

The Effect of Cryogenic Treatment on Wear Resistance and Hardness of Tool Steel AISI “D2”

Mohamed. M. Salem

The Advanced Center of Technology,
Tripoli – Libya
mmohelby@gmail.com

Adel. M. Burakhis

The Nuclear Research Centre, Tripoli –
Libya
a_burkhis@yahoo.com

Ibrahim. K. Husain

The Advanced Center of Technology,
Tripoli – Libya
ibrahimzg@yahoo.com

Abstract— The purpose of this paper intended to study different heat treatments in conjunction with a special Cryogenic treatment process and its effect on the wear behavior, and hardness of AISI D2 tool steel. An experimental demonstration of this study was first carried out by a set of specimens left for one week in air after conventional treatment to make stabilization and the other set conditioned at 60°C for one hour before shallow and deep cryogenic treatment. This investigation has shown that an improvement of hardness and wear resistance in deep cryogenic (DCT) process compared to shallow cryogenic treatment (SCT) for different soaking time which was approved by micro-structural examination especially in the stabilized specimens. This finding is in agreement with other results found in the research using similar tool steel and other specific cryogenic processes.

Index Terms: Deep cryogenic treatment (DCT), Shallow cryogenic treatment (SCT), Conventional heat treatment (CHT), Retained austenite, Wear resistance, Microstructure, Tool life.

I. INTRODUCTION

The technology of cryogenic treatment has been used as the finishing operation between the quenching and tempering treatment which applied to tool steels with the purpose of increasing hardness and wear resistance. The important aspect of this process is the changes occur in the crystal structure of the materials which can lead to an improvement in the mechanical properties. For several years great effort has been devoted to the study of cryogenic treatment on the properties of metals [1].

Even though; the Conventional heat treatment gives improvements in hardness, toughness, wear resistance, and ductility to the steel. On the other hand, the conventional process cannot remove all of the retained austenite (large and unstable particles of carbon carbide) in the steel. The retained austenite is a soft phase if present in steels could reduce the tool product life, and during the working conditions it can be transformed into martensite and causes several problems for the working tools. The new martensite evolved during conventional process is a very brittle and causes micro-cracks that can severely reduce the product life. Regarding the problems mentioned above, the controlled transformation of retained austenite into martensite is essential to many types of service applications. In order to obtain this microstructural transformation a cold treatment is used. The cold treatment is generally classified as either so called “sub-zero treatment” at temperatures down to about (-80°C) or “deep cryogenic treatment” at liquid nitrogen temperature (-196°C). More recent evidence shows that the wear resistance is further enhanced by virtue of cryogenic treatment at liquid nitrogen temperature. Most researchers believed that there are two mechanisms to improve the mechanical properties of the work that has been treated cryogenically. The first mechanism is attributed to the transformation of retained austenite to martensite; and the second is to initiate a nucleation sites for precipitating a large number of fine carbides in the matrix of martensite [2, 3]. The Cryogenic treatment is an add-on process to the conventional heat treatment of tool/die steel. It consists of controlled cooling of conventionally hardened steel specimens to some selected cryogenic temperature (-50°C to -196°C) and holding there for sufficiently long duration (20 to 75 hours) before being heated back to the ambient temperature at a predetermined rate for subsequent tempering treatment. It is different from the age-old cold treatment, which is carried out in between (- 600°C and –

Received 8 Aug 2017; revised 23 Aug 2017; accepted 18 Sept 2017.

Available online 20 Sept 2017.

800°C) and without any significant duration of soaking at the lowest temperature of treatment. [4, 5, 6] Due to the cryogenic treatment, the problems occurred in conventional heat treatment is reduced by controlled transformation of the retained austenite into martensite, which is essential to many types of components in service. Cryogenic treatment in tool steels produces fine dispersed carbide precipitates in martensite matrix and converts soft unstable austenite into martensite. Cryogenic treatment enhances wear resistance, hardness, toughness, resistance to fatigue cracking, as well as dimensional stability and decreases residual stresses. The greatest improvement in properties is obtained by selecting proper heat treatment process sequence (cryogenic treatment in between quenching and tempering), heating and cooling rate, stabilization (keep at room temperature for one week after quenching), hardening temperature, and soaking time (Cryo-process time) [7, 8, 9, 10].

