Effect of Cryogenic Treatment on Mechanical Properties of Cold Work Tool Steels

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Abstract: *In this paper effect of cryogenic treatment on mechanical properties of cold work tool steels at various combination of heat treatment cycle (process sequence) are studied. The material selected for these processes are AISI D2 and D3.Cryogenic treatment is commonly referred as cryotreatment, is an add-on process to the conventional heat treatment of tool steel. Cryogenic treatment improves hardness, microstructure of metal (retained austenite to marten site), dimensional stability and decreases residual stresses. The effects of these process sequences of cryotreatment on properties of tool steel were studied by conducting some laboratory tests. Study of microstructure characterization of different specimens is carried out using optical microscope. Also hardness measurement was done by using Vickers indentation method.*

Keywords: *Cryogenic treatments, hardness, microstructure, Phase transitions, Tool steel.*

I. INTRODUCTION

Tool steel refers to a variety of carbon and alloy steels that are particularly well-suited to be made into tools. Their suitability comes from their distinctive hardness, resistance to abrasion, their ability to hold a cutting edge, and their resistance to deformation at elevated temperatures (red-hardness). Tool steels are used in cutting tools, punches, and other industrial tooling. Different tool steels are developed to resist wear at temperatures of forming and cutting applications.

Tool steels are broadly divided into six categories like cold work, shock resisting, hot work, high speed, water hardening, plastic mold and special-purpose tool steels. Among them, cold work tool steels are the most important category, as they are used for many types of tools, dies and other applications where high wear resistance and low cost are needed.[1]

Tool steel is generally used in a heat treated state. Conventional heat treatment gives hardness as well as toughness, wear resistance and ductility to steel. Even performed properly, conventional heat treating cannot remove all of the retained austenite (large, unstable particles of carbon carbide) from steel. The retained austenite as a soft phase in steels could reduce the product life and in working conditions, it can be transformed into marten site. This new marten site could cause several problems for working tools. This new marten site is very brittle and differs from the tempered one, which is used in tools.

Furthermore, this marten site causes micro cracks and reduces the product life. Regarding the problems mentioned above, the controlled transformation of retained austenite into marten site is essential to many types of component. In order to obtain this transformation the cold treatment is used.

Cold treatment is generally classified as either so called “sub-zero treatment” at temperatures down to about -80 ℃ or “deep cryogenic treatment” at liquid nitrogen temperature (-196℃). 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 marten site. The second is to initiate the nucleation sites for precipitating a large number of fine carbides in the matrix of marten site. [2, 3]

Cryogenic treatment commonly referred to as cryotreatment, 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 ($–50^0$c to$–196^0$c) and holding there for sufficiently long duration (20 to 75 h) 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 $– 60^0$c and - 80^0 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 marten site, which is essential to many types of component. Cryogenic treatment in tool steels causes the precipitation of finely dispersed carbides in marten site and also converts soft unstable austenite to martensite. Cryogenic treatment improves wear resistance, hardness, toughness, resistance to fatigue cracking, microstructure of metal (retained austenite to marten site), dimensional stability and decreases residual stresses. Also reduces tool consumption and down time for equipment setup thus leading to cost reduction of about 50%.Cryogenic treatment gives machining, grinding and polishing finish due to little soft austenite. The greatest improvement in properties is obtained by selecting proper heat treatment process sequence (cryogenic treatment in between quenching and tempering), soaking time (cryoprocess time), stabilization (keep at room temperature for one week after quenching), hardening temperature, heating and cooling rate. [7, 8, 9, 10].

II. EXPERIMENTAL METHODOLOGY

2.1 Material selection

AISI D2 and D3 tool steel materials are select for studying effect of process sequence on behavior of cryotreated cold work tool steel at different process cycles. The chemical composition of specimen (dia.10mm, Height 35mm) was analyzed in optical emission spectroscope (OES). The machine used for the analysis of raw material chemical composition was named as Spectrophoto Analyzer [AS 200(Switzerland)].

2.2 Treatments

The material chosen in this work was given various treatments and treatment cycles indicated in fig.1 and table I. Specimens were subjected to conventional heat treatment and deep cryogenic treatment separately. Conventional heat treatment consist of hardening and tempering, while deep cryogenic treatment involve an additional low temperature treatment cycle to conventional heat treatment process.

