International Journal of Current Engineering and Technology
ISSN 2277 - 4106

Available at http://inpressco.com/category/ijcet

Research Article

Effect of Deep Cryogenic Treatment on AISI T42 High Speed Steel

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Accepted 02 August 2013, Available online 01 October 2013, **Vol.3, No.4 (October 2013)**

Abstract

The study examines the effects of deep cryogenic treatment, applied at -196˚C with 24 hours soaking, on a super alloy high speed steel, AISI T42. Presence of retained austenite at the end of conventional heat treatment and at the end of cryogenic treatment was studied using X-ray diffractometry. XRD analysis confirms elimination of retained austenite from 19 % at the end of conventional heat treatment to less than 1 % at the end of deep cryogenic treatment. Increase in hardness from 67 HRC to 69.5 HRC and increase in impact energy from 5.2 J/mm^2 to 5.4 J/mm^2 were observed at the end of cryogenic treatment. Sliding abrasion wear study was conducted using pin on disc wear tester. Wear rate of conventionally heat treated samples was found to be as high as 3.18 times deep cryogenic treated specimens. Analysis of SEM images of microstructure confirms presence of ultrafine carbides of size less than 0.2 µ in specimens subjected to cryogenic treatment. Increase in M_{23} C_6 carbides, primarily chromium carbides, along grain boundaries was confirmed by microstructure analysis. SEM images of fractured surface of impact test specimens confirm that cryogenic treatment has not affected the toughness of specimen. The material exhibits an excellent hardening response to cryogenic treatment with increase in hardness and wear resistance.

Keywords: *Cryogenic Treatment, Microstructure, Hardness, Wear Resistance, Fractography.*

1. Introduction

High – speed steels (HSS) are very widely used tool materials in medium and small scale industries owing to their versatility and economic production of tools with intricate geometry. They are high alloy, tool steels with carbon, chromium, vanadium, molybdenum or tungsten or blends thereof and in some cases sizeable amounts of cobalt. The levels of alloying elements and realm of heat treatment confer high achievable hardening response, wear resistance, resistance to softening at elevated temperatures and good toughness for effective use in metal cutting applications.

Though HSS was introduced in the year 1903, with time, it had undergone several modifications. Today there are more than 150 grades of HSS that cater to wide ranging demands for various metal cutting applications. Subsequent to HSS, a number of superior cutting tool materials such as cemented carbides, ceramics and cermets made their inroads at different points in time. These materials revolutionized metal cutting in terms of higher cutting speeds and material removal rate. But the economic cutting speed of these materials is too high for operations such as tapping and reaming. In certain operations such as deep drilling HSS is the only logical choice, as it could absorb shocks and vibrations due to its excellent toughness & bending strength compared to other advanced tool materials. In the light of the above, cutting tools made of HSS still continue to dominate in shop floor especially in small and medium scale industries.

Further, enhancement in wear resistance of HSS tools is achieved by applying wear resistant coating. Though the coating extends service life of HSS tools, their performance bounces back to that of plain tool, once if the coating wears out. The other alternative option is Cryogenic treatment, a bulk hardening method which is applied at the end of conventional heat treatment, primarily to augment wear resistance and hardness. Since cryogenic treatment instills phase transformations across entire volume of cutting tool, the advantages of cryogenic treatment could be exploited after every regrinding. As consequence, cryogenic treatment can reduce consumption of tool materials.

Use of cryogenic treatment (Wilkins, C *et al* 1999), to enhance the properties and service life of tool materials have been widely researched from as early as 1940s. Until 1960 attempts of sub-zero treatment were made by directly immersing tools and metal parts in liquid nitrogen. Increase in hardness and wear resistance were the major beneficial effects, observed in components subjected to sub-zero treatment apart from improved surface finish, reduced co-efficient of friction at the interface between work piece and tool. Detrimental effects such as surface

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cracks, excessive brittleness were also encountered in many cases. Thermal shock due to sudden exposure to liquid nitrogen was identified as the major reason for unfavorable effects of sub-zero treatment.