II. EXPERIMENTAL WORK

A. Materials

In this research AISI D2 tool steel were selected for studying the effect of Cryo-Treatment cycle on the microstructure and some mechanical properties on the cryo-treated tool steel, the AISI D2 it has high carbon high chromium and used in production of blanking and deep drawing dies, thread rolling, forming dies, punishing tools, shear, and slitter knives. Given that it is a comparative study, therefore two sets of specimens have been prepared for all different cycle tests. The specimens were cut from AISI D2 bar of 45 mm diameter with 10mm thickness, and were machined according to the testing standards for the selected treatment cycles and specified mechanical testing required, and the steel chemical composition presented in table (1).

Table 1. Chemical Composition of the AISI D2 Steel

| Element | (Weight %) |
|---------|------------|
| C | 1.54 |
| Mn | 0.38 |
| Si | 0.39 |
| Cr | 12.4 |
| Mo | 0.69 |
| V | 0.80 |

B. Heat Treatment Process

B.1. Conventional heat treatment

The material selected for this work was subjected to a various heat treatments. The samples were first heat treated as per ASTM (681-08), then the tool steel specimens were conventionally heat treated (CHT) followed by cryogenic treatment as shown in Figure (1).

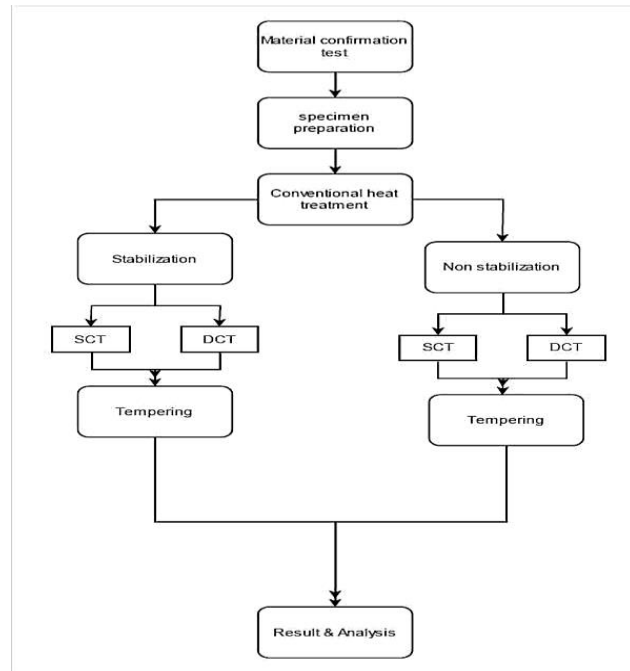


Figure 1. Schematic Diagram Showing the Methodology Adopted.

Initially all the specimens were first preheated at 650°C for 30 minutes, then austenized at temperature of 1040°C for 30 minutes and quenched in oil at 60°C. Then all samples were tempered at temperature of 200°C for 2 hours for the stabilization to occur, and then the samples were air-cooled to room temperature. Finally; the lot of the treated samples were conditioned at 60°C for one hour and divided into two sets. The first set was swiftly transferred to cryo-treatment, where the second set lifted for one week at room temperature for stabilization to take place before cryo-treatment. Then, both sets of samples were subjected to shallow cryogenic treatment at -80°C which designated to A1 and B1 and deep cryogenic treatment at -196°C and designated to A2 and B2 as illustrated in table (2), were both sets being carried out for two different isothermal holding times of 6 and 20 hours respectively.

Table 2. Designated Code for Experimental Specimens

| Cryo-treatment | Stabilizing | Non stabilizing | Period |
|----------------|-------------|-----------------|-----------|
| SCT | A1 | B1 | 6&20 hrs. |
| DCT | A2 | B2 | 6&20 hrs. |

B.2. Cryo-treatment

Cryogenic treatment of steels are quite often studied and they play an important role in developing the tribological properties of steels. One of the most common claims in cryogenic treatment is an increase in wear resistance. Cryogenic treatment (CT) specimens of tool materials

consists of two stages; that involving cooling of tool materials from room temperature at slow rate to temperature as low as -80°C for shallow cryogenic treatment (SCT) and -196°C for deep cryogenic treatment (DCT) in the cryogenic freezer which pressurized fully by nitrogen. The shallow cryo-treatment has been carried out by controlled dropping of temperature from room temperature to -80°C in the liquid nitrogen soaking chamber wherein they are stored for 6 and 20 hours as shown in Figure (2). The specimens were slowly cooled until reached the final soaking temperature of -80°C . The Deep cryogenic treatment has been carried out at -196°C with soaking time of 6 and 20 hours, then cooled at same cooling rate until they reached the final soaking temp of -196°C , then the cycle was reversed such that temperature ramp up at the same rate to room temperature. The benefits are usually attributed to reduction or eliminate of retained austenite from hardened steel and accompanied by homogenous structure of martensite.

