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Optimization of Cryogenic Treatment on Wear Behaviour of D6 Tool Steel by Using DOE/RSM

Rahul H.Naravade, U.N.Gujar, R.R.Kharde

Abstract— In this work, the effects of cryogenic treatment on the wear behavior of D6 tool steel were studied. For this purpose, two temperatures were used: -63 ºC as shallow cryogenic temperature and -185 ºC as deep cryogenic temperature. The effects of cryogenic temperature (Shallow and deep), cryogenic time (kept at cryogenic temperature for 20 and 40 h) on the wear behavior of D6 tool steel were studied. Wear tests were performed using a pin-on-disk wear tester to which different loads and different velocities were applied. 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 shallow cryogenic 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. The combination of heat treatment would have to be optimised. For that purpose Design of Experiment (DOE) is performed. The DOE is done with help of statistical tool i.e. minitab 16. Produced optimum runs with help of Response surface methodology (RSM) by Box-Behnken design.

Keywords: - AISI D6 tool steel, cryogenic treatment (CT), wear behaviour, Design of Experiment (DOE), Response Surface Methodology (RSM), retained austenite (_R).

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. In general cryogenic treatment is still in the dormant level as far as metallurgical mechanisms are concerned.

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.

Cryogenic treatment is not, as often mistaken for, a substitute for good heat treatment, but is a supplemental process to conventional heat treatment before tempering. It not only gives dimensional stability to the material, but also improves abrasive and fatigue wear resistance, and increases strength and hardness of the material. The main reason for this improvement in properties are the complete transformation of retained austenite into martensite and the precipitation of fine carbides into the tempered martensitic matrix. [1]

Akhbarizadeh et.al studied the effects of cryogenic treatment on the wear behavior of D6 tool steel. For this purpose, two temperatures were used: -63ºC as shallow cryogenic temperature and -185ºC as deep cryogenic temperature. The effects of cryogenic temperature (Shallow and deep), cryogenic time (kept at cryogenic temperature for 20 hours and 40 hours) and stabilization (kept at room temperature for one week) on the wear behavior of D6 tool steel were studied. Wear tests were performed using a pin-on-disk wear tester. 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. By keeping the samples for a period of one week at room temperature after quenching (stabilization), more retained austenite was transformed into martensite and higher wear resistance and higher hardness were achieved. [4]

Amini et.al studied the effect of cryogenic treatments on the wear behavior of 80CrMo12 5 tool steel was studied using two different temperatures: -80 ºC as the shallow cryogenic temperature and -196 ºC as the deep cryogenic temperature. The cryogenic treatments increase the wear resistance and harnesses. Shallow cryogenic treatment (SCT) decreased the retained austenite by 6% for the SCT24 sample. In deep cryogenic treatment (DCT), as well as the retained the hardness and wear resistance of the DCT samples depend on the holding time, showing a maximum at 48 h holding time. The decrease beyond 48 h was attributed to the decrease in the carbides homogeneous distribution. [5]

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II. EXPERIMENTAL PROCEDURE

A. Material for Disc

The disc of diameter 160mm and thickness 8 mm is selected as the rotating counter surface. The tungsten carbide coated EN 31 steel is to be select. The disc having four equidistance holes at 145 mm pitch circle diameter as fig.2.1.

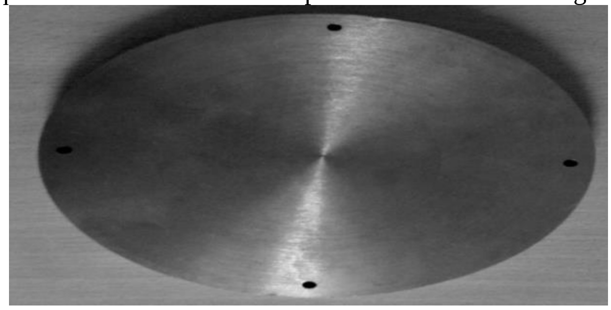

Fig.2.1. Disc

B. Material for Pin

The investigation has to be conduct with samples of AISI D6 tool steel. AISI D6 tool steel is also designated as T30405 by Unified Numbering System. The round bars of diameter 10mm to be select for this experimentation as shown in fig 2.2. and height 30mm. For hardness test specimens were prepared of diameter 10mm and height 10mm. The chemical composition of specimen (diameter 8mm; height 15mm) as analyzed by vacuum spectrometer is given in Table.2.1.

