

# CRYOGENIC PROCESSING OF HSS M2: MECHANICAL PROPERTIES AND XRD ANALYSIS

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**Abstract.** The purpose of this study was to reveal the changes in properties of deep cryogenic treatment (DCT) processed high speed steel, in comparison to conventional heat treatment for secondary hardness. Tool properties were assessed in terms of hardness, and tensile strength. Statistically significant enhancement in the mechanical properties was observed. Cryogenic processing of HSS tool steel eliminated the retained austenite, and hence increased the hardness of the material. This treatment initiated nucleation sites for precipitation of large numbers of very fine carbide particles. Tensile values for cryogenically treated HSS samples can be attributed to the fact that the tool becomes more brittle after the treatment. XRD analysis illustrated the contraction in lattice of martensite and austenite. Deep cryogenic treatment practically removed all traces of austenite in the sample. The superior performance of cryogenically treated HSS can be attributed to the transformation of almost all retained austenite into martensite, a harder structure and precipitation of fine and hard carbides.

## 1 Introduction

Recently, application of cryogenic treatment in enhancing properties of tool materials has received wide acceptance by researchers and industries. Research has shown that cryogenic treatment increases product life, and provides additional qualities to the product, such as stress relieving[1,2]. Tool life is a major factor that is considered in production of finished product in manufacturing industry as any improvement in tool life will have a direct impact on the cost of production, tool changing time and maximum production target. It has been reported that cryogenic treatment can double the service life of HSS tools, and also increase hardness and toughness simultaneously [3].

Tool steel (carbon and alloy steels) having distinctive hardness, resistance to abrasion, ability to hold a cutting edge, and resistance to deformation at elevated temperatures, are generally used for cutting tools, punches, and other industrial tooling. Different tool steels are developed to resist wear at temperatures of forming and cutting applications.

Cold work tool steels are specific for tool bits, dies and other applications where high wear resistance and low cost are required [3].

Tool steel is generally used in a heat treated state. Conventional heat treatment gives hardness, wear resistance and ductility to steel. However, it cannot remove all of the retained austenite (large, unstable particles of carbon carbide) from steel. The retained austenite (a soft phase) in steels could reduce the product life and affects the working. Therefore, there is a need for transformation into martensite. 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 °C or “deep cryogenic treatment” at liquid nitrogen temperature (-196°C). 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 (-500c to-1960c) and holding there for sufficiently long duration (20 to 75

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h) before being heated back to the ambient temperature at a predetermined rate for subsequent tempering treatment [4,5]. Cryogenic treatment improves wear resistance, hardness, toughness, resistance to fatigue cracking, microstructure of metal (retained austenite to martensite), dimensional stability and decreases residual stresses. Cryogenic treatment gives machining, grinding and polishing finish due to little soft austenite[6].

In this direction, the present experiment was carried out to assess the enhancement in the mechanical properties of HSS tool and the compositional analysis by XRD analysis.

## 2 Experimental

### 2.1 Materials Selected:HSS M2

### 2.2 Manufacturing of Tool Bits

CuttingGrinding→Heat→ treatment→Final grindingTOOL BITS

### 2.3 Heat processing of Tool Bits

#### Conventional heat treatment

The conventional heat treatment for the M2 steel tool bits was conducted based on the alloy heat treatment. Hardening (austenitizing) at 1123 K (850 °C) for two and half hour, is followed by oil quenching and tempering at 723 K (450 °C) for one hour.

#### Deep Cryogenic Treatment

The HSS tool bits steel which had undergone the conventional hardening and were slowly cooled from room temperature to 85 K (-196°c), soaked at 85 K for 24 h, and finally heated back to room temperature. The processor is a well-insulated chamber with liquid nitrogen as the working fluid. The cryogenic processor consists of a treatment chamber, which is connected to a liquid nitrogen tank through a vacuum insulated hose pipe. The liquid nitrogen passes through the spiral tubes of heat exchanger and enters the duct leading to the bottom of the chamber as nitrogen gas (evaporated state). The blower at the top of the chamber sucks the gas coming out at the bottom and makes it to circulate inside the chamber and reduces the chamber temperature drastically. The samples taken out from the processor were tempered at 473 K (200°C) for an hour.

### 2.4 Composition analysis

The composition of tool bits from HSS steel for various elements was assessed. (Instrument used: Spark Emission Spectrometer BAIRD, DV6, USA)

## 2.5 Mechanical Properties

### Hardness test

Rockwell hardness testing (fig.) method was used for measuring the bulk hardness of metal. (Digital Rockwell Hardness, Make: FIE, Model: RASNET-1, Resolution; 0.1 HRc, Sr No 03/2006-1339). This method consists of indenting the test material with a diamond cone or hardened steel ball indenter. The indenter is forced into the test material under a preliminary minor load usually 10 kgf. The permanent increase in depth of penetration, resulting from the application and removal of the additional major load is used to calculate the Rockwell hardness number for the following test samples:

Tool Steel: Raw

→CHT (Conventional Heat Treated)  
→DCT (Deep Cryogenic Treated)

### Tensile test

The tensile test is to determine the ultimate tensile strength. As per the IS 1608-2005 test method, the tensile test is carried out for the HSS Tool bits specimens (Raw, CHT and DCT). The specimen (bone shaped) is gripped in the tensile testing machine (i.e. Universal Testing Machine UTM, Capacity 500KN, resolution 1N, Make MTS, USA). After clamping the specimen on the holder the machine is switched on. The load is applied on the specimen along the uniaxial direction. Tensile strength was noted in Mpa.

