high-chromium cold work D6 tool steel has extremely high wear and abrasion resistance. Again, the high alloy content provides excellent hardenability and good dimensional stability. D6 tool steel contributes approximately 21% of total tool steel application. Although the high abrasion resistance of the D6 tool steels is desirable for cold-work applications, the machining and grinding operations during manufacturing of finished dies and molds are difficult.

### III. LITERATURE REVIEW

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. Moreover, the retained austenite-to-martensite transformation provides dimensional instability. Recent studies have indicated that cryogenic treatment is an essential supplementary treatment, which is performed on products after conventional heat-treatment in order to increase their wear resistance in some materials and to produce dimensional stability in others. The cryogenic treatment is conducted on tool steels, maraging steel, cast iron, carburized steel, tungsten carbide, polymers and composites. In all of the materials mentioned, the cryogenic treatment increases the wear resistance and subsequently increases the product life. The cryogenic treatment has been used as a finishing process in the past few decades. This process is also being used in aircraft and automobile industries as well as many other areas. Over the last decade, several researchers [1–10] have reported that the Deep Cryogenic Treatment (DCT) considerably improves the wear resistance (WR) of AISI D6 tool steels than those obtained either by Cold Treatment (CT) or by Conventional Heat Treatment (CHT). Also it is found that the considerable reduction in wear rate ( $W_R$ ) and coefficient of friction ( $\mu$ ). In addition, it has also been reported that DCT and multiple tempering after cryogenic treatment enhances the dimensional stability and reduces the residual stresses. These favourable effects increase the service life of the components made of AISI D6 tool steels. 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. In general cryogenic treatment and multiple tempering is still in the dormant level as far as wear rate, wear resistance and coefficient of friction is concerned. This is the main focus of the present work on D6 tool steel.

#### a. Problem Definition

Effects of cryogenic treatment on wear behavior of D6 tool steel and effect of multiple tempering before and after the cryogenic treatment.

#### b. Objectives

Therefore the present investigation is based on the effect of cryogenic treatment on friction and wear behaviour of AISI D6 tool steel and has following objectives,

1. To study the effect of wear test parameters like normal load, velocity of counter disc on friction and wear behaviour of D6 tool steel.
- 2) To study the effect of different heat treatments like hardening, cryogenic treatment and multiple tempering on wear volume, coefficient of friction, wear rate and wear resistance of D6 tool steel.
- 3) To study the relationship between normal loads, velocity of counter disc, coefficient of friction, wear rate and wear resistance.
- 4) To find percentage improvement in wear resistance due to combination of different heat treatments on D6 tool steel.
- 5) It is intended that, this research will be useful in promoting the applications of cryogenic treatment on D6 tool steel.

#### c. Proposed Experimental Work

For improvement of friction and wear behaviour of AISI D6 tool steel, combination of different heat treatments like hardening, multiple tempering, cryogenic treatments are used. In this study hardening temperature 1020°C for one hour, tempering temperature 210°C for two hours, cryogenic temperature -185°C for 36 hours, soft tempering temperature 100°C for one hour were selected. The reduction in wear rate, coefficient of friction; improvement in wear resistance of specimens were assessed by a pin-on-disk wear testing machine. For wear test the disc are made of EN-35 (68 HRC maintained) of surface roughness,  $R_a =$



0.5  $\mu\text{m}$ . Testing conditions were used as follows,

a. Normal Load, FN = 3.1Kg, 5.1Kg b. Sliding speed, V = 1.5, 2.5 m/s

c. Test Duration, T = 60 Min.

Change in hardness of specimens was measurement by using Rockwell Hardness tester. Microstructural characterizations of the differently heat treated specimens have been done by image analyzer software with inverted microscope.

#### IV. RESULTS AND DISCUSSION

##### a. Metallography

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 D6 tool steel. Microstructures were then recorded by image analyzer system as shown in Fig. 4.1. Chromium carbides are divided in two categories viz. small size carbides, maximum dimension less than 1 micron, and big carbides, any larger dimension more than 1 micron.

