

The Effect of Cryogenic Treatment on the Hardness, Friction and Wear Resistance of Austenitic Ductile Iron Type D3 Tool Steel

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Abstract: Investigations carried out in the recent few decades reveal the advantages of cryogenic treatment as one of the promising techniques to enhance wear resistance in certain tool steels. Thus the cryogenic treatment has significant influence on the tribological performance of tool steels. It is a one time permanent treatment process affecting the entire section of the part, unlike coatings. Enhancing the wear resistance and service life of the steel tools subjected to rubbing condition is of important concern. Literature provides information about the investigations performed on some high-speed steels which reveal remarkable improvement in wear resistance from 92% to 817%. Furthermore, the studies conducted on conventional D3 tool steel reveals the betterment of certain tribo-mechanical properties such as hardness and wear resistance. In the present investigation the effect of cryogenic treatment on austenitic ductile iron type D3 tool steel is studied by sliding the test specimen against the same mating material. The study reveals increase in hardness, reduction in friction coefficient and enhancement in wear resistance.

Keywords: Austenitic ductile iron, Cryogenic treatment, Friction, Wear resistance, Vickers hardness, D3 tool steel, Pin-on-disc tribometer.

I. INTRODUCTION

Techniques like heat treatment and coatings are commonly used to improve the wear resistance of some tool steel components [11-15]. The perfect combination of alloying elements and the domain of heat treatment processes confer materials with excellent hardness and wear resistance properties allied to good toughness [15]. As a result of technological advance a great variety of HSS tools are actually available, including coated and powder metallurgy tools. However, cryogenic treatment has emerged as one of the fruitful techniques for the improvement of tribo-mechanical properties of materials [1,2,5,6].

Investigations carried out over the recent four decades have shown interests in low temperature effects and have been demonstrated particularly during heat treatment cycles of tool steels. Initial studies conducted by Barron on various tool steels show a remarkable improvement in tool life and wear resistance. Barron [2] investigated the effect of cryogenic treatment for several materials including the M2 high speed steel at 84°C. The soaking temperature is maintained at 84 °C for 24 hours. A significant improvement in wear resistance of cryo-treated steels is observed in sliding abrasion tests when compared to tool steels that are conventionally heat treated, quenched and tempered. The reduction in temperature of the cryogenic treatment further to -196°C results in further enhancement in the wear resistance. Literature data indicates that the lives of tools and other steel components increase significantly after being submitted to subzero temperatures. The results are found to be good depending on the application. Improvement in the performance of tool steels from 92% to 817% is observed due to the cryogenic treatment at -196°C.

Barron [1] attributes the improvement in the wear resistance of tool steels to another mechanism besides the transformation of the retained austenite into martensite. The study verifies that tool steels submitted to conventional heat treatment presented only a small amount of retained austenite. Tool steels submitted to cryogenic treatment show even better performance during machining. Barron, thus, attributes this achievement

to transformation of retained austenite to martensite and the presence of both hard and small carbide particles that are well distributed among the larger carbide particles within the martensite matrix.

Paulin [3], in his work, verified the presence of fine precipitated carbide particles and their importance related to the material properties. The precipitated carbides reduce internal tension of the martensite and also micro cracks susceptibility is minimized. The uniform distribution of fine carbides of high hardness enhances the wear resistance. Study conducted by Huang and associates [5] also confirms that the cryogenic treatment not only facilitates the carbide formation, it can also make the carbide distribution more homogeneous.

The study conducted by Yun [4] verifies changes in the micro-structure of M2 high speed steel when the material is subjected to different cycles of cryogenic treatment at -196 °C. The results attribute to the transformation of retained austenite into martensite and precipitation of ultra-fine carbides.

The effect of deep cryogenic treatment (DCT) on the properties of some tool steels is studied by Molinari and others [6]. The investigation is carried out with both field tests and laboratory tests on real tools. 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%. The laboratory investigation on an AISI M2 and an AISI H13 steel confirms the effectiveness of DCT followed by conventional heat treatment in increasing the wear resistance and toughness.