Technological breakthrough in refrigeration cycles cross fertilized the development of cryogenic treatment systems that were able to carry out effective crack less cryogenically treated parts. The first ever sub-zero treatment system as established by Ed Busch (CryoTech – Detroit, MI) in 1960s. It was further upgraded by Peter Paulin with a feedback temperature control system, on cooling and warm up circuits. Effects of sub-zero treatment on HSS cutting tools was confirmed by Barron *et al* (Barron R.F *et al* 1982) in 1982.He reported that cryogenic treatment eliminates retained austenite present in tool steels subjected to conventional treatment. Also he confirmed the precipitation of fine eta carbides in cryogenically treated tool steels. After cryogenically treating several tool steels including AISI M2 HSS he revealed that deep cryogenic treatment carried out at -196 ˚C was more beneficial than shallow cryogenic treatment carried out at -84 ˚C.

Volume reduction of martensite, during cryogenic treatment, was confirmed by Popandopulo *et al* and Zhukova *et al* (Popandopulo, A.N *et al* and Zhukova, L.T *et al 1980)*. Dilatometry studies carried out during cryogenic treatment showed volumetric reduction of martensite in the temperature range of -90 to 20 ˚C. This behavior was as a consequence of partial decomposition metastable martensite due to thermal instability at low temperatures.

Meng *et al* (Meng,F *et al* 1994) studied the formation of η-carbides in tool steel during cryogenic treatment. He proposed that high degree of volumetric contraction, forces out carbon atoms from supersaturated martensite. Neighboring lattice defects, act as preferential sites for these wandering carbon atoms. These carbon atoms combine with alloying elements and precipitate ultra-fine η-carbides upon heating and tempering. These η-carbides differ in their crystal structure, with the usual ε - carbides. He attributed improvement in wear to uniform distribution of η-carbides in the martensite matrix.

Darwin *et al* (Darwin,J.D *et al* 2007) studied the effect of process parameters such as temperature, soaking time, cooling rate and tempering temperature, on 18% Cr martensitic stainless steel. He reported that soaking temperature was the most significant factor with 72% contribution, followed by soaking time with 24% contribution and thirdly cooling rate with 10% contribution. Tempering temperature contributes only 2% and tempering duration was reported as insignificant factor.

Presence of austenite is inevitable at the end of conventional heat treatment of tool steels. The presence of alloying elements and rate of cooling tend to suppress the transformation of austenite to martensite during quenching. Since HSS are high alloy steels with considerable proportion of alloying elements, a sizable amount of austenite is present at the end of conventional heat treatment. Effect of alloying elements on martensite start and martensite finish temperature is given in the following equations.

$$M_s = 539 - 423(C) - 30.4(Mn) - 12.1(Cr) - 17.7(Ni) - 7.5(Mo) \ldots\ ^{\circ}C$$

$$M_f = M_s - 215 \ldots\ ^{\circ}C$$

Also, during quenching at normal cooling rates, the martensite start temperature is lowered by 20 ˚C. Cooling rates followed in case of hardening cycles for HSS, is much higher and hence the martensite start temperature is further lowered. For eutectoid steel with 0.78 %C, transformation of martensite reaches its completion at approximately -50 ˚ C. It has been reported that the transformation of austenite to martensite which was terminated in the conventional heat treatment was restarted during cryogenic treatment. In most of the cases (Akhbarizadeh, A *et al* 2009; Das,D *et al* 2009; Shaohong Li *et al* 2010; Mahdi Koneshlou *et al* 2011;Kamran Amni *et al* 2012) it was reported that austenite is either completely eliminated by deep cryogenic treatment or was found at undetectable levels. X-ray diffraction spectral analysis was ideal to confirm the elimination of retained austenite at the end of cryogenic treatment. Concurrent increase in the proportion of carbides was also reported. Precipitates of fine carbides was evident in the microstructure of cryogenically treated specimens. Soaking time of 24 hours was reported to be adequate in most of the studies.

Presence of austenite in HSS tools, at the end of conventional heat treatment makes them vulnerable to wear. As it is the softest phase in the microstructure of HSS, tool wear starts only at these sites by ploughing action of hard particles from work piece material during metal cutting operation. Also retained austenite is metastable at room temperature and it gets converted to martensite under stress during metal cutting. Martensite formed in this manner is brittle and promotes failure of tool. Since cryogenic treatment is the ideal choice to alleviate the problem of austenite in HSS, it attracted more research in the recent years. But so far, only a few research findings have been reported on a limited grades of HSS. In order to study and justify the applicability of cryogenic treatment on a grade of HSS containing higher proportions of alloying elements AISI T42 grade of HSS has been selected for the present study.