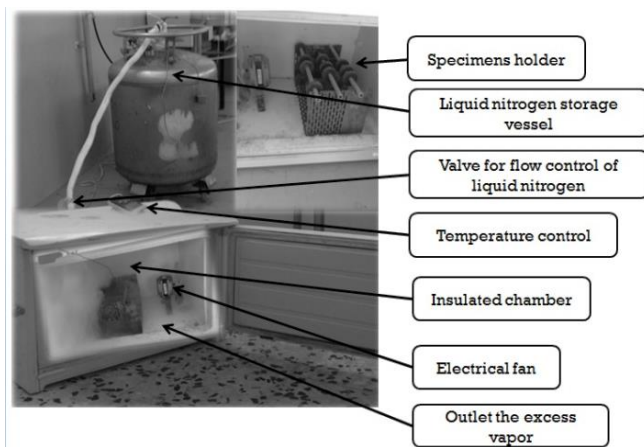


Figure (2) The Experimental Set up for Deep Cryogenic.

B.3. Tempering

The process consists of heating the hardened component to about (200°C) as per ASTM 681-A, then holding for one hour and then cooling to room temperature. Double tempering is the repeating of the above process, where the purpose of tempering is to relieve the internal stress, which is developed due to rapid cooling of tool steel after hardening process.

B.4. Hardness measurement

Hardness test of specimens subjected to shallow cryogenic treatment (SCT) and deep cryogenic treatment (DCT) was carried out as per ASTM standard. For results to be accurate at least, five hardness measurements were taken for each test sample. The hardness test machine (Officine Galileo) was used where the standard penetration was

obtained with diamond cone indenter on the Rockwell scale and the applied major load was 150 kg.

B.5. Wear test

To study the effect of shallow and deep cryogenic treatment on wear behavior, the block on ring wear test system (ASTM G77) was selected and used. The wear testing machine (Make Amsler type A-135) as shown in Figure (3) was used. This system consists of a block as stationary specimen and the ring as rotational specimen, the block has a shape of a plate with a concave recess which radius conforms to that of the ring. Wear test measurements were performed using a standard test method for ranking the resistance of materials to sliding wear using Block-on ring wear test with lubricant oil (15w40) at room temperature in according to ASTM G77 (Approval 2010). The dimensions of block are $10\text{mm} \times 30\text{mm} \times 55\text{mm}$ where the diameter of the ring was 45mm as in Figure (4). Both Block and ring were treated with the same heat treatment. For this test the following parameters were selected;

Test load = 1000N

The rotation speed $N = 360$ RPM

The interval duration time = 20 min.

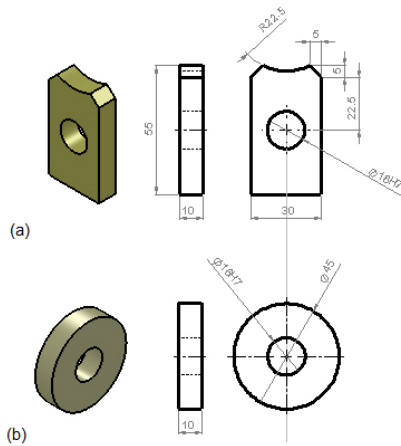
The Sliding distance 1018 meters at 20 minutes.

Total sliding distance = 5090 meter.

The weight loss after all five interval distances was measured.



Figure 3. Block on Ring Wear Test Machine.



Dimensions in mm

Figure 4. The Wear Test Work Pieces [REF]
 (a) Stationary Block Specimen, (b) Ring Specimen.

III. RESULTS AND DISCUSSION

A.1. Hardness study

The effect of shallow and deep cryogenic heat treatment at soaking times 6 and 20 hours were tested respectively, then the hardness were plotted as shown in figure (5). we can conclude that stabilizing heat treated specimens (A1,A2) for both shallow and deep cryogenic treatment showed higher hardness than the non-stabilizing heat treated specimens (B1,B2) at the same soaking time 6 and 20 hours respectively.

