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ANALYSIS ON THE ENHANCEMENT IN MECHANICAL PROPERTIES OF SS410 BY USING CRYOGENIC TREATMENT

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INTRODUCTION

Steel finds wide range of applications in various industrial sectors. Heat treatment of steel is the most important parameter that affects properties of various grades of steel. Hardening and tempering heat treatment imparts properties of hardness, wear resistance, toughness to different grades of steel such as plain carbon steel, alloy steel, tool steel etc. One of the reason that limits the property enhancement tendency of hardening and tempering is that 100% austenite is not converted into martensite during quenching in hardening process. This results in entrapment of austenite in matrix of martensite at room temperature known as retained austenite. Retained austenite causes significant changes in mechanical properties.

LITERATURE REVIEW

In the heat treatment of steels, the problem of the retained austenite has prevailed since its development. Mohan Lalet al (2001) reported that this retained austenite is soft and unstable at lower temperatures that are likely to transform into martensite. This martensite transformation yields a 4 % volume expansion causing a distortion of the component. So, the retained austenite should be converted into martensite to the maximum possible extent, before any component is put into service. Also, the conversion of the retained austenite into hard martensite results in the improvement of the wear resistance. Over the past few decades, interest has been shown in the effect of low-temperature treatment on the performance of steels. According to Barron (1974), low-temperature treatment, in addition to or as an extension of the quench cycle, continues the process of martensite formation. Huang et al (2003) states that, as the material is chilled to lower temperatures, a greater amount of retained austenite is decomposed to martensite. Low-temperature treatment is generally classified as either ‘‘cold treatment’’ at temperatures of about -80^o C or ‘‘deep cryogenic treatment’’ at liquid nitrogen temperature (-196^o C). For simplicity, the latter is referred to as cryogenic treatment in this discussion. Cold treatment (subzero treatment), an indispensable part of the heat treatment of alloy steels, offered a significant increase in the wear resistance. It is widely accepted that a major factor contributing toward its success is the removal of the retained austenite. Conventional cold treatment has been carried out at higher than -100^o C. This temperature is believed to be sufficient to fully transform any retained austenite into martensite in the quenched microstructure. However, more recent evidence has shown that wear resistance is further enhanced by cryogenic treatment at ultra-low temperatures, such as liquid nitrogen temperature. In recent years, many small businesses have been set up to cryogenically treat finished steel products, such as drills, cutters, etc., claiming significant improvements in their wear resistance and other related properties. Despite the numerous practical successes of cryogenic treatment and research projects undertaken worldwide, no conclusive metallurgical understanding of this treatment has been established. There is no clear-cut understanding of the mechanism by which cryogenic treatment improves the performance of these steels. There are a few publications related to this field by some researchers, but they come up with different procedures and conclusions. The purpose of this review is to summarise some of the experimental works published on cryogenic treatment, to understand the mechanism by which the properties are improved and to compare the effect of the different levels of the cryogenic treatment parameters.

Tschiptschin et al (1996) studied that the nitrogen alloyed austenitic stainless steels have been successfully used in application involving pitting corrosion, crevice corrosion and stress corrosion cracking in hot chloride solutions. Bahrami et al (1995) reported that high solutionising temperature will be required to obtained a super saturated solid solution of nitrogen bearing steel. They also studied the effect of nitrogen content on heat-treated microstructure in different conditions using SEM and XRD. The stable precipitates of CrN, VN and Cr_2N occurred at tempering temperature of 400-500^oC from metastable martensitic alloys during tempering. Strength and hardness increased linearly with increasing nitrogen content upto 0.45wt % nitrogen content and superior properties were observed on tempering. Other Investigators observed the secondary hardening effect and very fine precipitates on nitrogen alloyed martensitic stainless steels during tempering

Mohan Lal et al (2001) describe cryogenic treatment as an add on process over the conventional heat treatment, in which the samples are cooled down to the prescribed cryogenic temperature level around -180^o C at a slower rate, maintained at this temperature for a long time and then heated back to room temperature. Yong et al (2006) have described cryogenic treatment as a controlled lowering of temperature from room temperature to the boiling point of liquid nitrogen (-196^o C), maintaining it at that temperature for about twenty four hours, followed by a controlled raising of the temperature back to room temperature. Subsequent tempering processes may follow. Bensely et al (2005) explain cryogenic treatment as an inexpensive one time treatment that influences the properties of the full cross section of the component, unlike surface treatment techniques like coatings. All the other authors also viewed cryogenic treatment in more or less the same way as mentioned above. Satish Kumar et al (2001) refer that cryogenic treatment is a post heat treatment process in steels, where the mass of products to be treated is cooled to very low temperature, usually around -180^o C, held at that temperature for a specific period of time, and warmed back to room temperature at a specific rate.