2. Research Methodology

The specimens were prepared out of commercial AISI T42 grade high-speed steel with 1.5 % C, 0.291% Si, 0.232% Mn, 4.21 %Cr, 3.19 %Mo, 0.422%Ni, 9.75% Co, 8.95%W and the rest Fe. Since it is a comparative study on the effects of cryogenic treatment, two sets of specimen were prepared for all the tests, including Charpy impact test and pin- on- disc wear test. Initially all the specimens were subjected to conventional heat treatment. At the end of conventional heat treatment the specimens were divided into two batches, of which only one batch was subjected to cryogenic treatment.

2.1 Conventional Heat Treatment

Since salt bath furnaces are ideally suited for through hardening of high speed steel, the conventional heat treatment was accomplished in barium chloride salt bath followed by triple tempering.Fig.1 shows the sequence of operations followed during the conventional heat treatment.

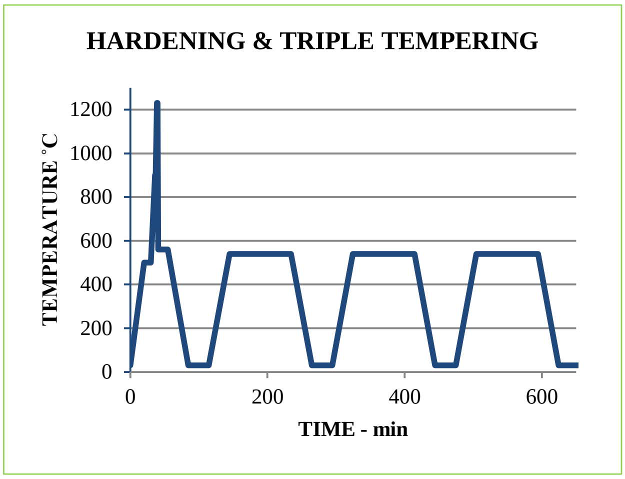


Fig.1 Conventional Heat Treatment applied to AISI T42

Specimens were preheated in a forced air circulation furnace to a temperature of 450˚C for 30 minutes followed by preheating in salt bath furnace maintained at 950˚C for a period of 6 minutes. The specimens were subsequently transferred to salt bath maintained at 1230˚C and soaked for a period of 40 seconds. Soaking period was chosen based on the cross section of the specimen. After austenitization in hardening furnace, the specimens were quenched in salt bath maintained 560˚C. In order to stabilize phases the specimens were soaked in the quench media for a period of 20 minutes. Finally the specimens were air cooled to room temperature. Hardness at the end of quenching was found to be 64.5 HRC.

Later, the specimens were triple tempered in a salt bath furnace maintained at 540 ˚C. Soaking period of one and half hours was adopted for each tempering cycle. Hardness at the end of triple tempering was found to be 67 HRC.

2.2 Cryogenic Treatment

From the batch of specimens subjected to conventional heat treatment, only half of specimens were subjected to deep cryogenic treatment. The other half of the specimens were retained for comparing the effects of deep cryogenic treatment on properties and wear behavior. Surface of specimens were thoroughly cleaned to remove traces of salt before transferring them to cryogenic treatment chamber. After loading the specimens in the cryogenic treatment chamber, the cooling rate was set at -0.5˚C per minute. When the temperature reached -195˚C the cryogenic treatment system was set to soaking mode for a period of 24 hours. During soaking, the temperature was steadily maintained at -195˚C. Then the cycle was reversed such that temperature within the cryogenic treatment chamber builds up at the rate of 0.5˚C per minute until it reached room temperature. Subsequently the specimens were subjected to double tempering at 200 ˚C. Soaking time of 2 hours was adopted for each tempering cycle. The hardness after double tempering was found to be 69.5 HRC.