TABLE I: Process sequence for both tool steel D2 & D3

Sr. No.	***Group Name***	***Process Sequence***
1	Group A	Conventional Heat Treated specimens (CHT)
2	Group B	Austenitizing Quenching Cryogenic (AQC)
3	Group C	Austenitizing Quenching Cryogenic tempering (AQCT)
4	Group D	Austenitizing Quenching Cryogenic tempering tempering (AQCTT)
5	Group E	Austenitizing Quenching tempering Cryogenic tempering (AQTCT)

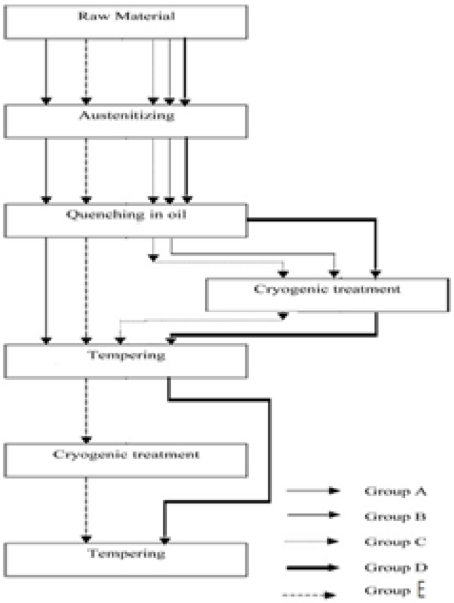


Figure 1: Flow chart for process sequence

2.2.1 Hardening

Pre heating at 720^0C was done before hardening and Hardening at 1020^0C was done in electrically heated protected atmosphere type furnace, for both the specimens of tool steel D2 & D3.Hardening was done at 1020^0C to achieve the austenitization point & grain uniformity (Structural balance).

2.2.2 Quenching

Quenching is done at 500^0C in oil, with continuous agitation, to avoid thermal shocks.

2.2.3 Tempering

Tempering was done in electrically heated protected atmosphere type furnace at 520^0C to relieve the stresses.

2.2.4 Cryogenic Treatment

Cryogenic treatment was done on tool steel D2 & D3 at -185^0C, for 10 hrs. The cryogenic treatment box was made by America Cryogenics.

III. EXPERIMENTAL SETUP FOR CRYOGENIC HEAT TREATMENT

Fig. 2 shows the schematic representation of Cryogenic Treatment equipment is used to obtain -125°C to -196°C temperature. It comprises an insulated box (Cryo-box), one motor with a circulating fan, one thermocouple to measure the cryogenic temperature inside the box connected to a temperature controller and programmer, a liquid nitrogen tank and a solenoid valve for the gas inlet. The actual temperature of the mass loaded in the box is recorded by a thermocouple.

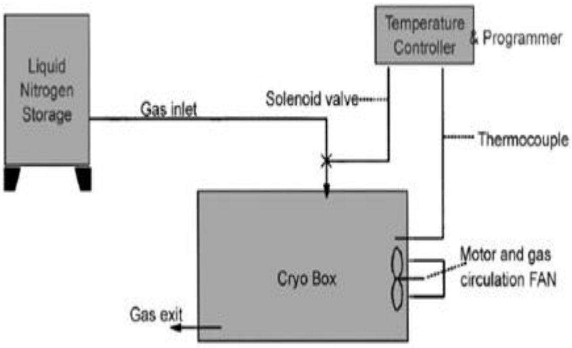


Figure 2: Block diagram of the cryogenic treatment equipment

IV. RESULTS OF CHEMICAL ANALYSIS

The composition of the raw material was analyzed in optical emission spectroscope (OES). The machine used for the analysis of raw material chemical composition was named as Spectrophoto Analyzer [AS200 (Switzerland)].

V. MICROSTRUCTURAL CHARACTERIZATION

Microstructure examinations were carried out by using inverted optical microscope of Carl Zeiss AL350 at magnification 450X. Different phases like retained austenite, untendered marten site, tempered marten site were checked. Also secondary carbide sizes & shapes were checked.

VI. HARDNESS MEASUREMENT

For all hardness measurement, Vickers indentation method was used. The flat surface was prepared by polishing paper on 1/0. For D-2 and D3 steel material HV scale was used.

VII. RESULTS AND DISCUSSION

7.1 Results of chemical analysis of D2 and D3 raw material

The material considered for the study was obtained in the form of 10mm diameter rod & length 40mm of D2 & D3 tool steel.