The mechanical properties such as hardness and wear resistance of the tool and die steel samples treated by conventional tempering and cryogenic quenching with tempering have been evaluated employing Vickers indentation and sliding wear techniques, respectively. The investigation demonstrates deep cryogenic treatment leads to considerable micro-structural changes which results in enhanced tribo-mechanical properties [7].

Study conducted by Dhokey and Nirbhavane [8] describes the effect multiple tempering after cryogenic treatment for D3 tool steel. The hardness, micro structure and wear loss are studied for different kinds of treatments. The treatments are like HT (Hardening and Tempering), HC (Hardening and cryo-treatment), HCT, HCTT, HCTTT (Hardening, cryogenic treatment, tempering and multiple tempering). Wear test is conducted by holding D3 sample pin against the SAE 52100 steel disc of pin on disc tribometer. The study unveils decrease in hardness with multiple tempering of cryo-treated D3 tool steel. The study also reveals the reduction in wear resistance with multiple tempering of the cryo-treated D3 tool steel.

The instrumentation for the measurement of tribo-mechanical properties are of many types and the methods of evaluation of the properties are also of various kinds. There are different forms of tribometers used for the evaluation of tribo-mechanical properties and one of the types, advanced reciprocating tribometer, is studied and published by the author as a co-worker [10]. In the current study pin-on-disc tribometer, conforming with ASTM standards, is used for the evaluation of tribo-mechanical properties of the materials.

II. AUSTENITIC DUCTILE IRON TYPE D3 TOOL STEEL

2.1 COMPOSITION

In the present work, unlike the D3 tool steel used in the study [8], the material selected for the study is austenitic ductile iron (ADI) type D3 tool steel. The composition of austenitic ductile iron is 2.6% of carbon, 2.5-3.5% of chromium, maximum of 1.0% of manganese, 28.0-32.0% of Ni, maximum of 0.08% of phosphorus, maximum of 1.0-2.8% of silicon and iron being the balance percentage of the structure.

2.2 IMPORTANCE OF AUSTENITIC DUCTILE IRON TYPE TOOL STEELS

The study is intended to determine the tribo-mechanical properties of cryo-treated D3 Tool Steel. Since the Pin-On-Disc tribometer could be operated at higher linear speeds when compared to reciprocating tribometer, in the present study an improved version of Pin-On-Disc tribometer with modified pin holding attachment [Fig. 1.] is used to evaluate friction and wear of D3 tool steel. The evaluation of the friction and wear properties is significant and must be done with well defined procedure. Hence The samples are prepared to conform the Pin-On-Disc tribometer specifications.

III. INTRODUCTION TO CRYOGENIC TREATMENT AND ITS INFLUENCE ON SOME TOOL STEELS

Cryogenic treatment process is not a supplement process for heat treatment. Rather, it is an extension of heat treatment process which would be helpful in obtaining improvement in some of the tribo-mechanical properties for some of the materials. The subzero treatment on metals has been extensively employed for many

decades for various applications like stabilizing dimensions of precision machined parts and gauges, removal of internal stresses, improving tool life of cutting tools etc.

When metals are gradually cooled to cryogenic temperatures, soaked for a prolonged period and allowed to warm to room temperature at a predetermined rate, the lattice structure changes due to stresses being relieved during cryogenic treatment cycle. In the case of ferrous metals, the soft, ductile, FCC structured austenite gets converted to strong and harder BCC structured martensite. Apart from this change, a wide precipitation of newly formed carbides into the hard martensite structure induces a dense lattice structure [1,3].

Earlier studies have revealed the fact of newly precipitated carbide particles are largely responsible for the increase in wear resistance characteristics by the process, due to, a denser, uniform and fine micro structure. This results in larger surface area of contact between the tool and metal which reduces friction, heat and wear. Obvious changes have been found in the improvement of tool life of high-speed tool steels.