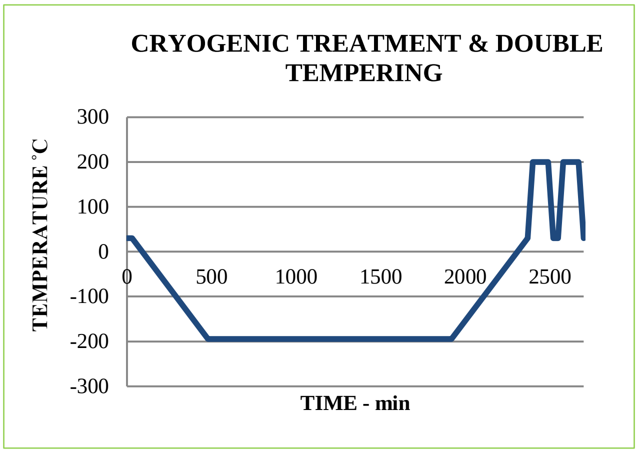


Fig.2 Deep Cryogenic Treatment applied to AISI T42

3. Experimentation

3.1 Hardness Test

Hardness of specimen was measured at the end of conventional heat treatment and at the end of cryogenic treatment. Bulk hardness was measured in Rockwell hardness C – scale as per ASTM E18 – 12 guidelines. Micro hardness tests were conducted as per ASTM E384-10 guidelines. Four tests were conducted at the end of conventional heat treatment and at the end of cryogenic treatment. The average value has been reported.

3.2 Charpy Impact Test

Specimens having 10mm square cross section and 55 mm long with a V-notch were prepared as per ASTM – E23 – 02a guidelines, from square rod of 12mm cross section. Four tests were conducted for conventionally heat treated specimens and another four tests were conducted for cryogenically treated specimens. Impact tests were conducted at a constant room temperature of 25˚C and relative humidity 60%, the average value of energy absorbed during fracture was reported.

3.3 Pin-on disc Wear Test

Ducom pin on disc wear tester with stationary pin and rotating disc configuration was used for conducting sliding abrasion wear study. The wear tests were conducted at a constant room temperature of 25˚C and relative humidity 60%, under dry condition. Cylindrical pins of diameter 10 mm with h7 tolerance and length 20mm were prepared from AISI T42 bar of 12 mm square cross section. Ends of the pin were polished using progressively finer grades of sand paper up to 1200 mesh. The weight of the specimens was recorded using a scale with precession of 10^{-5} gm, before loading them on wear tester. Discs of Ø55mm and thickness 10 mm with four holes of Ø3.5mm on pitch circle of Ø47 mm were made out of En 24 steel with C - 0.41%, Mn - 0.57%, Si - 0.24%, S - 0.01%, P - 0.03%, Cr - 1.21%, Ni – 1.47%, Mo – 0.28% and the balance Fe. Surface of disc was polished to the same finish as that of pins, before conducting the wear study. The sliding abrasion wear tests were conducted for a constant sliding distance of 3000m and track diameter of 25mm. Normal load was varied between 65 – 135 N and sliding velocity was varied between 0.28 – 1.2 m/s during the wear test. Weight loss of pin was measured at the end of each wear test.

4. Results and Discussion

Table-1 Results of Hardness Test

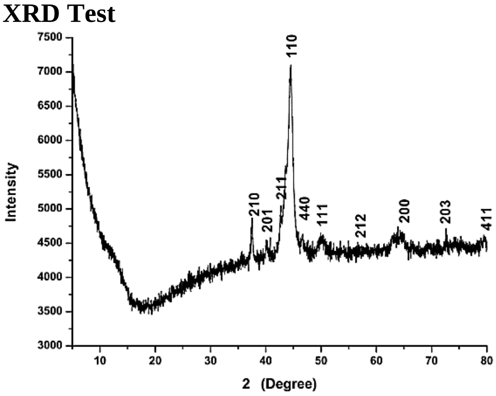
Process	Rockwell Hardness(Observation in HRC)	Micro Hardness (Observation in HV1kg)
Conventional Heat Treatment	67.1	996
Cryogenic Treatment	69.6	1020

Charpy Impact Test

Table-2 Results of Charpy Impact Test

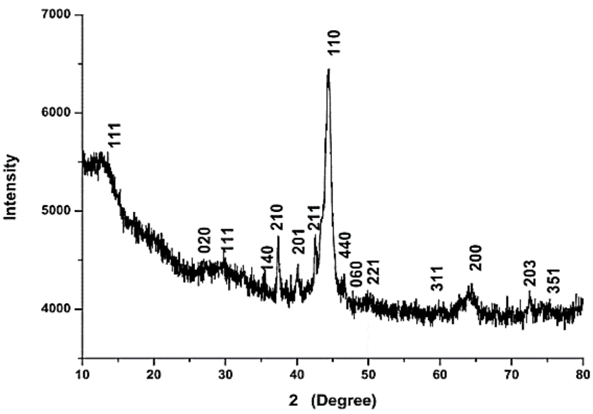
Treatment	Energy Absorbed (J/mm2)
Conventional Heat Treatment	5.2
Cryogenic Treatment	5.4

XRD Test



a. XRD Spectrum of conventionally heat treated AISI T42

The X-ray diffraction spectrum of conventionally heat treated AISI T42 specimen shows austenite peak corresponding to $2\theta = 50.978^\circ$. In the X-ray diffraction spectrum corresponding to cryogenically treated specimen the austenite peak at $2\theta = 50.978^\circ$ is completely absent. It is obvious from the above result that retained austenite present in conventionally heat treated samples have been transformed to martensite by cryogenic treatment.