Table 2.1.AISI D6 steel chemical composition

C	Si	Mn	Cr	Mo	Ni	V	W	Co	Ti	S
2.10	0.35	0.35	12.00	-	-	-	0.70	-	-	-

C. Principle alloying element in Pin

Principle allowing elements and their effect on material properties are summarized as below:

- Carbon (C)

Carbon is by far the most important alloying element for the hardening properties of all steel types, including tool steels.

- Silicon (Si)

In general silicon improves resistance against softening of martensite, and displaces tempered martensite embrittlement to higher temperatures.

- Manganese (Mn)

It increases the depth of hardening; Manganese containing steels can be hardened in oil, even though manganese augments the retained austenite content.

- Chromium (Cr)

Chromium forms carbides and increases hardness, wear resistance, corrosion resistance and tempering resistance.

- Cobalt (Co)

Cobalt is a hard ferromagnetic, silver-white, hard, lustrous, brittle element. Addition of nickel increases the strength of the steel by entering into solid solution in ferrite. It is used in low alloy steels to increase toughness and harden ability. Presence of nickel reduces lattice distortion and cracking during quenching.

- Tungsten (W)

Tungsten and molybdenum exhibit similar effects. Addition of both elements results in grain refinement.

- Vanadium (V)

Originally vanadium was used as a scavenger to remove slag, impurities, and to reduce nitrogen dissolved in the matrix and to act as de-oxidant during the production of the steel. It is found that vanadium formed very hard and thermally stable carbides usually as isolated particles. These carbides improve the resistance against abrasive wear and provide very good cutting performance.

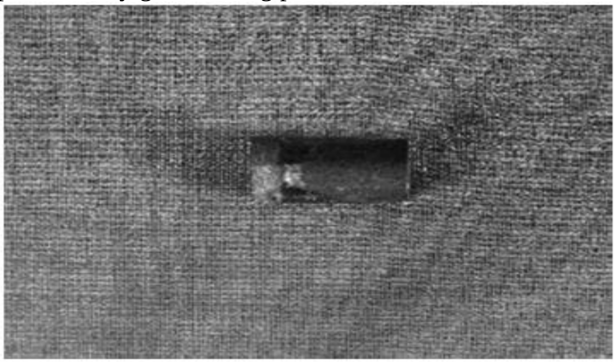

Fig.2.2. pin.

D. Treatments

The material selected for this work was given various treatments and treatment combinations.

- Hardening

The first step in the heat treatment of AISI D6 tool steel was hardening. The purpose of hardening is to harden steel to increase the wear resistance, cutting ability. Hardening of AISI D6 tool steel was done in the tubular furnace (3.5KW, 230V AC, 15A, 1200±10°C) at a temperature of 950-1024°C for 1 Hour. During hardening process, inert gas Argon to be supply in tubular furnace to avoid oxidation. Harden AISI D6 tool steel was follow air cooling which provides great benefit of minimizing distortion and dimensional changes.

- Tempering

The process which consists of heating the hardened components to a temperature between 100°C ato700°C, holding at this temperature for specific period and cooling to room temperature, usually by air is called as “tempering”. For D6 tool steel tempering within range of 210°C for 2 hours.

Purpose of tempering is as follows

1. To relieve the internal stresses developed due to rapid cooling of steels after hardening process and due to volume changes occurring in the austenite to martensite transformation, to reduce brittleness,
2. To reduce hardness and to increase ductility and toughness,
3. To eliminate retained austenite (_R) [12].

- Cryogenic treatment

The subzero treatment means the treatment carried out below 0°C for predetermined soaking time. As per the level of temperature chosen below subzero and prevailing understanding about the subzero treatments, the following classification/definition is proposed

1. Deep Subzero Treatment (DT) is meant for temperature regime from -80°C to -150°C.
2. Cryogenic Subzero Treatment (CT) is meant for temperature regime below -150°C. [3]

Table.2.2. Specification of Tribometer (TR-20)

Specifications of pin on disc Tribometer (TR-20) Make	Ducom Ltd, Bangalore.
Pin Size	3 to 12 mm diagonal
Disc Size	160 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	200 N Maximum

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Friction Force	0-200 N, digital readout, recorder output
Wear Measurement Range	4 mm, digital readout, and recorder output
Power	230 V, 15A, 1 Phase, 50 Hz

Cryoprocessor is capable of maintaining shallow subzero treatment, deep subzero treatment and cryogenic subzero treatment temperatures for any length of time through software based program which control the flow of liquid nitrogen via solenoid valve as per the demand of programming logic controller. To understand the effects of cryogenic processing it is essential that one 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. The Fig shows cryogenic cycle as given.