Leskovesek et al (2006) explained another type of cryo treatment, in which the components are directly immersed into the liquid nitrogen bath, called as cryo-quenching. In this process, after equalisation of the temperatures (i.e. when the liquid nitrogen ceased boiling) the specimens were soaked for 1 hr in the liquid nitrogen. But, as the initial temperature of the component is atmospheric, due to large variations in temperature during immersion, there is every possibility of micro cracking on the surface due to thermal shocking.

As discussed earlier, cryogenic treatment involves different steps, viz, controlled cooling to cryogenic temperature, prolonged hold at the cryogenic temperature, controlled heating back to room temperature followed by tempering. Johan Singh et al (2003, 2005) described that controlled cooling is called as ramp down, prolonged hold is called as soaking and controlled heating is called as ramp up. Then tempering follows for a predetermined time and temperature.

According to Molinary et al (2001), the most critical parameter is the cooling rate in the ramp down, which should be selected, so as to avoid rupture of the component because of the thermal stresses. Collins and Dormer (1997) observed that soaking at cryogenic temperature is also an important parameter, as the increase in the number of fine carbides in the microstructure after the cryogenic treatment is time-dependent. Next, the ramp up also has to be at a slow rate, since exposing the component during the cryogenic treatment directly to the atmosphere is analogous to dropping ice directly into water, when the ice will break. The same thing can happen to cryo-treated components also. The tempering temperature and time depend upon the materials selected. However, different researchers in their investigations used different cooling rates, soaking temperature and time, warm up rates, and tempering temperature and time.

Barron and Mulhern (1980) subjected 16 samples of the AISI-T8 and C1045 materials to cryogenic treatment involving four different levels of the cooled down rate, soak temperature and soak time, and studied the effect. They found that a cryogenic treatment consisting of a slow cooled down of 6^{o} C/min from ambient to liquid nitrogen temperatures (-196^{o} C) followed by a 24-hour soak at the cryogenic temperature resulted in superior wear resistance. But in another study by Barron (1982), he used 3^{o} C/min, cooling rate and claimed an improvement in the wear resistance. Collins and Dormer (1997), Collins and Rourke (1998) first cooled the material from atmospheric temperature to -140^{o} C by a controlled cooling rate of 2.5^{o} C/min, and then, cooling from -140^{o} C to -196^{o} C was carried out by immersion in liquid nitrogen. They warmed up the treated component by exposing it to still air at ambient temperature. Molinari et al (2001) suggested 20-30^{o} C cooling rate per hour and 35 hours soaking time at liquid nitrogen temperature. Mohan Lalet al (2001) made extensive studies on the effect of different parameters at different levels in tool steels and concluded that materials treated at -180^{o} C and for a soak period of 24 hours are better. Flavio et al (2006) in their studies used 1o C/min cooling rate, 20 hours soak at temperature -196^{o} C and warm up rate of 1o C/min. Johan Singh et al (2005) suggested ramp down times in the 4-10 hour range, soak temperature of -185^{o} C, soak period of 20-30 hours and ramp up period of 10-20 hours. Barron and Mulhern (1980) suggested 1^{o} C/min cooling rate, 24 hours soaking and 6 hours ramp up period. But comparing the end results of all the researchers, a better performance of cryogenically treated components is obtained with 1^{o} C/min cooling rate, 24 hours soaking and 6 hours ramp up period. The tempering temperature and time differ from material to material.

In almost all the references it is mentioned, that the retained austenite is converted into martensite during the cryogenic treatment. In the investigations of Barron and Mulhern

(1980), Barron (1982), cryogenic treatment improves the resistance of the material to wear, due to the conversion of austenite into martensite, and this extends the useful life of the components. The hardness of the cryo-treated materials improved by 5 %.Moore and Collins (1993) found that increase in hardness was dependent on cryogenic temperature and that can affect the obtainable hardness. Huang (2003) found that cryogenic treatment can facilitate the formation of carbon clustering and increase the carbide density in the subsequent heat treatment, thus improving the wear resistance of steels. Mohan Lal et al (1996, 2001) in their studies, stated that this conversion increases the dimensional stability and toughness of the component. The cryo-treated tools have good surface finish. The red hardness of the D3 steel is also improved by the cryogenic treatment.