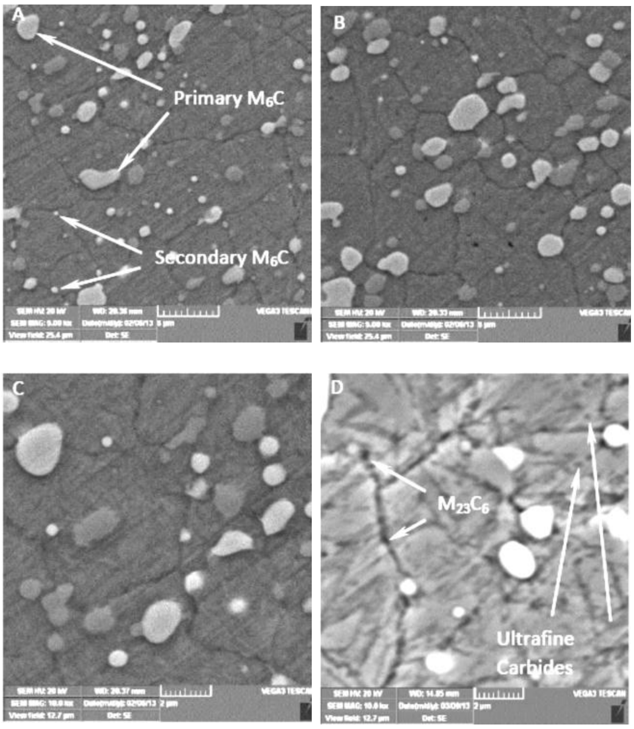
b. XRD Spectrum of deep cryogenically treated AISI T42

Fig.3 XRD spectrum

Microstructural analysis

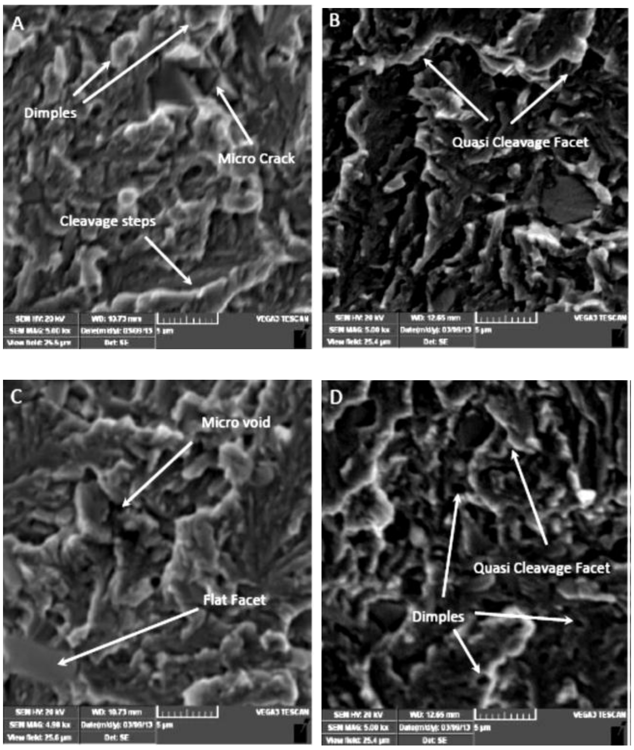
Effect of cryogenic treatment on microstructure was examined using scanning electron microscope. Samples for microstructure study, were cut across Ø10 x 20mm specimens by wire electro discharge machining. These samples were cut from both conventionally heat treated specimens and those subjected to cryogenic treatment. Cut specimens were moulded into bakelite and polished using SiC water proof papers from 120 to 1000 grit. Finally the mirror polished surface was etched using 2% nital. At least 10 images were taken per sample to study the presence of different phases and distribution of carbides. Referring to Fig.4 A and B, the distribution of primary M_6C carbides and secondary M_6C carbides are similar in character in both conventional heat treated and cryogenic treated specimens. Referring to Fig.4 C and D, the SEM images taken at 10,000X magnification discloses the difference in microstructure of conventionally heat treated and cryogenic treated specimen.