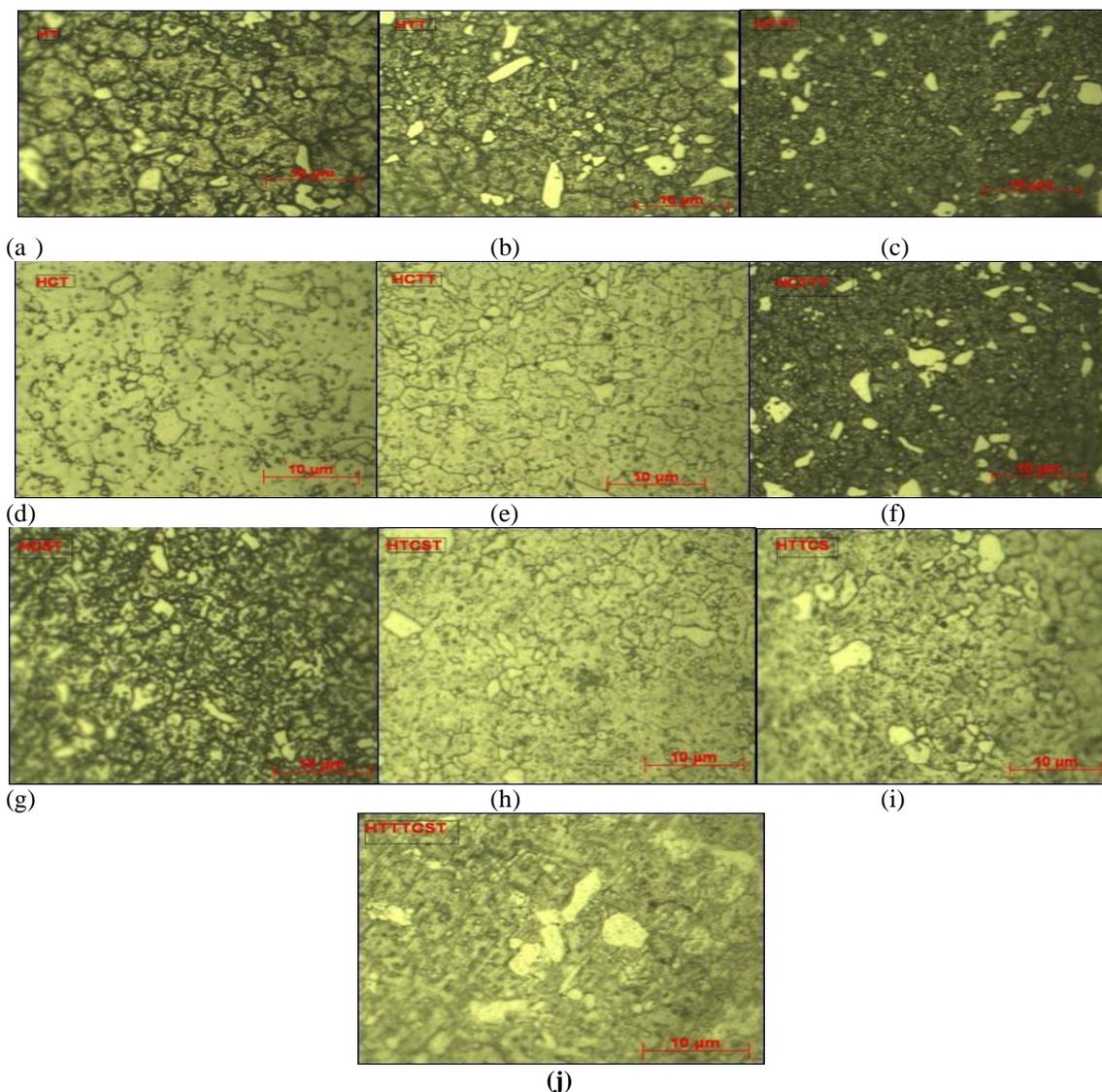


Fig. 4.1 Microstructure of AISI D6 tool steel for different heat treatments samples at 1000x: a) HT, b) HTT, c) HTTT, d) HCT, e) HCTT, f) HCTTT, g) HC, h) HTC, i) HTTC, j) HTTTC