IV. SAMPLE PREPARATION AND THE PROCESS OF CRYOGENIC TREATMENT

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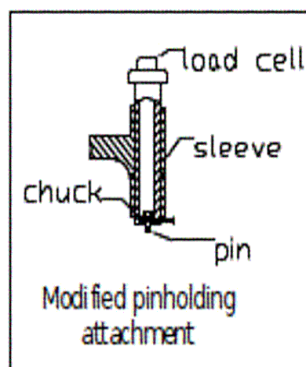


Figure 1. Modified pin holding attachment

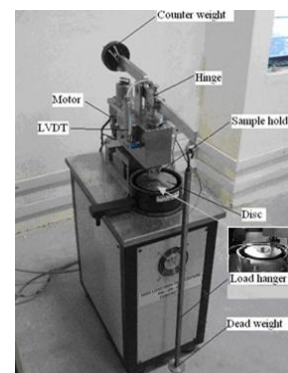


Figure 2. Pin-On-Disc Tribometer

4.2 PREPARATION OF MATERIAL SAMPLES FOR TRIBOLOGY TESTS

It is of interest to test some of the mechanical properties of the selected material. In order to conduct the wear resistance test on the wear testing rig: A disc of D3 tool steel of diameter 110mm having a thickness of 8.1 mm is required. The surface finish of 0.24 micrometer is required for the disc which is accomplished by surface grinding operation. The disc has a central hole of 8.5mm in diameter and an eccentric hole of 4mm in diameter at a distance 11.25mm from the center of the disc. The pins required for wear testing are obtained by cutting and finishing a rod of length 300mm and diameter of 6mm. The length of each pin is 25mm. Square slabs of dimension 50mmx50mmx10mm are fabricated for hardness tests.

4.3 CRYOGENIC TREATMENT FOR THE TEST SAMPLES

Fabricated samples are categorized into two different batches. One batch is taken through a cycle of cryogenic treatment. The hardness specimen, specimen pins and specimen discs are placed inside the cryogenic system Fig. 4. The cryogenic processor system is provided with a container in which liquid nitrogen gets filled. Initially, during the cryo-treatment, the container temperature is maintained at room temperature of 300K. The temperature of the system is gradually reduced to the soaking temperature of 98 K with a cooling rate of 0.417 K/min. This takes about 8 hours to attain the temperature 98 K. The cooling is controlled by a data acquisition system which regulates the liquid flow through the solenoid valve. If the rate of fall in temperature is quick compared to the preset value then the the flow of the liquid nitrogen is stopped so that the temperature drop is maintained at preset value. The amount of liquid required for attaining this temperature is about 250 litre.

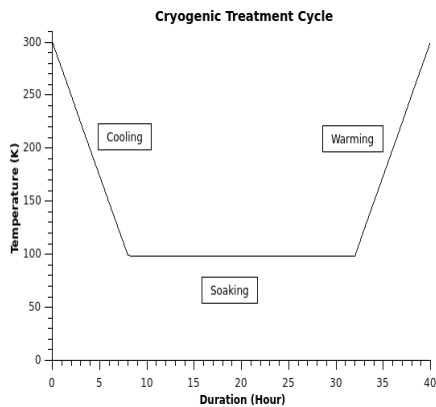


Figure.3. Cryogenic treatment cycle

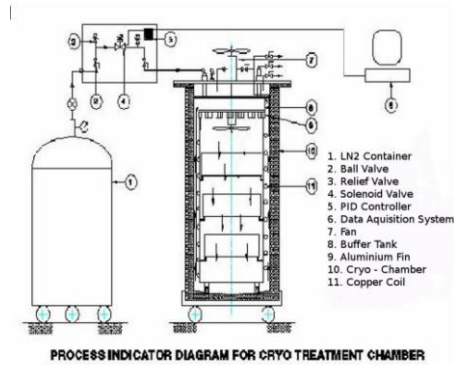


Figure. 4. Schematic Diagram of cryo-processor

Once the specimens are cooled to a temperature of 98 K the temperature is maintained constant for a period of 24 hours [9]. In order to hold the temperature constant for a period of 24 hours it takes about 450 litre of liquid nitrogen. The beneficial changes of the cryogenic treatment occur during this phase of the treatment. After the 24 hours the system is allowed to gradually heat to the room temperature. The warming at rate 2.4 min\K and hence it takes 8 hours for the specimens to attain the room temperature 300K Fig. 3.