Uniform distribution of fine precipitates of carbides of size less than 0.2μ are identified in cryogenically treated specimens. Also the increase in population of $M_{23}C_6$ carbides along the grain boundaries point to the fact that carbon atoms squeezed out from martensite during cryogenic treatment migrate to the grain boundaries and upon subsequent heating and tempering these carbon atoms act as nuclei for the growth of fine carbides. These $M_{23}C_6$ primarily carbides of chromium contribute for the augmentation of hardness. The elimination of austenite due to cryogenic treatment and concurrent increase in carbides is also confirmed by the respective peaks in the XRD analysis.



A-SEM image of conventionally heat treated specimen at 5000X magnification
B-SEM image of cryogenically treated specimen at 5000X magnification
C-SEM image of conventionally heat treated specimen at 10,000X magnification
D-SEM image of cryogenically treated specimen at 10,000X magnification

Fig.4 SEM image of AISI T42 specimens



A & C Fractography of conventionally heat treated specimen
B & D Fractography of cryogenically treated specimen

Fig-5 Fractography of specimens subjected to Charpy Impact Test

To characterize the nature of fracture SEM images of fractured specimens were taken at 5000X magnification. At least ten observations were made in both conventionally heat treated specimen and cryogenically treated specimen. Only few representative images have been given here. The images of conventionally heat treated specimen (Refer to Fig.5 A & C) reveals several dimples in scattered form caused by cleavage and carbides being pulled off. Presence of micro-cracks and flat facets have been observed. Scattered presence of flat facets and micro-crack indicates onset of brittleness is higher than that of cryogenically treated specimens. Also the presence of wide-ranging micro-voids point out that carbides got pulled off during rupture. Presence of equiaxed dimples, indicate that the type of failure is ductile fracture in tension.

In the fractured surface of cryogenically treated specimen (Refer to Fig.5 B & D) quasi-cleavage facets are comparatively more in population. The type of fracture is mainly due to trans granular rupture. Evidences of more fine carbides of size 1µ pulled off from these specimen. Trans granular rupture points to the fact that the bonding between grains has increased due to the precipitation of fine carbides along grain boundaries during cryogenic treatment. Also the extensive presence of cleavage facets and dimples indicate that cryogenically treated specimen has comparatively more ductility than conventional heat treated specimen.

Pin on disc wear test

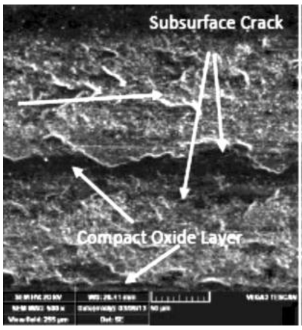
The wear pin specimens subjected to conventional heat treatment were higher than cryogenically treated specimens. It was found that sliding velocity is dominant factor compared to normal load. Up to sliding velocity of 0.6 m/s there was not much of a variation in the wear behavior between conventionally heat treated specimens and cryogenically treated specimens. Beyond sliding velocity of 0.6 m/s the wear rate increases drastically. Fig.7 shows the comparative wear rate of conventionally heat treated specimen and cryogenic treated specimen. At sliding velocity of 1.2 m/s and normal load of 75 N wear rate of conventionally heat treated specimen was 3.18 times higher than cryogenically treated specimens. Also for sliding velocity of 1.2 m/s and normal load of 125 N wear rate of conventionally heat treated specimen was 2.493 times higher than cryogenically treated specimen.

Morphology of Worn Pin

To characterize the wear behavior of both conventionally heat treated and cryogenic treated pin specimens, the scar of pin after wear test, was examined under scanning electron microscope. At least five images were taken in each specimen to study the mode and mechanism of wear. Based on the wear rate and presence of oxide particles and compact oxide layer it has been deduced that mild oxidative mode of wear was the dominant mode and mechanism of wear. Fig.6 shows the SEM images wear scar of both conventionally heat treated (CHT) specimen