Deep cryogenic treated (DCT) specimens showed higher hardness compared with shallow cryogenic treated (SCT) specimens at the same soaking as shown in figures (5).

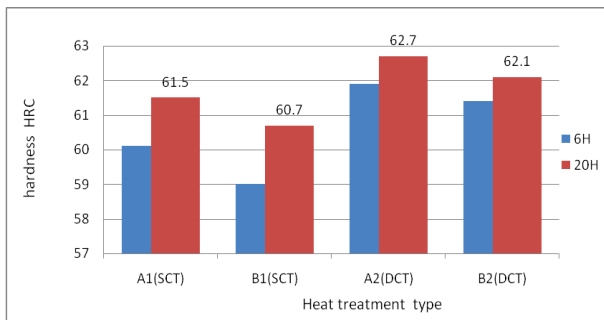


Figure 5. Hardness Test of d2 Tool Steel for Different HT

A.2. Wear study analysis

To study the different heat treatment on wear behavior of test sample, a block on ring test was employed according to ASTM G77, where the both block and ring have the same heat treatment regime in the experiment. The discontinuous course of the test been applied in order to evaluate wear resistance by mass loss measuring method. According to ASTM G77 (reapproved 2010) the wear rate is supposed to be determine after the interval sliding distance in (g/m).

In figure (6) the stabilized (A1,A2) specimens emphasis less mass loss in comparison with a non-stabilized (B1,B2) specimens for both 6 and 20 hours period which also led to less wear rate as shown in figure (7). The deep cryogenic specimens in (DCT) revealed less wear rate and more improvement in wear resistance compared with shallow cryogenic specimens especially in (20) hour period according to the SEM analysis , this improvement indicated to reduction of retained austenite and transformed to martensite with refine of small carbide and more uniform distribution in microstructure as showed in figure (9).

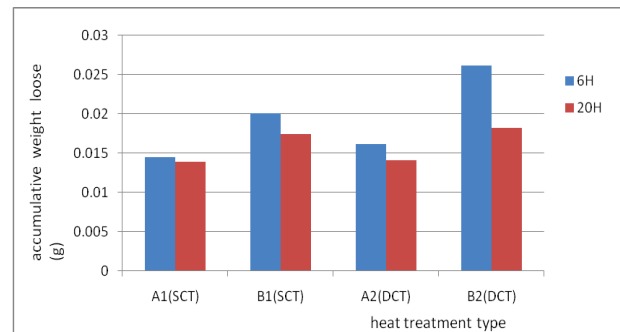


Figure 6. Accumulative Weight Loose vs Heat Treatments Type.

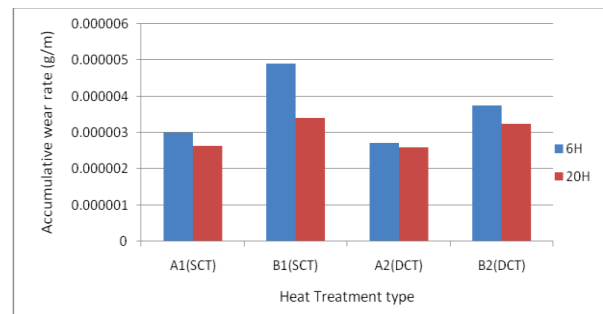


Figure 7. Accumulative Wear Rate vs. Heat Treatments Type.

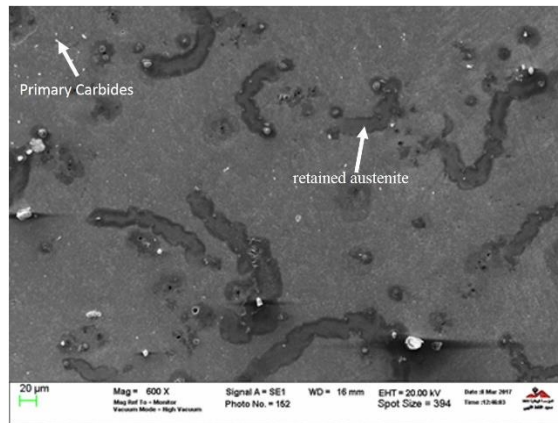


Figure 8. Shallow Cryogenic Treatment (20h)