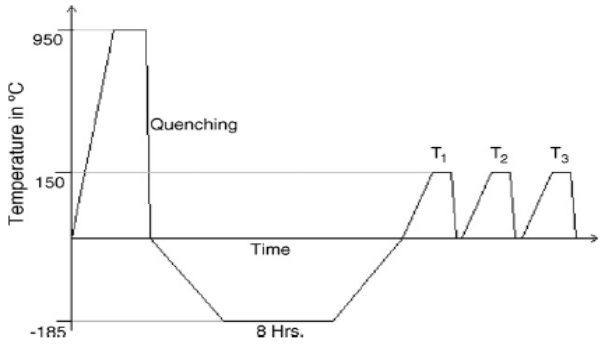

Fig.2.3. Processing routes of the cryogenic treatment.

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 [4].

E. *Evaluation of wear behavior*

Fig. 2.4. Pin-on-disc wear testing machine

Dry sliding wear tests were carried out on this computerized pin-on-disc wear testing machine (DUCOM: TR 20LE, India). The specifications of pin-on-disc tribometer are shown in Table no. 2.2. Specimens of 10mm diameter and 30mm length clamp in a holder and held against the rotating counter disc made of WC-coated EN-31 (68 HRC).The faces of the pin specimens to be polish and clean in acetone in an ultrasonic cleaner.[7]

During testing, pin of D-6 steel specimen to be keep stationary while the circular disc rotates. The test sample was clamped in a holder and held against the rotating steel disc as shown in fig. 2.4. schematically. The wear rate is reported as an average of wear rates of three samples. [8]

F. *Hardness measurement*

For the hardness measurement, Rockwell hardness tester was to be use. For D-6 steel material HRC scale was used. Minor loads first apply to seat the specimen. The major load then applies for HRC scale for duration of 30 s. The depth of resistance to indentation was automatically recorded on the dial gauge. [9]

G. *Cryogenic equipment*

Fig.2.5. is a schematic representation of cryogenic equipment. It comprises an insulated box (cryo box), One motor with circulating fan, one thermocouple to measure the cryogenic temperature inside the box connected to a temperature controller and programmer, a liquid nitrogen tank and solenoid valve for gas inlet.[10]

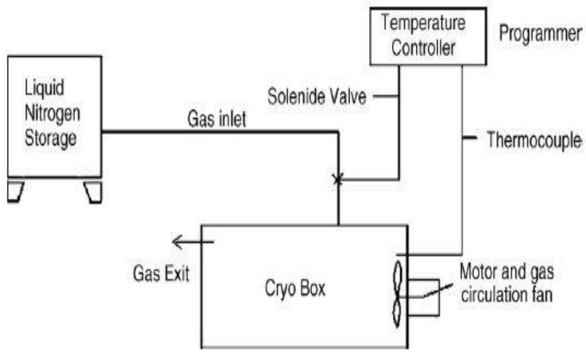

Fig.2.6. Block diagram explaining operating principle of the cryogenic equipment.

III. DEVELOPMENT OF SYSTEM FOR D.O.E.

A. *Design of Experiment*

It is methodology based on statistics and other discipline for arriving at an efficient and effective planning of experiments with a view to obtain valid conclusion from the analysis of experimental data. Design of experiments determines the pattern of observations to be made with a minimum of experimental efforts. To be specific Design of experiments (DOE) offers a systematic approach to study the effects of multiple variables / factors on products / process performance by providing a structural set of analysis in a design matrix.

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More specifically, the use of orthogonal Arrays (OA) for DOE provides an efficient and effective method for determining the most significant factors and interactions in a given design problem.