**b. Hardnes Measurement**

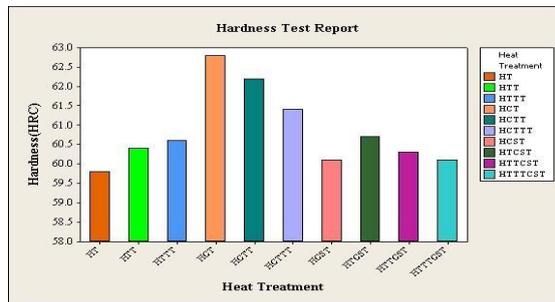


Fig. 4.2 Hardness test report of D6 tool steel for different heat treatment

The results show that the cryogenic treatment increases hardness. From Fig. 4.2, it is observed that for D6 tool steel the hardness of HCT specimens improves approximately 5% as compared to HT specimens. It is also observed that the hardness value for multiple tempering before and after cryo treatment 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 [5]. The obtained results thus infer that increase in hardness of D6 tool steel specimens occurs due to cryo treatment.

**c. Wear Mechanism i. Wear Volume**

The wear volumes of the D6 tool steel versus sliding time are presented in Fig. 4.3 - 4.6 for different wear test parameters. It is observed that both normal load and sliding velocity affect the wear volume, which attains values between 1.596 mm<sup>3</sup> to 11.365 mm<sup>3</sup>. The wear volume increases with increasing normal load, but wear volume of the D6 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, HCTCST, HCTTCST, HTTT, HTTTTCST, HTT, 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.

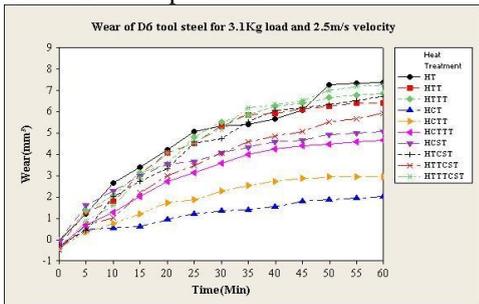


Fig. 4.3 Wear of D6 tool steel for 3.1Kg load and 1.5m/s Velocity for different heat treatment

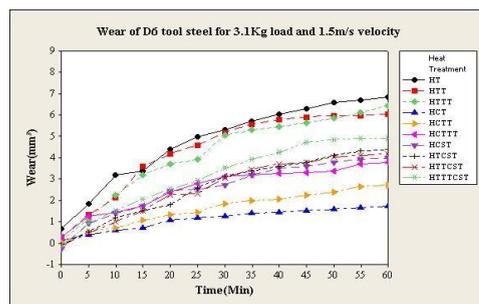


Fig. 4.4 Wear of D6 tool steel for 3.1Kload and 2.5m/s velocity for different heat treatment

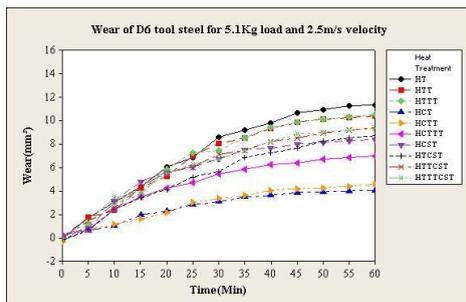


Fig. 4.5 Wear of D6 tool steel for 5.1Kg load and 1.5m/s velocity for different heat treatment

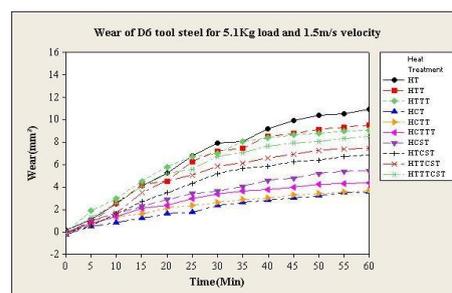


Fig.4.6 Wear of D6 tool steel for 5.1Kg load and 2.5m/s velocity for different heat treatment



**d. Coefficient Of Friction**

The coefficient of friction of D6 tool steel for the different wear conditions for tests carried out and it is observed that, the value of coefficient of friction varies approximately from 0.5324 to 0.7449. The comparison of coefficient of friction between different heat treated specimens is as shown in Fig. 4.7 – 5.0. It is observed that the coefficient of friction decreases with increase in normal load. Wear test report reflect that the coefficient of friction of D6 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 D6 tool steel observed to be fluctuating with increase in sliding speed.