TABLE II: The result of chemical analysis of D2 raw material

Carbon	Chromium	Manganese	Vanadium	Molybdenum	Iron
1.5	12.53	0.36	1.04	0.83	Remaining

TABLE III: The result of chemical analysis of D3 raw material

Carbon	Chromium	Manganese	Silicon	Iron
2.23	12.31	0.49	0.26	Remaining

7.2 Hardness study

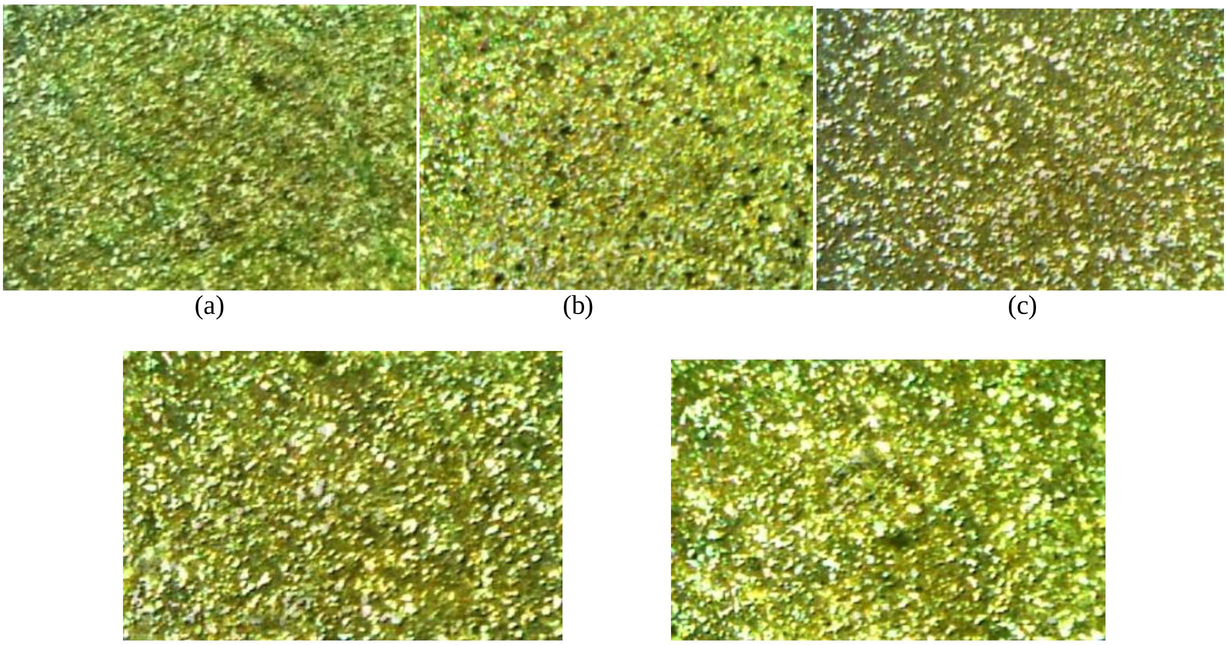
The hardness values for all samples are shown in table no. IV. It is clear from the table that for D2 material CHT specimen has less hardness than cryotreated specimen but there is gradual decrease in hardness observed from AQC to AQTCT. For D3 material CHT specimen has less hardness than cryotreated specimen but there is gradual increase in hardness observed from AQCT to AQTCT. The AQC specimen of D3 material has highest hardness.

TABLE IV: The hardness (HV) of the heat-treated D2 & D3 tool steel samples

Process	**Hardness (HV)**	
	D2	**D3**
Conventional Heat Treated specimens (CHT)	805	811.8
Austenitizing Quenching Cryogenic (AQC)	927	1022.3
Austenitizing Quenching Cryogenic tempering (AQCT)	916	724.3
Austenitizing Quenching Cryogenic tempering tempering (AQCTT)	841	731.5
Austenitizing Quenching tempering Cryogenic tempering (AQTCT)	828.7	767.3

7.3 Microstructure analysis

7.3.1 D2 Tool steel



(a) (b) (c)

(d) (e)

Figure 3 : Microstructure of various combinations of treatments for the specimen of D2 steel
a) CHT b) AQC c) AQCT d) AQCTT e) AQTCT