V. EXPERIMENTS

The Untreated and cryo-treated samples are fabricated as per the required standards for the Vickers hardness test and conforming to the specifications of the Pin-On-Disc tribometer designed as per ASTM standards.

5.1 DETERMINATION OF VICKERS HARDNESS FOR D3 TOOL STEEL

Hardness measurement is done for both untreated and cryo-treated test specimen. Vickers hardness measurement technique is used to determine the hardness of the selected material specimens. The hardness is determined by taking average of six readings. The average Vickers pyramid number (HV) or Diamond pyramid hardness (DPH) determined using Vickers indentation method is given in the Table. 1.

Sample	F (kg wt)	d(mm)	HV
Untreated	20	0.411	220
Cryo-treated	20	0.379	258

Table. 1. HV – Untreated and Cryo-treated austenitic ductile iron type D3 tool steel.

5.2 FRICTION AND WEAR TESTS

Specifications of the Pin-On-Disc tribometer used for the tribo-mechanical tests are as described below. The loading capacity is from 5N to 250N having the resolution of 1N. The maximum friction force of 100N can be measured with 1N resolution Fig. 2. The resolution of linear variable differential transformer (LVDT) is 1 micron. The Pin-On-Disc tribometer is calibrated using standard specimen for which the tribo-mechanical properties are well defined. Experiments are conducted by sliding D3 pin against D3 Disc, for both untreated and cryo-treated samples, using Pin-On-Disc Tribometer. The idea of making the pin and disc of same material is to study the effect of cryogenic treatment on the material when it is under rubbing condition with identical material. Friction and wear tests were first conducted for untreated D3 pin sliding against untreated D3 disc. In the later part of the experiments friction and wear tests were conducted for the cryo-treated D3 pin sliding against cryo-treated D3 disc. Friction and LVDT Displacement were acquired using data acquisition system for a constant load of 40N and keeping the sliding distance same 1000m. The experiments were conducted for two different sliding speeds of the D3 disc 1m/s and 5m/s. During the tribo-mechanical tests wear debris is not removed from the surface of the disc to simulate practical real time sliding environment.

VI. RESULTS AND DISCUSSION

Vickers hardness measurements are done with six trials and an average value is obtained. The average of the HV is 221 before cryo-treatment. The average of the HV is 258 after cryo-treatment. The results show enhancement by 17% after cryo-treatment. It is evident from the present study that there is appreciable increase in the hardness of the material as a result of cryo-treatment. This is attributed to the transformation from retained austenite to martensite and the formation of carbide particles.

The comparison of co-efficient of friction between untreated and cryo-treated samples conclude the betterment of the performance of the material with lower friction after cryo-treatment. The plots in Fig. 5. reveal the lowering of friction after cryo-treatment, for the sliding speeds 1m/s and 5m/s.

The fluctuations in the values of co-efficient of friction were observed during the experimentation and is due to the lumps of wear particles formed during the three body abrasive wear. It is evident from the visual inspection and the plots that the wear particle size is comparatively high in case of lower speed 1m/s than higher speed 5 m/s