B. Principles of Design of Experiments

Design of Experiment (DOE) is a statistical approach that could clearly show how much several parameters of a system as well as their interactions are important on the plant output and how these parameters could affect the objective function. DOE Method is capable to investigate simultaneously the effects of multiple parameters on an output variable (response). To illustrate statistically reasonable conclusions from the experiment, it is necessary to integrate an efficient statistical method into the methodology of experimental design. In the context of DOE in designing, one may encounter two types of plant variables or factors: qualitative and quantitative factors. For quantitative factors, the range of settings must be decided by designer. For instance, pressure, temperature or heat transfer surface are examples of quantitative factors. Qualitative factors are discrete in nature. For example, types of materials, nature of heating source, and types of equipments are examples of qualitative factors. A factor may take different levels, depending on the character of the factor- quantitative or qualitative. In general, compared to a quantitative factor, more levels are required by a qualitative factor. "Level" in this chapter refers to a specified value or setting of the factor that would be examined in the plant experiment. [14]

C. Minitab 16 Software for DOE

Meet Minitab introduces you to the most commonly used features in Minitab. Throughout the book, you use functions, create graphs, and generate statistics. The contents of Meet Minitab relate to the actions you need to perform in your own Minitab sessions. You use a sampling of Minitab's features to see the range of features and statistics that Minitab provides. Most statistical analyses require a series of steps, often directed by background knowledge or by the subject area you are investigating. Design of experiments (DOE) capabilities provides a method for simultaneously investigating the effects of multiple variables on an output variable (response). These experiments consist of a series of runs, or tests, in which purposeful changes are made to input variables or factors, and data are collected at each run. Quality professionals use DOE to identify the process conditions and product components that influence quality and then determine the input variable (factor) settings that maximize results. Minitab offers four types of designed experiments: factorial, response surface, mixture, and Taguchi (robust). The steps you follow in Minitab to create, analyze, and graph an experimental design are similar for all design types. After you conduct the experiment and enter the results, Minitab provides several analytical and graphing tools to help you understand the results. While this chapter demonstrates the typical steps for creating and analyzing a factorial design, you can apply these steps to any design you create in Minitab.

Features of Minitab DOE commands include:

- Catalogs of experimental designs from which you can choose, to make creating a design easier
- Automatic creation and storage of your design once you have specified its properties
- Ability to display and store diagnostic statistics, to help you interpret the results
- Graphs that assist you in interpreting and presenting the results

We want to further improve the amount of time it takes to get orders to customers from the Western shipping center. After evaluating many potentially important factors, you decide to investigate two factors that may decrease the time to prepare an order for shipment: the order processing system and packing procedure. The Western center is experimenting with a new order processing system and you want to determine if it will speed up order preparation. The center also has two different packing procedures and we want to investigate which one is more efficient. You decide to conduct a factorial experiment to find out which combination of factors results in the shortest time to prepare an order for shipment. The results of this experiment will help you make decisions about the order processing system and packing procedures used in the shipping center.

D. DOE by Response Surface Methodology (RSM)

In this section the basics of Response Surface Methodology (RSM) and how they work together with Design of Experiments methods is explained.

In many applications dependence between the input parameters and the output parameters of a complex system is unpredictable. RSM comprises a group of statistical techniques for empirical model building and model exploitation.

A second order model is generally used to approximate the response once it is realized that the experiment is close to the optimum response region where a first order model is no longer adequate. The second order model is usually sufficient for the optimum region, as third order and higher effects are seldom important.

$$y = \beta_0+\beta_1x_1+ \beta_1x_1+ \beta_2x_2+ \beta_kx_k+ \beta_{11}x_1^2+\ldots\ldots+\beta_{kk}x_k^2+\varepsilon \qquad (3.1)$$

The model contains regression parameters that include coefficients for main effects (β_1, $\beta_{2,\ldots}$ β_k), coefficients for quadratic main effects (β_{11}, $\beta_{22,\ldots}$ β_{kk}) and coefficients for two factor interaction effects (β_{11}, $\beta_{12,\ldots}$ $\beta_{k-1,k}$). A full factorial design with all factors at three levels would provide estimation of all the required regression parameters. However, full factorial three level designs are expensive to use as the number of runs increases rapidly with the number of factors. For example, a three factor full factorial design with each factor at three levels would require 2^3=27 runs while a design with four factors would require 3^4=81runs. Additionally, these designs will estimate a number of higher order effects which are usually not of much importance to the experimenter. Therefore, for the purpose of analysis of response surfaces, special designs are used that help the experimenter fit the second order model to the response with the use of a minimum number of runs.

For both input (x) and output parameters (y), transformations can be specified, so additional knowledge about the system behavior can be included. It should be noticed that a transformation for input parameters of the response surface should be used in the DOE step. Additionally, it is also possible to automatically select the best out of a given set of transformation functions for a given set of sample points. This is done by computing several response surfaces where any possible variation of available transformations is applied to all input parameters. The selection criteria for the best set of transformations are determined by the minimum fit error of the sample points calculated by the total sum of squares of the residuals.