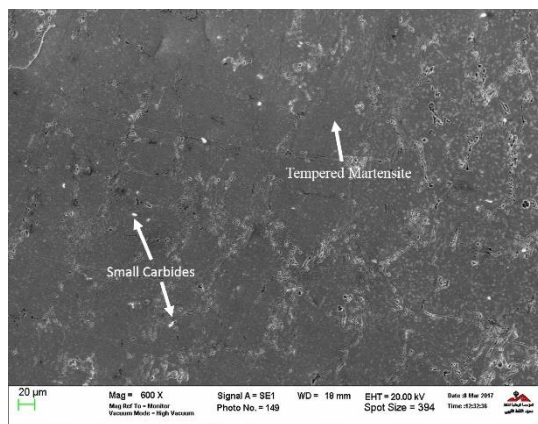


Figure 9. SEM Micrograph of D2 Tool Steel after Subjected to Deep Cryogenic Treatment (20hrs)

IV. Conclusion

The comparative studies made on the effect of cryogenic treatment on AISI D2 mechanical properties assist to infer the following conclusions:

- 1- There is an increase in hardness about (2%) for deep cryogenic treated specimens of AISI D2 comparing to shallow cryogenic treated specimens at 20h for stabilization process.
- 2- Improvement of wear resistance for stabilized specimens of AISI D2 for both shallow & deep cryogenic treatment comparing to non-stabilized specimens at different soaking time 6 and 20 hours.
- 3- Deep cryogenic treated specimens of AISI D2 have higher hardness and better wear resistance for both stabilization and non-stabilization process.

ACKNOWLEDGMENTS

The authors would like to thank and appreciate the help and assistance from the Machining workshop and Research Laboratories in the Advance center of Technology (Tripoli-Libya), also appreciate the efforts of Fathi Owahida and Ibrahim alsharif in wear testing in central research laboratories.

REFERENCES

- [1] L. Bourithis, G.D. Papadimitriou, J. Sideris, Comparison of wear properties of tool steels AISI D2 and O1 with the same hardness, *Tribology International*, 39, 2006, pp 479-489.
- [2] Fanju Meng, Kohsuke Tagashira, Ryo Azumu and Hideaki Sohma, Role of eta-carbide precipitations in the wear resistance improvements of Fe-12Cr-Mo-V-1.4C tool steel by cryogenic treatment, *ISIJ international*, 34, 1994, 205-210.
- [3] Chai Hung Sun, The effect of microstructure and the mechanical properties of AISI D2 tool steel by deep cryogenic treatment, Tatung University, Thesis for MS, July 2006.
- [4] D. Das, A.K. Dutta, K.K. Ray, Correlation of microstructure with wear behavior of deep cryogenically treated AISI D2 steel, *Wear*, 267, 2009, 1371-1380.
- [5] D. Das, A.K. Dutta, K.K. Ray, Optimization of the duration of cryogenic processing to maximize wear resistance of AISI D2 steel, *Cryogenics*, 49, 2009, 176-184.
- [6] D. Das, A.K. Dutta, K.K. Ray, On the enhancement of wear resistance of tool steels by cryogenic treatment, *Philosophical Magazine Letters*, 88 (11), 2008, 801-811.
- [7] A. Molinari, M. Pellizzari, S. Gialanella, G. Staffellini, K. H. Stiansy, Effect of deep cryogenic treatment on the mechanical properties of tool steels, *Journal of Materials Processing Technology*, 118, 2001, 350-355.
- [8] N.B. Dhokey, S. Nirbhavne, Dry sliding wear of cryotreated multiple tempered D-3 tool steel, *Materials Processing Technology*, 209, 2009, 1484-1490.
- [9] A. Akhbarizadeh, A. Shafyei, M.A. Golozar, Effects of cryogenic treatment on wear behavior of D6 tool steel, *Materials and Design*, 30, 2009, 3259-3264.
- [10] Cord Henrik Surberg, Paul Stratton, and Klaus Lingenhole, The effect of some heat treatment parameters on the dimensional stability of AISI D2, *Cryogenics*, 48, 2008, 42-47.
- [11] Standard Test Method for Ranking resistance of Materials to Sliding Wear Using Block-on-ring Wear Test, norm G77 - 05 (Reapproved 2010), ASTM International, United States.