Effect of Cryogenic Treatment on Various Tool Steels: A Review

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Abstract- *Cryogenic treatment (CT) is a thermal treatment process in steels, by deep-freezing materials at cryogenic temperatures to enhance the mechanical and physical properties of treated materials being treated .Various advantages, increase in hardness, increase in wear resistance, reduced residual stresses, fatigue Resistance, increased dimensional stability, increased thermal conductivity, toughness, by transformation of retained austenite to martensite, the metallurgical aspects of eta-carbide formation, precipitation of Ultra fine carbides, and homogeneous crystal structure, Different approaches have been applied for CT to study the effect on different types of steels and other materials. Steels by application of appropriate types of CTs from cryogenic conditioning of the process. The conclusion of the paper discusses the development and outlines of the trends for the research in this field.*

Keywords- Cryogenic Treatment, Deep Freezing, Increase Hardness, Increase Wear Resistance, Homogeneous crystal structure.

I. INTRODUCTION

Cryogenic treatment (CT) of tool materials consists of three stages, that involves cooling of tool material from room Temperature, at an extremely slow rate ranging from 0.5 to 1 ^{0}C/ min, to temperature as low as -84^{0}C, for shallow cryogenic treatment (SCT) and 196^{0}C for deep Cryogenic treatment (DCT), followed by soaking for a period ranging from 24 to 36 hours and finally heating up at the rate of 0.5 to 1^{0}C/ min, to room temperature, though cryogenic treatment you have been around for many years it is truly in its infancy when compared to heat-treating. On the use of CT on tool materials are spotty and subjective .Therefore it requires rigorous experimentations and investigations to ascertain and evaluated the process before commercial exploitation could begin.

Cryogenic treatment involves the following sequence:

1. Slow cooling to predetermined low temperature.
2. Soaking for predetermined amount of time
3. Slow heating to room temperature
4. Tempering Before proceding for cryogenic treatment.

The batch of heat treated specimens was cleaned to remove the dirt, impurities and traces of salt layer found on their surface.

1.1 SHALLOW CRYOGENIC TREATMENT

Shallow cryogenic treatment has been carried out at -85 ^{0}C. Since rate rate of cooling is a very sensitive factor and it seriously affects the results of Cryogenic treatment, the specimens were very slowly cooled at the rate of -0.5 ^{0}C /min until they reach the fine soaking temperature of -85^{0}C . A soaking period of 8 hours was taken to allow for reactions to take place after which the cycle was reversed such that temperature builds up at the rate of 0.5^{0}C / min up to room temperature.

1.2 DEEP CRYOGENIC TREATMENT

Deep cryogenic treatment has at -195 °C with a soaking time of 24hr. Specimens were cooled at the rate of -0.5 ° C/min until they reach the final soaking temperature of -195 °C. Soaking time of 24 hours was taken to allow for complete phase transformation to take place. Then the cycle was reversed that temperature ramp up at the rate of 0.5°C/min up to room temperature.

1.3 LOW TEMPERATURE TEMPERING

Tempering at 200˚C with 90 minutes soaking is essentially to be followed after cryogenic treatment. The carbon diffused during cryogenic treatment forms aggregates. Since the martensite resulting from transformation of retained austenite during cryogenic treatment results in brittleness and also as there is a 4% volumetric expansion during the transformation of austenite internal stresses creep in. To alleviate brittleness, relieve internal stresses and to allow for precipitates of fine carbides specimens were double tempered in forced air circulation furnace.

Though addition of alloying elements conforms the desired cutting characteristics making it suitable for a specific metal cutting application, it adds to the cost of the material with increasing content of cobalt and tungsten. In addition to alloying, further enhancement in wear resistance and tool life is made possible by use of hard abrasive coating, over the functional part of the tool .But, once the abrasive layer wears out the wear resistance switches back to that of plain tool. Cryogenic Treatment (CT) is another option available that helps improve the wear resistance and life of tool, by bringing about property changes across the entire volume of the material unlike the coated tools where in the enhancement in wear resistance takes place only at the surface of the tool.