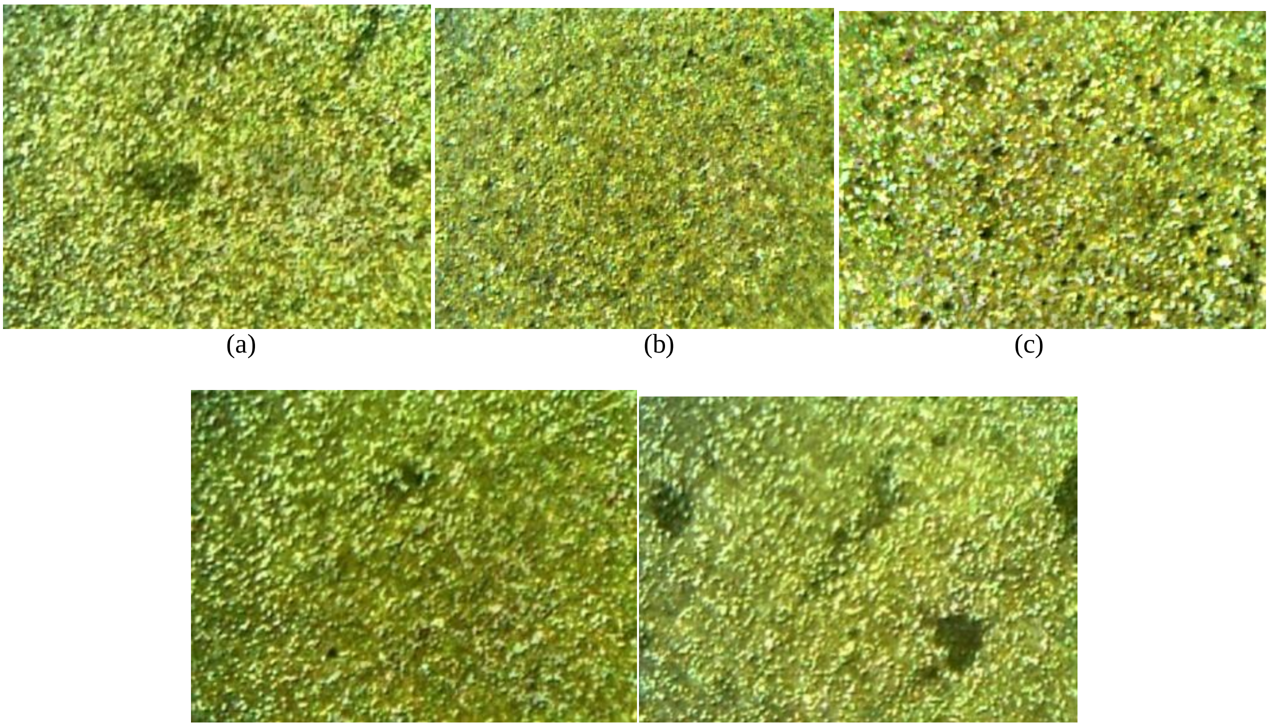
Microstructure of various combinations of treatment for the specimen of D2 steel is shown in fig. 3(a-e). Fig 3(a) shows microstructure of CHT in which Globular shape carbides up to 9 micron and nodular shape carbides of 11X 4 micron size. Martensite etches dark .Untempered martensite observed is more up to 10% and retained austenite is seen up to 50%. Fig 3 (b) shows microstructure of AQC. The structure consists of globular carbides are up to 2 micron and nodular carbide are of 2x5 micron in size. Untempered martensite observed is below 2%. Massive carbides not seen and 95% unstable austenitic structure seen. Fig 3 (c) shows microstructure of AQCT. Globular and nodular shape carbide up to 10% is observed in 5 micron and 10x3 micron in size respectively. Untempered martensite observed is more up to 4 %. Retained austenite is present up to 20% under tempered structure. Fig 3 (d) shows microstructure of AQCTT. Globular and nodular shape carbide up to 12% is observed in matrix of tempered martensite. Globular carbides are upto3 micron and nodular carbide is of 14x5 micron in size. Untempered martensite observed is more up to 5 % and retained austenite up to 50% under tempered structure. Fig 3 (e) shows microstructure of AQTCT. Nodular shape carbide up to 7% is of 13x6 micron in size, observed in matrix of tempered martensite. Untempered martensite observed is more up to 4%. Retained austenite up to 50%.under tempered structure. Microstructure of all combination treatment shows globular and nodular carbides are uniformly distributed in the austenite matrix.

