Effects of Cryogenic Treatment on the Strength Properties of Heat Resistant Stainless Steel (07X16H6)

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Abstract. Cryogenic treatment on metals is a well known technology where the materials are exposed to cryogenic temperature for prolonged time duration. The process involves three stages viz. slow cooling, holding at cryogenic temperature and warming to room temperature. During this process, hard and micro sized carbide particles are released within the steel material. In addition, soft and unconverted austenite of steel changes to strong martensite structure. These combined effects increase the strength and hardness of the cryotreated steel. In this experimental study, the effects of cryogenic treatment, austenitising and tempering on the mechanical properties of stainless steel (07X16H6) have been carried. After determining the strength properties of the original material, the specimens were cryotreated at 98K for 24 hours in a specially developed cryotreatment system. The effects of austenitising prior to cryogenic treatment and tempering post cryotreatment on the mechanical properties of steel samples have been experimentally determined and analysed.

1. Introduction
The subzero treatment on metals has been extensively employed since many decades for various applications. However this treatment was restricted to about 193K (dry ice temperature). In the recent years extensive research has been carried out in studying the effect of the cold treatment down to cryogenic temperatures by varying operating parameters [1]. Even though major application of cryogenic treatment has been in the field of tooling industry in improving the tool life of the cutting tools, it has other important applications like reduction of internal stresses in metals, imparting dimensional stability to gauges and precision machined parts, enhancement of strength properties, etc. Cryogenic treatment follows a procedure where the test samples are cooled down to cryogenic temperature at a definite rate, held for longer time duration and warmed to room temperature at a definite rate. This cryo treatment cycle can be operated for various conditions of temperature, duration, rate of cooling and warming. A typical cryotreatment cycle is shown in figure 1.
During the cryotreatment process on steels, hard and micro sized carbide particles are released within the material and they are evenly spread over the entire volume. In addition, soft and unconverted austenite of steel changes to strong martensite structure [2] [3]. These combined effects tend to increase the strength and hardness of the cryotreated steel [4] [5]. In this experimental study, the effects of cryotreatment on the mechanical properties viz. strength (tensile and impact) and hardness of stainless steel (07X16H6) are studied.

2. Development of cryotreatment unit

In order to cryotreat the steel samples, a cryotreatment unit was designed and developed with capability to cryotreat for various rates of cooling, soak and warming periods. This developed cryotreatment unit has the unique feature that the test samples are be cooled to cryogenic temperature by indirect cooling without causing thermal shocks by spray of LN2 droplets.

The cryotreatment system incorporates mainly the cryotreatment chamber and an auxiliary LN2 supply system to supply controlled quantity of LN2 to the chamber to maintain the rate of cooling, soaking and warm up. The controlled LN2 supply to the chamber is carried out by using a solenoid valve activated by a PID controller. The Schematic diagram of the cryotreatment system is as shown in the figure 2.
wall of the metallic shroud housed around the meshed tray. The photograph of the developed unit is shown in figure 3.

![Figure 3. Photograph of the cryotreatment unit.](image)

The LN2 supply is regulated by a solenoid valve operated by a PID controller with predetermined set points. Various cryotreatment cycles can be programmed to suit the requirements. The temperatures of the specimens are measured using Platinum Resistance Temperature Detectors (RTD).

3. Cryotreatment trials and tests

In order to study the effects of cryotreatment on mechanical properties, trials were conducted on stainless steel (07X16H6). This is a corrosion resistant stainless steel used for general engineering applications. The valve material for LH2 and LOX in cryogenic engine are produced by using this selected grade of steel. The material was machined to produce test samples for the Tensile, Hardness and Impact tests as per ASTM standards E8, E10 and E23 respectively [6] [7] [8]. The machined test samples were categorized into four different groups in order to try various combinations of cryotreatment and heat treatment processes. It was ensured that each batch has a minimum of 6 samples in order to obtain authentic and reliable results. The samples were divided into 4 batches as under:

- Batch A: Raw material (in annealed) condition.
- Batch B: Cryotreatment alone at 98K for 24 hours.
- Batch C: Cryotreatment at 98K for 24 hours followed by tempering at 400 Celsius for 1 hour.
- Batch D: Austenitisation at 990 Celsius for 1 hour followed by cryotreatment at 98K for 24 hours and tempering at 400 Celsius for 1 hour.