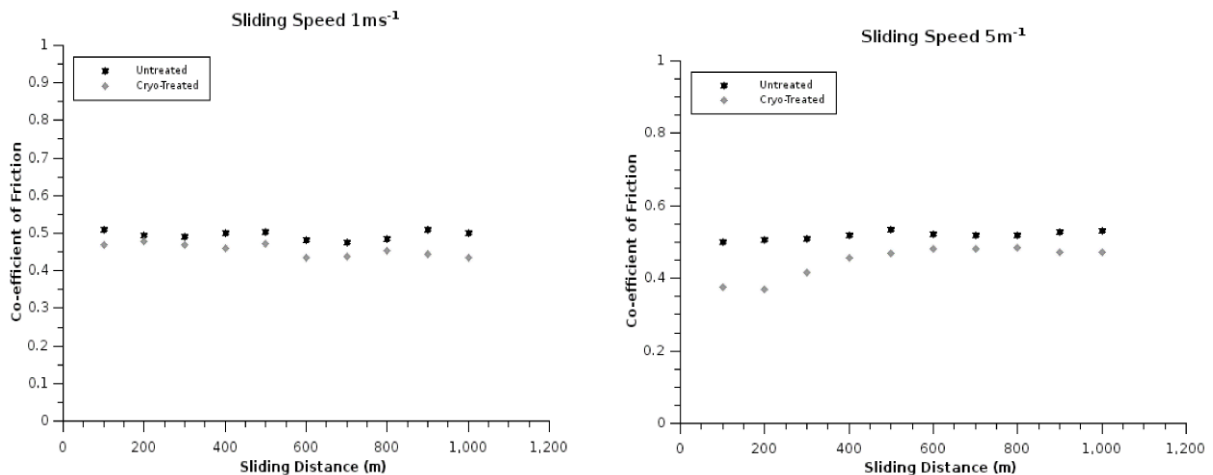


Fig. 5. Variation of friction as a function of sliding distance for linear speeds 1m/s and 5 m/s

Plots of displacement measured by linear variable differential transformer (LVDT) as a function of sliding distance for linear speeds 1m/s and 5m/s are made for both untreated and cryo-treated samples. The plots are as shown in Fig. 6. The plots show appreciable increase in the wear resistance of D3 steel after cryo-treatment. The percentage change in LVDT displacement per kilometer sliding is calculated from trend lines (least square fit). Even though the LVDT displacement does not exactly bring out the precise value of linear wear it represents a value proportional to linear wear since the normal load is constant.

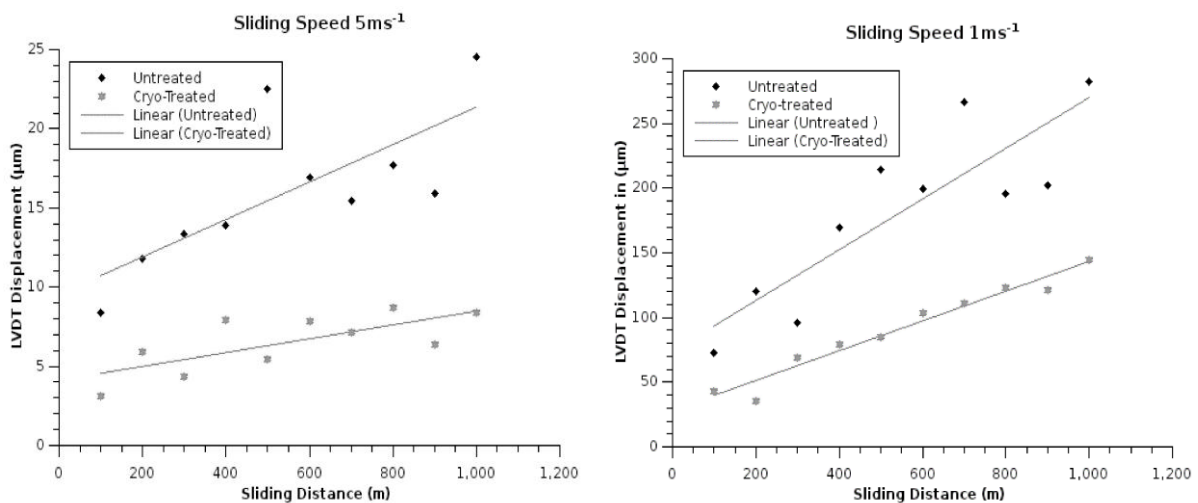


Figure. 6. LVDT displacement function of sliding distance for linear speeds 1m/s and 5 m/s

Wear resistance enhancement in cryo-treated specimen is found to be better by 40% for linear speed 1m/s in comparison with untreated specimen for the same linear speed. Similar plot for the linear speed of 5m/s is made and the plot reveals enhancement in wear resistance better by 80% for cryo-treated specimen in comparison with untreated specimen for the same linear speed.