## 2.6 XRD Analysis

X-ray diffraction (XRD) relies on the dual wave/particle nature of X-rays to obtain information about the structure of crystalline materials. A primary use of the technique is the identification and characterization of compounds based on their diffraction pattern.

## 3 Results and Discussion

**3.1Composition:** Table 1 depicts the concentration of various elements present in M2 HSS

Table 1. Chemical composition of HSS M2

Element s	Concentratio n (%)
C	0.68
Si	0.33

<b>Mn</b>	<b>0.33</b>
<b>P</b>	<b>0.026</b>
<b>S</b>	<b>0.004</b>
<b>Ni</b>	<b>0.27</b>
<b>Cr</b>	<b>4.16</b>
<b>Mo</b>	<b>4.91</b>
<b>V</b>	<b>1.87</b>
<b>W</b>	<b>5.89</b>

### 3.2 Hardness

Table 2 illustrated the hardness (HRC) of HSS Tool bits samples (raw, CHT and DCT) assessed by Rockwell Hardness tester. The mean values pertaining to hardness were 29.5, 65.5 and 68 HRC for raw, CHT and DCT tool bits, respectively; depicting about 3.81% improvement in the hardness of the tool bits on cryogenic processing. Increase in hardness of cryogenically treated samples is due to the conversion of retained austenite to martensite and due to the presence of fine carbides in the metal matrix. Most researchers believed that there are two mechanisms to improve the mechanical properties of the work that has been treated cryogenically. [7,8] The first mechanism is attributed to the transformation of retained austenite to martensite. The second is to initiate the nucleation sites for precipitating a large number of fine carbides in the matrix of martensite. [9]

**Table 2.** Hardness (HRC) of HSS tool bits

Type of sample	Hardness (HRC)		
	Value 1	Value 2	Mean
<b>Raw</b>	29	30	29.5
<b>CHT</b>	65	66	65.5
<b>DCT</b>	68	68	68.0
<b>Percent improvement in hardness 3.81</b>			

CHT- Conventional heat treated; DCT- Deep cryogenic treated

### 3.3 Tensile strength

The tensile strength of raw and CHT tool bits was 931.9 and 1420.0 MPa, respectively as inferred from the table 3. The cryogenic treated tool bits became very brittle showing the significant effect in quality of the tool bits. Our results are in confirmation with

the earlier reported studies. [10-11] Cryogenic treatment in tool steels causes the precipitation of finely dispersed carbides in martensite and also converts soft unstable austenite to martensite.

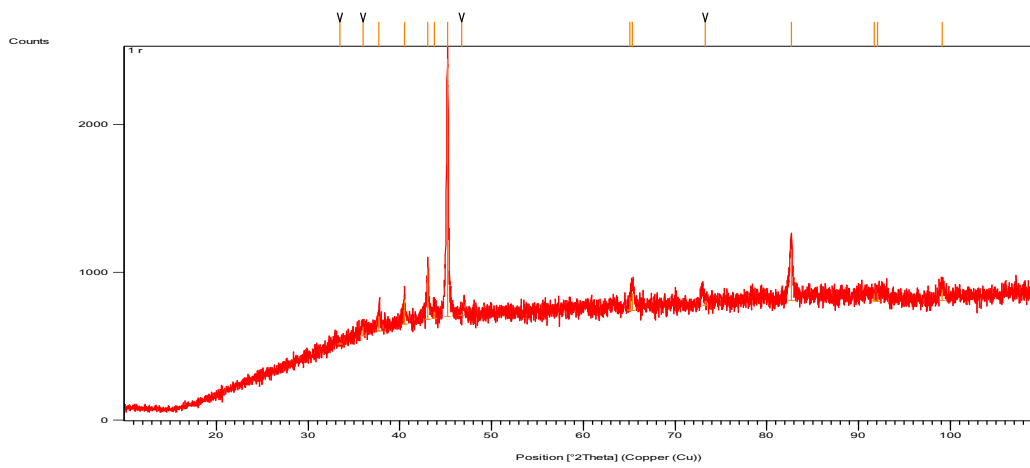
**Table 3.** Tensile Strength (MPa) of HSS tool bits

Type of sample	Tensile Strength (MPa)
<b>Raw</b>	931.9
<b>CHT</b>	1420.0
<b>DCT</b>	(very brittle)
<b>Percent improvement Tensile Strength 52.37</b>	