Wu Zhisheng et al (2003) applied cryogenic treatment to electrodes for spot welding, which improves the electrical and thermal conductivity. They also found that the electrode life is improved from 550 to 2234 welds by deep cryogenic treatment. Myeong et al (1997) observed that the high cycle fatigue life of austenitic stainless steel was increased greatly after cryogenic treatment without a decrease in the ductility. Johan Singh et al (2003,2005) have shown that the fatigue life of AISI 304L fillet and cruciform welded joints has been improved after the cryogenic treatment. Relief of residual stresses is also a benefit of the cryogenic treatment. Mahmudi et al (2000) described that the transformation of retained austenite, which is largely complete in more steels in the temperature range of -70^{o} C to -110^{o} C, results in better dimensional stability, higher hardness value, lower toughness and very modest improvement in the wear resistance. From the above discussion it can be inferred that the life of the cryogenic treated components is improved.

Thomas (1986) claimed that cryogenic treatment removes the kinetic energy of atoms, which is the energy of motion. There is a normal attraction between atoms that makes them wants to get together, but their energy of motion keeps them apart unless the energy is quelled by low temperature cooling. This final treatment at below -184^{o} C in a dry atmosphere transforms the retained austenite into the harder, more desirable, martensite. During this transformation, smaller carbon carbide particles are released and distributed evenly through the mass of the material. These smaller particles are in addition to the larger carbon carbides present before the cryogenic treatment. These smaller carbon carbide particles help to support the martensite matrix. In cutting tools, this reduces the heat build upon the cutting edge; this in turn, increases the wear resistance and the red hardness of the tool.

The results of this study indicate that metals such as tool steels, which can exhibit the retained austenite at room temperature, can have the wear resistance significantly increased, by subjecting the metal to a long soak at temperatures of the order of -196^{o} C. This lower temperature treatment was preferable to a soak at -196^{o} C for the stainless steels; however, the -84^{o} C soak was satisfactory in improving the wear resistance by as much as 25 %. The wear resistance of plain carbon steels and cast iron was not significantly affected by the low temperature treatment.

Wu Zhirafar et al (2007) investigated the effect of cryogenic treatment on the mechanical properties and microstructure of the AISI 4340 steel, and inferred that the hardness and fatigue strength of the cryogenic treated specimens were a little higher, whereas the toughness is reduced due to the cryogenic treatment.

EXPERIMENTAL SETUP

Grade 410 stainless steels are general-purpose martensitic stainless steels containing 11.5% chromium, which provide good corrosion resistance properties. However, the corrosion resistance of grade 410 steels can be further enhanced by a series of processes such as hardening, tempering and polishing. Quenching and tempering can harden grade 410 steels. They are generally used for applications involving mild corrosion, heat resistance and high strength.

The compositional ranges of grade 410 stainless steels are displayed below.

Table 1 - Composition ranges of grade 410 stainless steels

Grade		C	Mn	Si	P	S	Cr	Ni
410	min.	-	-	-	-	-	11.5	0.75
	max.	0.15	1	1	0.04	0.03	13.5	

CRYOGENIC TREATMENT

Cryogenics is defined as the branches of physics and engineering that study very low temperatures, how to produce them, and how materials behave at those temperatures. Rather than the familiar temperature scales of Fahrenheit and Celsius, cryogenicists use the Kelvin and Rankine scales. The word cryogenics literally means "the production of icy cold"; however the term is used today as a synonym for the low-temperature state. It is not well-defined at what point on the temperature scale refrigeration ends and cryogenics begins. The workers at the National Institute of Standards and Technology at Boulder, Colorado have chosen to consider the field of cryogenics as that involving temperatures below –180 °C (93.15 K). This is a logical dividing line, since the normal boiling points of the so-called permanent gases (such as helium, hydrogen, neon, nitrogen, oxygen, and normal air) lie below -180 °C while the Freon refrigerants, hydrogen sulphide, and other common refrigerants have boiling points above -180 °C.