VII. CONCLUSION

The current investigation reveals the effect of cryo-treatment on austenitic Ductile Iron type D3 Tool steel in terms of increase in hardness and enhancement in the the wear resistance of the material. The cryo-treated samples wear out slowly when compared to untreated samples confirming the enhancement in wear resistance there by increasing the tool life. The increase in hardness makes the material better with regard to mechanical properties.

The investigation also confirms cryo-treatment as an efficient onetime process to reduce wear in the austenitic ductile iron type D3 tool steel. The Cryogenic treatment leads to an appreciable increase in wear resistance of the material. It is found that the percentage change in wear is lower for higher speeds thus leading to dependence of wear resistance on speed. The lumps of wear particles formed suggest the initial adhesive wear. Furthermore the wear debris indulge in three body abrasive wear. It can be concluded that the coefficient of friction decreases when austenitic ductile iron type D3 tool Steel is cryo-treated.

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REFERENCES

JOURNAL PAPERS:

- [1] Barron F. R., Yes cryogenic treatments can save you money here is why, *Tapi*, 57(5), 1974, 35-40.
- [2] Barron R. F., Cryogenic treatment of metals to improve the wear resistance, *Cryogenics* 409-413, 1982.
- [3] Paulin P., Frozen gears, *Gear Technol* 26-28, 1993.
- [4] Yun D., Xiaoping L., Hongshen X., Deep cryogenic treatment of high speed steel and its mechanism, *Heat Treat. Met.* 55-59, 1998.
- [5] Huang JY, Zhu YT, Liao XZ, Beyerlein IJ, Bourke MA, Mitchell TE. Microstructure of cryogenic treated M2 tool steel. *Materials Science and Engineering*, A339 241(4), 2003.
- [6] Molinari A., Pellizzari M., Gialanella S., Straffelini S., Stiasny K. H., Effect of Deep cryogenic treatment on the mechanical properties of tool steels, *J. Mater. Process.technol.* 118, 2001, 350-355.
- [7] Mohan Lal D., Renganarayanan S., Kalanidhi A., Cryogenic treatment to augment wear resistance of tool and die steels, *Cryogenics*, 41, 149-155, 2001, 149-155 .
- [8] Dhokey N. B., Nirbhavane S., Dry sliding wear of cryo-treated multiple tempered D-3 tool steel, *Journal of Materials Processing Technology* ,209(3), 2009, 1484-1490.
- [9] Gopal Krishna P. V., Kishore K., Ramadevudu G., Sikandar Ali, Performance evaluation of cryogenic treated tools in turning, *Advances in Production Engineering & Management*, 3, 2012, 187-194 .
- [10] Mohan C. B., Divakar C., Venkatesh K., Gopalakrishna K., Mahesh Lohith K. S., Naveen T. N., Design and development of an linear reciprocating tribometer, *Wear*, 267, 2009, 1111-1116.
- [11] Garcia I., Fransaeer J., Celis J.P, Electro-deposition and sliding wear resistance of nickel composite coatings containing micron and sub-micron SiC particles *Surface and Coatings Technology*, 148, 2001, 171-178.
- [12] Yuji Chibaa, Toshio Omuraa and Hiroshi Ichimuraa, Wear resistance of arc ion-plated chromium nitride coatings, *Journal of Materials Research*, 8, 1993, 1109-1115.
- [13] Dutta Majumdar J., Galun R., Mordike B. L., Manna I, Effect of laser surface melting on corrosion and wear resistance of a commercial magnesium alloy, *Materials Science and Engineering*, A361, 2003, 119-129.
- [14] Sun Y., Li X., Bell T., Low temperature plasma carburising of austenitic stainless steels for improved wear and corrosion resistance, *Surface Engineering* 15(1), 1999, 49-54.
- [15] Feng B., Weng J., Qu S. X., Leng Y. X., Zhou Z. R., Improving wear resistance by heat treatment in different atmospheres, *key engineering Materials*, 288-289, 2005, 641-644.