In order to get the reference values of the mechanical properties of the selected material, tests were conducted to determine the strength (both tensile and impact strength) and hardness values [7] [8]. At the same time, microscopic structures were obtained using high resolution optical metallurgical microscope. (Model: NIKON-Epiphot 200) The test specimens were machined as per the specifications of ASTM E8M to carry out tensile strength properties. The tests were conducted on servo hydraulic dynamic test system (Make: Instron; Model: 1341). Properties viz. Ultimate tensile strength, Yield strength and percentage elongations were determined using the test system. Test samples for impact tests were machined in accordance with as per ASTM E23. Charpy impact strength were carried on FIT 300(EN) system. Hardness values (HRC) were also determined as per ASTM E 10.

After obtaining the initial data, the test samples of batch D were austenitised at 990 Celsius for 1 hour. All the samples (batches B, C and D) were successfully cryotreated in the newly developed cryotreatment unit at 98K for 24 hours with 4 hours of cool down and 9 hours of warm up time to
ambient condition. The entire cycle consumed around 260 litres of liquid nitrogen which was supplied to the unit at 22 psi from pressurized container. The temperatures of the samples were monitored throughout the run. After the cryotreatment run the samples of batch A and C were tempered at 400 Celsius for 1 hour.

All the samples of batches B, C and D were again subjected to tensile, impact and hardness tests (including microstructure study). These readings were compared with the original test results and comparative study was carried out.

4. Results and analysis

Comparative studies of test results were carried out. The results of the study are as under:

4.1. Tensile strength

The test results are listed in the table 1.

| Batch | A   | B   | C   | D   |
|-------|-----|-----|-----|-----|
| UTS (MPa) | 1954 | 1902 | 1987 | 2509 |
| YS (MPa)  | 1901 | 1834 | 1949 | 2404 |
| % Elongation | 16.13 | 17.89 | 23.7 | 9.29 |

The results clearly indicate significant increase in tensile strength of the samples of batch D compared to the raw material samples (batch A). This is due to the conversion of soft and ductile retained austenite into hard and strong martensite. At the same time, the micro-sized carbide particles are precipitated during cryotreatment and fill the voids between atoms. During tempering still finer nano sized carbide particles known as η particles further fill up the voids and make the structure much more dense and strong [9]. Not much significant changes were observed in strength properties of batch B and C.

4.2. Hardness

The test results are listed in table 2.

| Batch | A   | B   | C   | D   |
|-------|-----|-----|-----|-----|
| Hardness (HRC) | 35  | 37  | 38  | 41  |

The hardness values were recorded using the hardness tester with scale C, using diamond cone indenter with load of 150Kg. Due to precipitation of hard carbide particles both during cryotreatment and tempering, samples of batch A showed marked increase in hardness values. However, improvements in batches B and C were marginal.

4.3. Impact strength

The test results are listed in table 3.

| Batch | A   | B   | C   | D   |
|-------|-----|-----|-----|-----|
| Impact strength | 50.4 | 47  | 27  | 23.4 |

Extending the concept of formation of hard martensite and precipitation of hard carbide fillers it is expected that the material becomes harder and absorbs less energy during impact loading (Charpy). In the case of batches A and C (subjected to tempering operation in addition to cryotreatment), the results indicate proportional changes with regard to energy absorbed before failure [10].
4.4 Microstructure study
The photographic images of the microstructures were obtained using the metallurgical microscopes. The images are shown in the figure 4.

![Microstructure images](image)

**Figure 4. Photographs of microstructure**

Comparing the microstructures of all the samples the samples of batch D indicated marked presence of martensite plates nucleating at the grain boundaries of austenite region. No major observable changes were found in batches B and C compared with samples of batch A.

5. Conclusions
The entire experimental study was carried out on all the feasible combinations of austenitising, cryotreatment and tempering. It is clearly evident that the process of cryotreatment can render significant changes when the samples are subjected to austenitising before cryotreatment and tempering post cryotreatment.

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