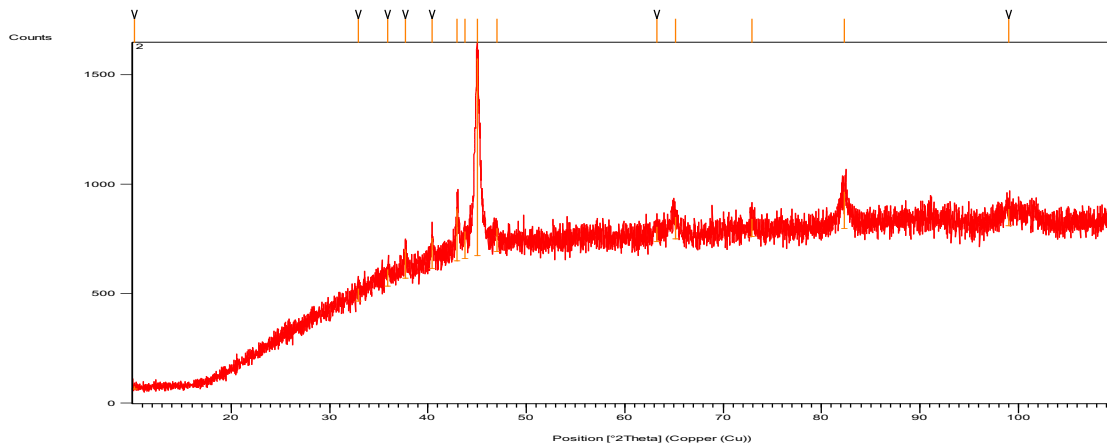
CHT- Conventional heat treated; DCT- Deep cryogenic treated

### 3.4 XRD analysis

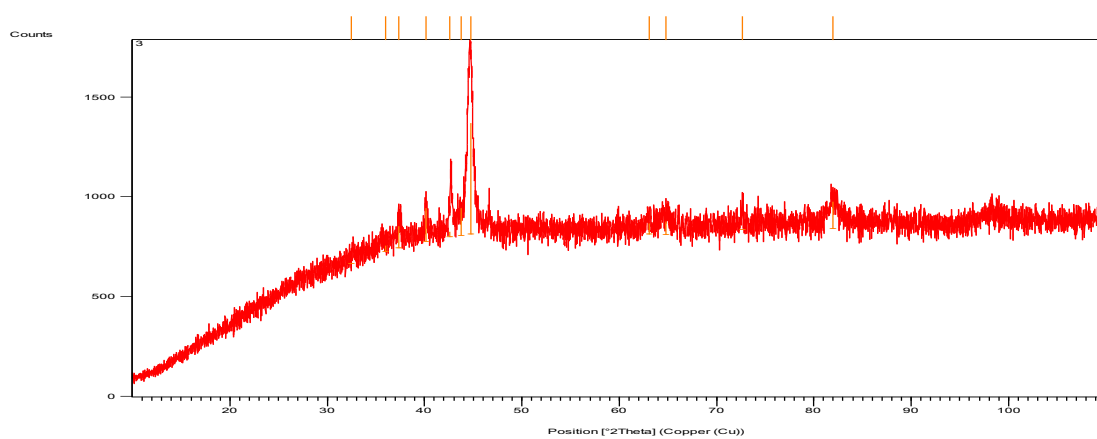
To monitor austenite content, X-ray diffraction method has been found to be most effective and accurate. The phases present in the Conventional Heat Treated and Cryogenic Treated Specimens have been studied using X-Ray Diffractometer for identification and characterization of compounds based on their diffraction pattern. The X-Ray diffraction pattern (Fig.1-3) showed that austenite, martensite and carbides of chromium and tungsten are the major phases present in the sample after conventional heat treatment. Amount of retained austenite present at the end of conventional heat treatment was found to be 15%. Presence of retained austenite deep cryogenic treatment has been found to be 4-5% respectively. During cryogenic treatment contraction in lattice of martensite and austenite took place. Due to super saturation martensite with carbon and thermodynamic instability the carbon atoms squeezed out of martensite, migrated to the neighbouring lattice defects and acted as nucleation sites for the growth of fine carbides. Upon tempering fine precipitates of carbides were formed, with concurrent softening of martensite. The X-Ray diffraction pattern (Fig.5) corresponding to deep cryogenic treatment showed no traces of austenite but increase in percentage of carbides by about 2%.



**Fig:1** X-Ray Diffraction Pattern of raw M2 HSS



**Fig:2** X-Ray Diffraction Pattern of Conventional Treated M2 HSS



**Fig.3.** X-Ray Diffraction Pattern of Deep Cryogenic Treated M2HSS

## 4 Conclusion

The lower temperature processing of HSS M2 leads to formation of a higher carbide concentration which results in enhanced hardness. XRD analysis reveals that after the cryogenic treatment the microstructure of material is refined, retained austenite is completely converted in martensite and more number of secondary carbides precipitate on the surface. Therefore, cryogenic processed HSS M2 tool exhibit better in terms of industrial applications.

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