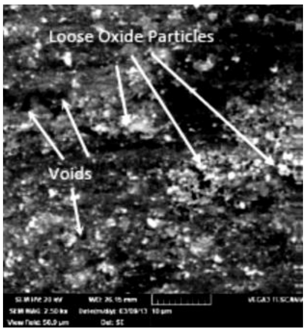
(Refer to Fig.6 A-CHT & C-CHT) and cryogenically treated (DCT) specimen (Refer to Fig.6 B-DCT & D-DCT). SEM image (Refer Fig.6 A-CHT) at 500X magnification shows very unique morphology of conventionally heat treated specimens with seemingly large layers of compact oxide in the direction parallel to sliding. Considerable plastic deformation took place and the deformation lips were stretched along the direction of sliding. Sub – surface crack are visible which grow in size and get peeled off from the surface. Presence of oxide particles at the deformation lips is clearly evident in the conventionally heat treated specimen. In the images taken at 2500X (Fig.6 C-CHT) randomly distributed voids are visible. Several instances of carbides pulled out due to shear force at the interface between pin and disc created voids.

The SEM image of cryogenically treated specimen shows a very unique pattern which is characteristically different from that of conventionally heat treated specimen. The SEM image taken at 500X indicates only a very mild surface deterioration for the same combination of sliding velocity and normal load. Morphology assist to infer that the cryogenically treated specimen resists the shear forces much better than the conventionally heat treated specimen. Also in the SEM image taken at 2500X voids are scarcely found. Similarly presence of loose oxide particles is also comparatively less. The above character of cryogenically treated specimens points to the fact that wear rate is lesser and the integrity of surface is comparatively much higher.



A-CHT


B-DCT



C-CHT

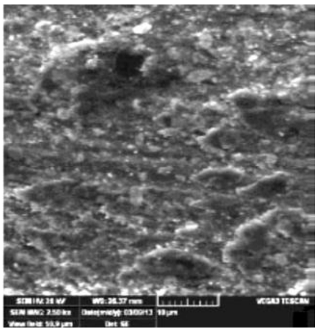

D-DCT

Fig.6.SEM image of worn surfaces of pins subjected to Conventional Heat Treatment(A&C) and Deep Cryogenic Treatment (B&D) for Sliding Velocity = 1.0 m/sec & Normal Load = 125N

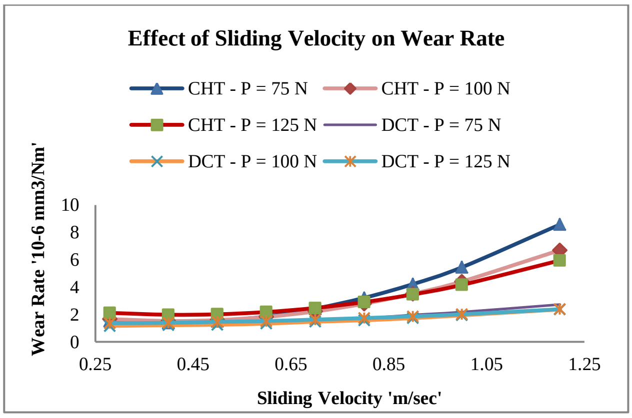


Fig.7 Effect of sliding velocity on wear rate of conventionally heat treated (CHT) and deep cryogenically treated (DCT) pin specimens

Conclusions

Outcome of the above experiments assist to infer the following conclusions

1. Deep cryogenic treatment carried out at -195 C with a dwell time of 24 hours promoted complete transformation of retained austenite to martensite. Increase in hardness of conventionally heat treated specimens from 67 to 69.5 HRC at the end of cryogenic treatment is attributed to the enrichment of martensite matrix with fine precipitates of carbides upon low temperature tempering.
2. Energy absorbed at fracture in the impact test, of conventionally heat treated and cryogenically treated specimens does not differ much indicating that cryogenic treatment does not result in undesirable brittleness. These results corroborate well with fractography analysis.
3. Within the range of test variables used for sliding abrasion wear test, in the present study, mild oxidative mode of wear was dominant. A maximum of wear rate of conventionally heat treated specimens is found to be 3.18 times more than deep cryogenically treated specimens. The main reason for the reduction in wear rate was due to the strengthening of martensite matrix by fine precipitates of carbides.
4. Fractography results assist to infer that the both cryogenically treated specimens and conventionally heat treated specimens have similar character. Presence of equiaxed dimples and cleavage facets designate ductile failure. Presence of micro cracks are comparatively higher in case of conventionally heat treated specimens than cryogenically treated specimens.

The above results assist to infer that AISI T42 has exhibited excellent hardening response under deep cryogenic treatment.

Acknowledgement

The authors acknowledge the services of Department of

Nano Technology, Periyar Maniammai University, Tamilnadu, India for their valuable suggestions and sparing lab facilities.

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