7.3.2 D3 Tool Steel

Microstructure of various combinations of treatment for the specimen of D3steel is shown in fig. 4(a-e)**.** Fig. 4(a) shows microstructure of CHT in which Elliptical Globular shape carbide upto 20% of 3x6 micron in size are observed in matrix of tempered martensite. Untempered martensite observed is up to 7%.The structure is well distributed. Martensite etches dark. Globular and nodular carbides uniformly distributed in the tempered martensite matrix. 20% Retained austenite seen slightly under tempered structure. Fig 4 (b) shows microstructure of AQC. Globular and erratic shape carbide upto 4% are observed in matrix of tempered martensite. Globular carbides are up to 8 micron and erratic carbide are of 10x3 micron in size. Untempered martensite observed in trace amount. Martensite not etches dark. Globular and nodular carbides uniformly distributed in the austenite matrix. Massive carbides are not seen. Micro cracks visible.95% unstable austenitic structure. Fig 4(c) shows microstructure of AQCT **.**More nodular shape carbide upto 15% are of 2x4 micron in size, observed in matrix of tempered martensite. Untempered martensite observed is more upto 20%. Martensite etches dark.

Globular and nodular carbides uniformly distributed in the tempered matrix. Retained austenite not seen. Fig 4 (d) shows microstructure of AQCTT. Nodular shape carbide up to 10% is of 3x5 micron in size, observed in matrix of tempered martensite. Untempered martensite observed is more upto 7%. Martensite etches dark. Globular and nodular carbides uniformly distributed in the tempered martensite matrix. Retained austenite not seen. Fig 4 (e) shows microstructure of AQTCT. Globular and erratic shape carbide upto 10% of 2x4 micron in size is observed in matrix of tempered martensite.

Untempered martensite observed is upto 5%. Martensite etches dark. Globular and nodular carbides uniformly distributed in the tempered martensite matrix. Retained austenite is not seen.


(a) (b) (c)

(d) (e)

Figure 4 : Microstructure of various combinations of treatments for the specimen of D3 steel
a) CHT b) AQC c) AQCT d) AQCTT e) AQTCT

Microstructure of tool steel D2 & D3 were subjected to various combinations of treatments as described in table 1. In the process sequence Austenitizing Quenching Cryogenic tempering (AQCT), Austenitizing Quenching Cryogenic tempering tempering (AQCTT) & Austenitizing Quenching tempering Cryogenic tempering (AQTCT) for tool steel D3 retained austenite was not seen. For D2 tool steel in process sequence Austenitizing Quenching Cryogenic tempering (AQCT) retained austenite is up to 20%. In process Austenitizing Quenching Cryogenic tempering tempering (AQCTT) and Austenitizing Quenching tempering Cryogenic tempering (AQTCT) retained austenite is up to 50%. For D2 tool steel massive carbides not seen and 95% unstable austenitic structure is seen. Also the general shape of carbides observed was Globular, Nodular or elliptical.

VIII. CONCLUSION

Cryogenic treatment is add on process to conventional heat treatment process of tool steel. Cryogenic treatment improves microstructure of metal i.e. controlled transformation of retained austenite into martensite. For D3 tool steel retained austenite was not seen in the process sequence Austenitizing Quenching Cryogenic tempering (AQCT), Austenitizing Quenching Cryogenic tempering tempering (AQCTT) and Austenitizing Quenching tempering Cryogenic tempering (AQTCT). For D2 tool steel retained austenite is up to 20% in process sequence Austenitizing Quenching Cryogenic tempering (AQCT). Also retained austenite is up to 50% in process Austenitizing Quenching Cryogenic tempering tempering (AQCTT) and Austenitizing Quenching tempering Cryogenic tempering (AQTCT). The effect of cryogenic treatment on hardness shows that for D2 and D3 tool steel CHT specimen has less hardness than cryotreated specimen. But for D2 tool steel there is gradual decrease in hardness observed from AQC to AQTCT. For D3 tool steel there is gradual increase in hardness observed from AQCT to AQTCT and AQC specimen has highest hardness. The multiple tempering decreases hardness in D2 tool steel where as increases hardness in D3 tool steel. For D2 and D3 tool steel in process sequence Austenitizing Quenching Cryogenic (AQC) massive carbides not seen and 95% unstable austenitic structure is seen. But both tool steel have maximum hardness value for this process sequence. In D3 tool steel the micro cracks were observed on the untempered samples. The general shape of carbides observed was Globular, Nodular or elliptical. In D2 tool steel retained austenite is not totally converted to martensite where as in D3 tool steel retained austenite is totally converted to martensite.

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