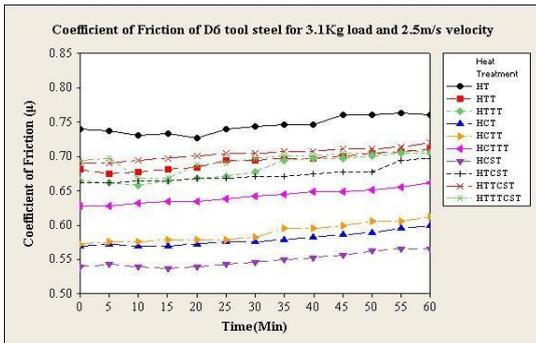


Fig. 4.7 Coefficient of Friction of D6 tool steel for 3.1Kg load and 1.5m/s velocity for different heat treatment

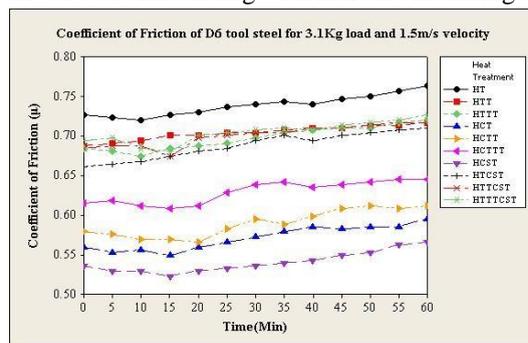


Fig. 4.8 Coefficient of Friction of D6 tool steel for 3.1Kg load and 2.5m/s velocity for different heat treatment

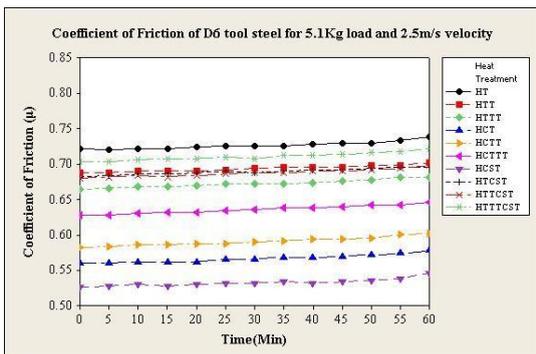


Fig. 4.9 Coefficient of Friction of D6 tool steel for 5.1Kg load and 1.5m/s velocity for different heat treatment

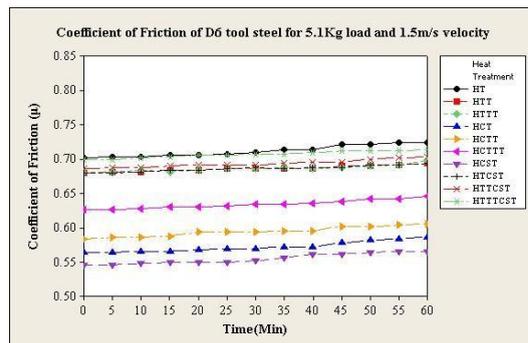


Fig. 5.0 Coefficient of Friction of D6 tool steel for 5.1Kg load and 2.5m/s velocity for different heat treatment

**e. Wear Rate (W<sub>r</sub>)**

The computed values of wear rate for all type of D6 tool steel for specimens tested under normal loads 3.1Kg and 5.1Kg are as shown in Fig. 5.1 - 5.4. It is observed that both normal load and sliding velocity affect the wear rate, which attains values between 0.2955 mm<sup>3</sup>/m to 1.9942 mm<sup>3</sup>/m.