TREATMENT PROCEDURE

The liquid nitrogen as generated from the nitrogen plant is stored in storage vessels. With help of transfer lines, it is directed to a closed vacuum evacuated chamber called cryogenic freezer through a nozzle. The supply of liquid nitrogen into the cryo freezer is operated with the help of solenoid valves. Inside the chamber gradual cooling occurs at a rate of 2^{o} C /min from the room temperature to a temperature of -80^{o} C. Once the sub zero temperature is reached, specimens are transferred to the nitrogen chamber or soaking chamber where in they are stored for 24 hours with continuous supply of liquid nitrogen. **Fig4.4** illustrates the entire set up for cryogenic treatment. The entire process is schematically.

MECHANICAL PROPERTY TEST

SEM TEST

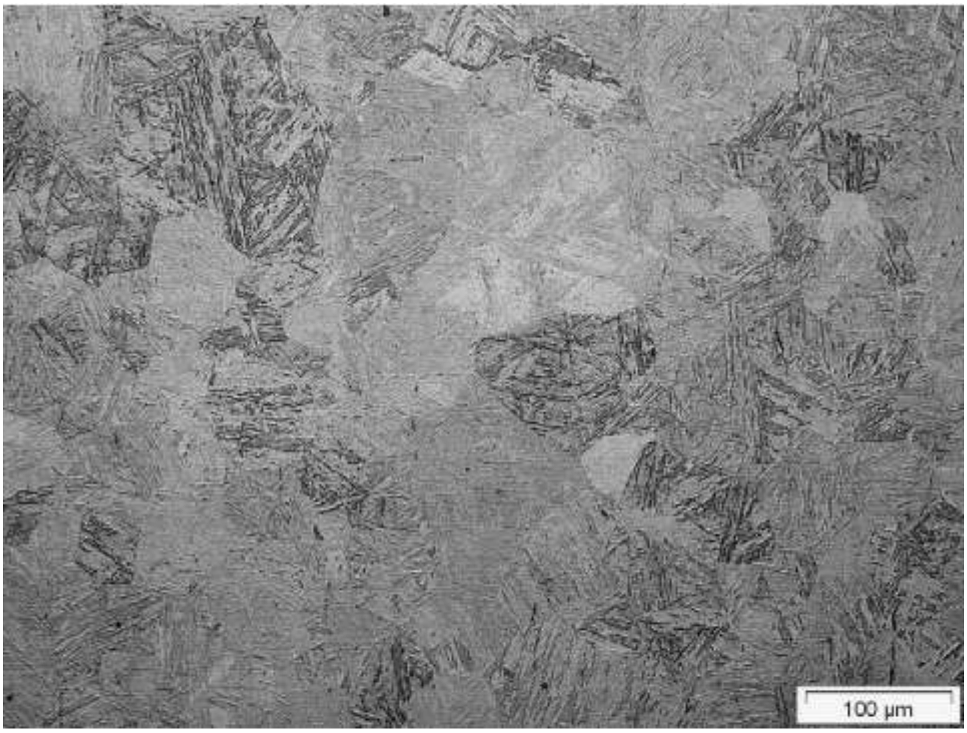

Fig:4.5 Model SEM image of SS410

TEST SPECIMENS AND SAMPLE PREPARATION

Materials

This test method may be applied to a variety of materials. The only requirement is that specimens having the specified dimensions can be prepared and that they will withstand the stresses imposed during the test without failure or excessive flexure. The materials being tested shall be described by dimensions, surface finish, material type, form, composition, microstructure, processing treatments, and indentation hardness (if appropriate).

Test Specimens

The typical pin specimen is cylindrical or spherical in shape. Typical cylindrical or spherical pin specimen diameters range from 2 to 10 mm. The typical disk specimen diameters range from 30 to 100 mm and have a thickness in the range of 2 to 10 mm.