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For all these surfaces the fit error of the sample points (the sum of squares) is used to select the best combination of transformations.

IV. RESULT AND DISCUSSION

The ingredients effects on three friction characteristics including load, speed and track radius were studied by 2^k DOE. Concerning the confounding of two- ingredient interaction effects and main effects, response surface methodology was employed to optimize given parameter.

Table.4.1.Input variable.

	Min. limit	Max. limit
Load (kg)	1	4
Speed, (RPM)	200	1000
Track radius(mm)	40	120

Result On Minitab 16 for RSM

Box-Behnken Design

Factors: 3 Replicates: 1
Base runs: 15 Total runs: 15
Base blocks: 1 Total blocks: 1
Center points: 3

Table 4.2.Design Table

Run	Blk	A	B	C
1	1	+	-	0
2	1	0	0	0
3	1	-	+	0
4	1	-	-	0
5	1	0	-	-
6	1	0	-	+
7	1	-	0	-
8	1	0	+	-
9	1	0	+	+
10	1	0	0	0
11	1	0	0	0
12	1	+	+	0
13	1	+	0	-
14	1	-	0	+
15	1	+	0	+

Optimal Design: LOAD, SPEED, TRACK LENGH
Response surface design selected according to D-optimality
Number of candidate design points: 15
Number of design points in optimal design: 10
Model terms: A, B, C, AA, BB, CC, AB, AC, BC
Initial design generated by Sequential method
Initial design improved by Exchange method
Number of design points exchanged is 1
Optimal Design
Row number of selected design points: 8, 6, 12, 7, 14, 3, 1, 5, 2, 4
Condition number: 782.855
D-optimality (determinant of XTX):4.15520E+48
A-optimality (trace of inv (XTX)): 4.3358
G-optimality (average leverage/max leverage):1
V-optimality (average leverage):1
Maximum leverage: 1

A three-level factor consideration of saturated fractional factorial experiment design with resolution .The experiment design matrix, which was generated by the software of MinitabRel.16, was shown in Table.5.1. The low and high levels were design at ‘‘-1’’ and ‘‘+1’’, respectively.

Table.4.3. Optimum runs by RSM.

	C1	C2	C3	C4	C5	C6	C7
	StdOrder	RunOrder	PtType	Blocks	LOAD	SPEED	TRACK LENGH
1	10	8	2	1	2.5	1000	40
2	11	6	2	1	2.5	200	120
3	4	12	2	1	4.0	1000	80
4	5	7	2	1	1.0	600	40
5	7	14	2	1	1.0	600	120
6	3	3	2	1	1.0	1000	80
7	2	1	2	1	4.0	200	80
8	9	5	2	1	2.5	200	40
9	15	2	0	1	2.5	600	80
10	1	4	2	1	1.0	200	80

The above runs performed before heat treatment and after heat treatment of each combination of heat treatments as CHT, HCT, HCTT, HCTTT.

V. CONCLUSION

In the present work based on the characterization of cryotreated materials viz. D6 steel and selecting optimum parameter, the following conclusions are drawn:

Wear is main cause of material wastage, is an important problem associated with industrial component. The D6 tool steel is high carbon high chromium (HCHCr) tool steel. This is promising tribological material with application of measuring tools, blanking dies, forming dies, coining dies, long punches, forming rolls, edging rolls, master tools, extrusion dies, drawing dies, etc

The configuration of pin material properties of D6 tool steel is studied and selected ranges for heat treatment temperature from Tool Steel handbook as and also selected disc material which should be higher than pin material.

The effect of different combination heat treatment like hardening within range of 950 C-1024 C for 1 hour, tempering within range of 210° C for 2 hours, Cryogenic treatment at -185 C for within range of 20 to 40 hours are will be carried out. Wear of pin with different load, speed and track radius are selected.

The speed, load and track radius are selected with lower and higher level .The optimum combination of all variables (speed, load, track radius) are selected by Response surface methodology (RSM) technique .This results shows optimum 10 runs before heat treatment and 10 runs for each combination of heat treatment as HCT, HCTT, HCTTT.

For study of variable and relation between wear rate and variable will going to plot by non-linear regression analysis.

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