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 and where as there is an increasing wear rate reflected in double and triple tempered D6 tool steel.

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 specimen's 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 HHTTCST 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.

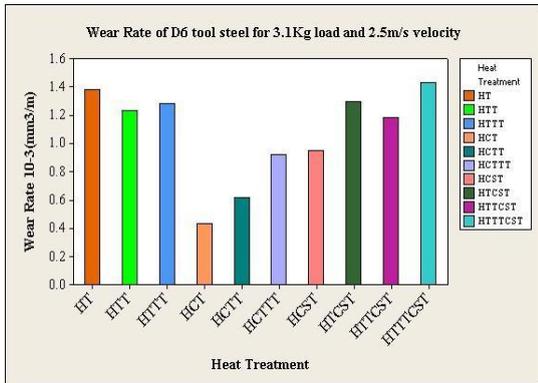


Fig. 5.1 Wear Rate of D6 tool steel for 3.1Kg load and 1.5m/s velocity for different heat treatment

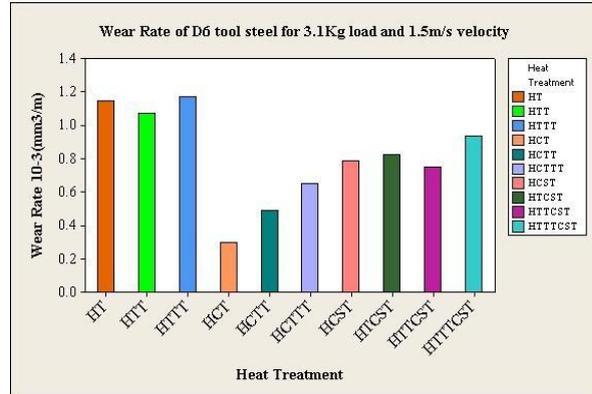


Fig. 5.2 Wear Rate of D6 tool steel for 3.1Kg load and 2.5m/s velocity for different heat treatment

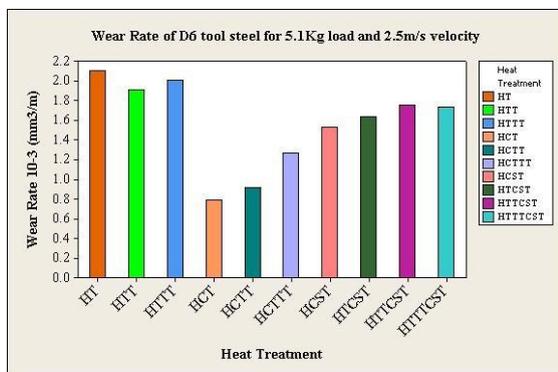


Fig. 5.3 Wear Rate of D6 tool steel for 5.1Kg load and 1.5m/s velocity for different heat treatment

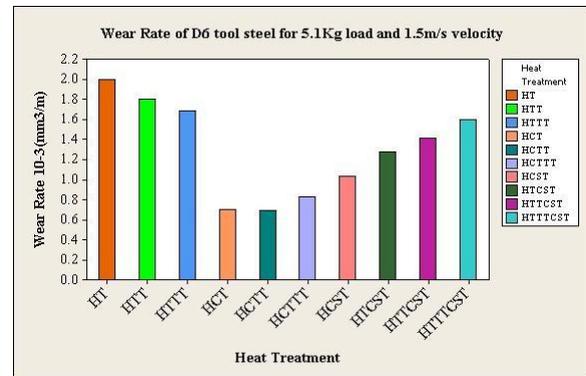


Fig. 5.4 Wear Rate of D6 tool steel for 5.1Kg load and 2.5m/s velocity for different heat treatment

**f. Wear Resistance (Wr)**

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). The calculated wear resistance of different heat treated specimens are as shown in Fig. 5.5 - 5.8. The results obtained in this study, in general, are in agreement with results reported by Dass et al [5]. In this study it is observed that the improvement in wear resistance is significant by cryogenic treatment.