TEST PARAMETERS

RESULTS AND DISCUSSION

5.1 SEM TEST

Micrograph and analysed micrograph of deep cryogenic treated sample are shown in the following figure5.1 The anlysed micrograph data of DCT sample is presented . This microstructure shows larger amount of martensite at tempered condition with finely dispersed precipitated carbide particles. It is understood from the figure5.2 that the presence of retained austenite decreased as a result of cryogenic heat treatment

Non treated sample

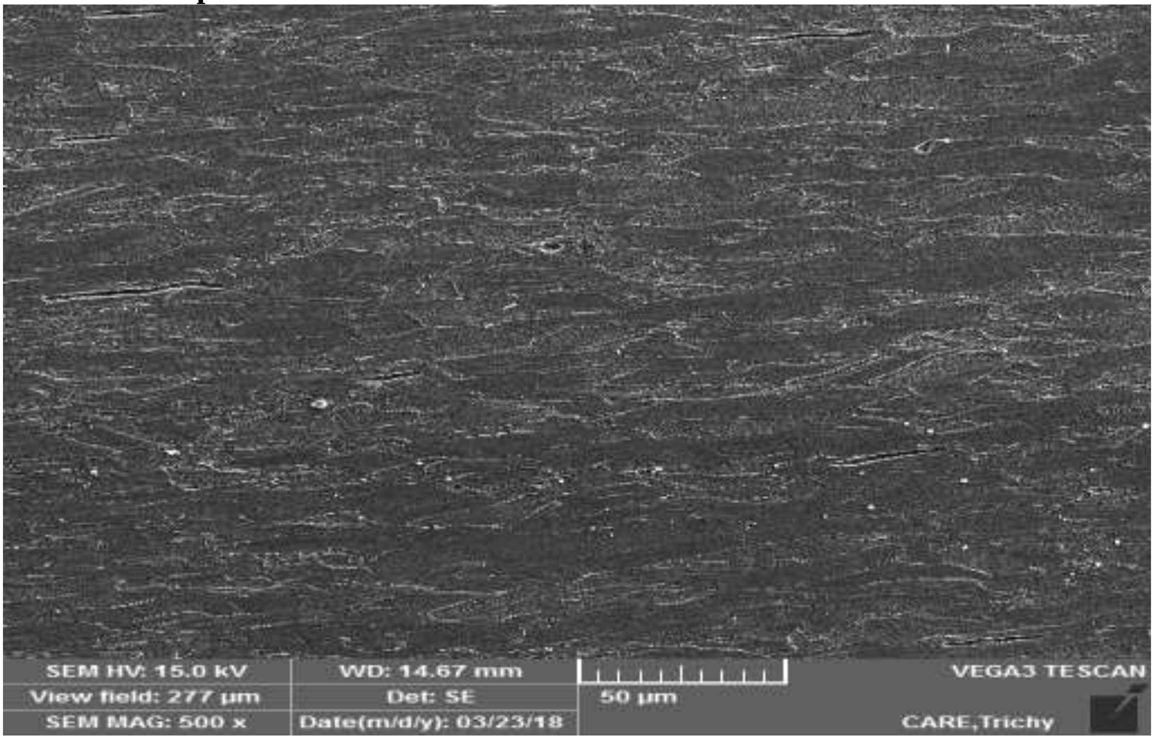

Fig 5.1. Non treated SEM test image

Treated sample

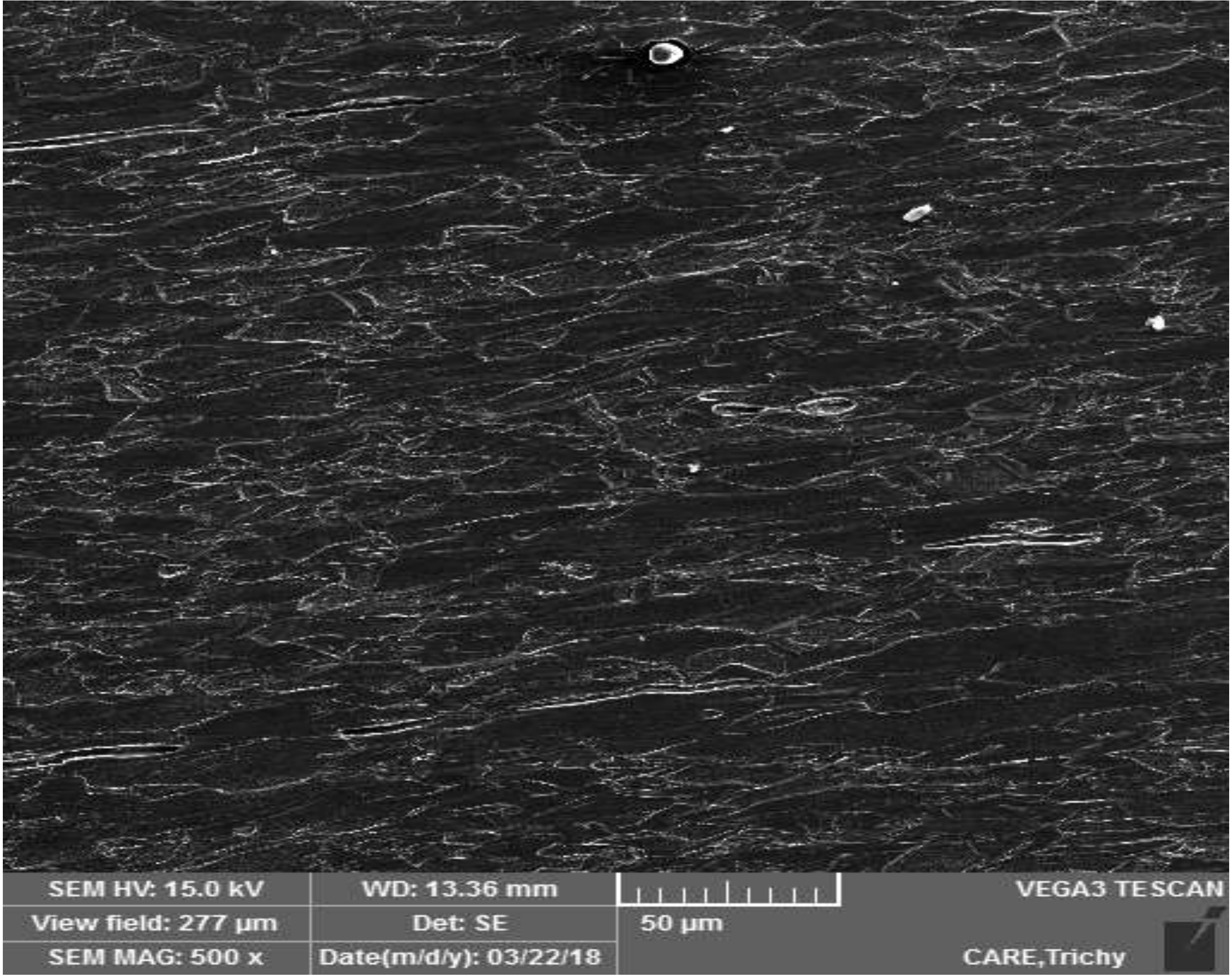

Fig 5.2 treated SEM test image

5.2 WEAR TEST

TABLE 5.2 The pin on disc wear tests indicate enhancement of wear resistance

Material	Test specimen	Wear(mm^3/min)
SS410	Non treated	0.0018
SS410	Treated	0.0009

Even though the wear resistance has increased with duration of cryotreatment process, the enchancement has not been very significant. This can be attributed to various factors like variation in percentage composition of alloying elements, heat treatment process followed post production,etc

HARDNESS TEST

TABLE 5.3 The hardness(HRC)of the untreated and the cryotreated specimen are

Material	Test specimen	Hardness (HRC)
SS410	Non treated	87
SS410	treated	105

Cryotreatment process increase the hardness value. During this treatment ,the micro sized hard carbide particles are released which fill up the gaps and vacancies present with in the atomic structure of the material . Filling of these carbide particles causes the atomic structure to be more dense ,strong and hard . Increasing the duration of the soak during cryotreatment has resulted in increased hardness which indicates that enhancement in hardness depends on the duration of cryotreatment.

CONCLUSION

- Comparative study on the hardness and toughness of cryogenically treated SS 410 with that of untreated SS 410.
- In the sliding wear test, the weight loss of cryogenically treated SS 410is more as compared to that of untreated SS 410.
- By this technique specially hardness, wear resistance, corrosion resistance, toughness increases. Cryogenics materials will be part of the dynamic future.
- We must not only continue to make incremental improvements in present materials but develop whole new technologies of manufacturing and processing for to achieve the highest performance in cryogenics materials field.
- Cryogenics-based technologies have applications in wide variety of areas as metallurgy, chemistry, power industry, medicine, rocket propulsion and space simulation, food processing.

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