**g. Improvement in wear resistance (α %)**

In order to quantify 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,

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

Where, WR is wear resistance, HCT and HT denote the type of the specimen [5, 7]. 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 shown that 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 D6 tool steel by 140.501% to 248.559%. In addition, the magnitude of for HT and HCT specimens decreases with the increasing normal load and velocity.

The relation between wear properties with the results of microstructure analysis reveals that the improvement in wear resistance is depends on the microstructure analysis generated by different heat treatments irrespective of its dependence on wear test conditions. It can be concluded at this stage that the reduction of retained austenite content, reduction in size of the secondary carbide and distribution of secondary carbides are basic factors responsible for the improvement in wear resistance in HCT specimens; whereas wear resistance property deteriorates with double and triple tempering as evident from Fig. 5.5 - 5.8. In the HT, HTT, HTTT, HCTT, HCTTT, HCST, HTCST, HTTCST, and HTTTCST of D6 tool steel specimens there is growth in secondary carbide size. This contributes in deteriorating the wear resistance of D6 tool steel.



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.

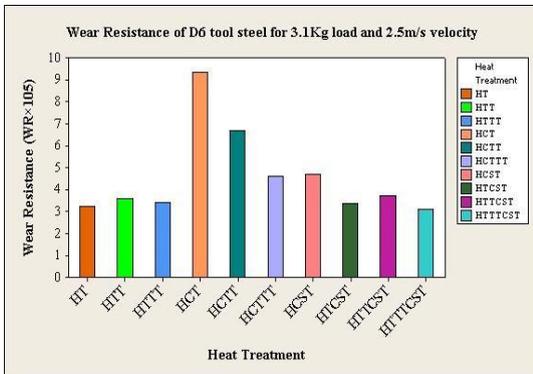


Fig. 5.5 Wear Resistance of D6 tool steel for 3.1Kg velocity for different heat treatment

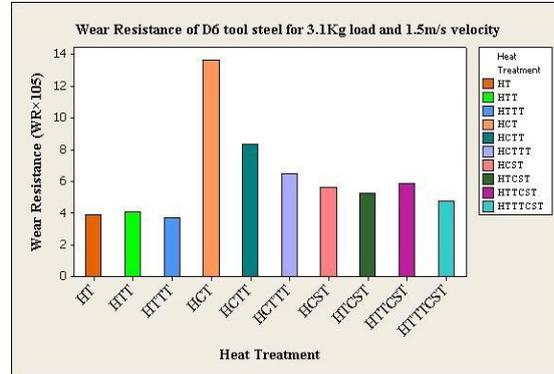


Fig. 5.6 Wear Resistance of D6 tool steel for load and 1.5m/s 3.1Kg load and 2.5m/s velocity for different heat treatment

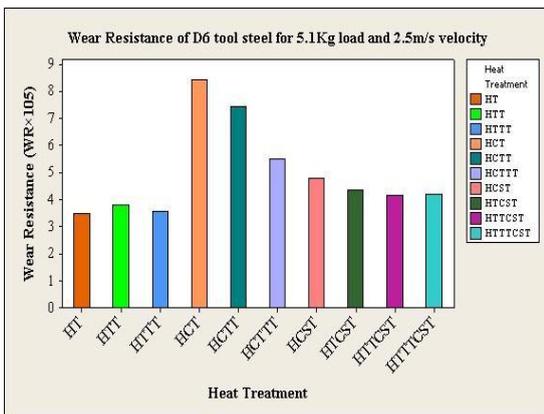


Fig. 5.7 Wear Resistance of D6 tool steel for 5.1Kg load and 1.5m/s velocity for different heat load

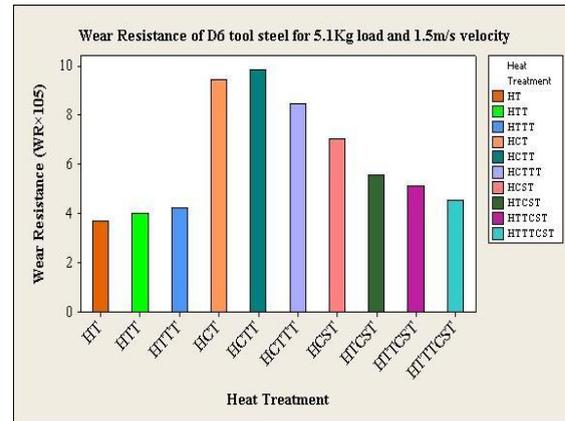


Fig. 5.8 Wear Resistance of D6 tool steel for 5.1Kg load and 2.5m/s velocity for different heat treatment treatment

**h. Statistical Regression Analysis**

Statistical regression analysis is the study of the relationship between two or more variables, used to establish the empirical equation relating input-output parameters, by utilizing least square method. Moreover, it is the most commonly used statistical modeling technique developed based on experimental data. There is difference between predicated and actual values of the response for the same set of independent variables. It is possible to attribute this difference to a set of independent variable and the difference due to random or experimental errors. The technique of analysis of variance (ANOVA) does this. With the help of regression coefficients, we can calculate the correlation coefficients. The square of coefficients, called coefficients of determination R<sup>2</sup>(R-Sq), measures the degree of association of correlation that exists between the variables. The value of R<sup>2</sup>(R-Sq) is the important criteria to decide the validity of regression model.

In general, greater the value of R<sup>2</sup>(R-Sq) better is the fit and more useful the regression equation is as predictive device. If value is 0.85(85%) or more, then relationship established by regression model is acceptable.

**5.5. Regression Analysis for D6 Tool Steel Specimens**

The regression analysis was done on Minitab 16 Software for Wear Rate versus Load, Speed and following regression equations has been developed to predict the wear rate for HCT specimen.

**Regression Analysis: Wear Rate versus Load, Speed**

The regression equation is

$$\text{Wear Rate} = - 0.000449 + 0.000191 \text{ Load} + 0.000111 \text{ Speed}$$

Predictor	Coef	SE Coef	T	P
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Constant	-0.00044934	0.00007108	-6.32	0.100
Load	0.00019063	0.00001222	15.59	0.041
Speed	0.00011075	0.00002445	4.53	0.138

S = 0.00002445 R-Sq = 99.9% R-Sq(adj) = 99.6%

Analysis of Variance

Source	DF	SS	MS	F	P
Regression	2	1.57617E-07	7.88086E-08	131.83	0.061
Residual Error	1	5.97802E-10	5.97802E-10		
Total	3	1.58215E-07			

Source	DF	Seq SS
Load	1	1.45352E-07
Speed	1	1.22656E-08

It was observed that the regression analysis carried out shows that, the value of  $R^2$ (R-Sq) for HCT specimen has greater the value **99.6%**, thus better is the fit and more useful the regression equation is as predictive device.

## VI. CONCLUSION

The present investigation is based on the effect of cryogenic treatment on friction and wear behaviour of AISI D6 tool steel. After conducting the experimental work, following conclusions are drawn from the results.

1. The wear volume and wear rate (WR) increases linearly with increasing normal load for all type of samples, whereas coefficient of friction decreases with increase in normal load.
2. It is observed that at higher velocities, wear rate is enhanced, but the coefficient of friction observed to be fluctuating.
3. It is seen that the largest improvement in wear resistance (WR) is observed in HCT specimens, which is 140% to 248% 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.
4. Wear volume, coefficient of friction, wear rate and wear resistance of the materials depends upon the combination of heat treatments.
5. The Rockwell hardness number of HCT specimens improves approximately 5% by HT specimens. Cryogenically treated specimen shows decreasing hardness from single stage to triple stage tempering.
6. Therefore, among different heat treatments like HT, HTT, HTTT, HCT, HCTT, HCTTT, HCST, HTCST, HTTCST and HHTTCST; the lowest wear volume, coefficient of friction and wear rate is observed in HCT specimens.

As a result of the lower retained austenite, the cryogenic treatment improves the wear resistance and the hardness of the D6 tool steel. This improvement is significant in the deep cryogenic treatment due to more homogenized carbide distribution, the retained austenite elimination and higher chromium carbide percentage in comparison with the conventional heat treatments.

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