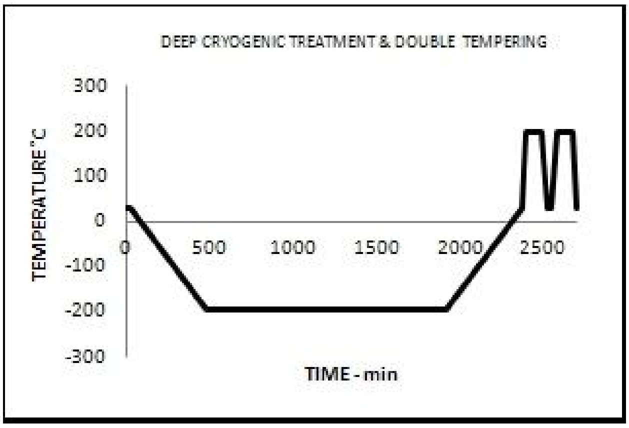

Fig-1: Cryogenic Treatment Applied to Conventionally Heat Treated Metal

II. LITERATURE REVIEW

Cryogenic treatment refers to the treatment of materials at very low temperatures generally around 196^{0}C which is much lower than cold treatment where temperatures are around -96 ^{0}C. The appreciable changes include the changes in the mechanical properties and in crystal structures of materials. However survey of literature shows various points such as , D.Candane had conducted the test on AISI M35 HSS and observed that ,19% of retained Austenite present at the end of conventional heat treatment was reduced to 5% at the end of shallow cryogenic treatment while deep cryogenic treatment removes all traces of asutenite in the sample. Fine precipitates of carbides of size 0.3-0.5µ were observed in cryogenically treated samples. R. G. Deshpande has done study on cryogenic treated carbide cutting tool and found that cryogenic treatment of tungsten carbide tool inserts results in improved wear resistance, and also the observed wear pattern in treated samples show regular and well established trend . K. K. More had concluded that the steels the hardness is independent of treatment time and he also find out the optimise austinising temperature and cryogenic temperatures and cryogenic temperatures within the liquid nitrogen were identified. Ilyas Uygur has done study on AISI D3 Steel and he determined the corrosion resistance on cryogenated sample and he had found that there was decrease in corrosion resistance after treatment. P. I. Patil and R. G. Tated have studied various research papers and found out that Different approaches have been applied for CT to study the effect on different types of steels and other materials and they also aim at at the comprehensive analysis of strategies followed in CTs and their effects on properties of different types of steels by application of appropriate types of CTs from cryogenic conditioning of the process. The conclusion of their paper discusses the development and outlines the trends for the research in this field. They have also studied that what is the effect of various austinitizing temperature and their effects on hardness value on component, they have also study the microstructure formed after cryogenic heat treatment,also effect of soking time , soking temperature , wear resistance , cooling rate.

III. EFFECT OF CRYOGENIC TREATMENT ON DIFFERENT TYPES OF TOOL STEEL

3.1. EFFECT OF CRYOGENIC TREATMENT ON MICROSTRUCTURE AND WEAR CHARACTERISTICS OF AISI M35 HSS

3.1.1. COMPOSITION

Specimens were prepared from AISI M35 HSS bar of 15 mm square cross section with a nominal composition of C - 0.889%, Mn - 0.273%, Si - 0.364%, S - 0.006%, P - 0.024%, Cr - 4.175%, Ni - 0.171%, Mo - 4.656%, V - 1.788%,W - 6.087%, CO - 4.551%. Suitable allowances have been adopted to account for surface preparations as necessary in case of laboratory tests.[3]

3.1.2. PROCEDURE

Initially all the specimens were subjected to conventional heat treatment in a barium chloride salt bath furnace in the following sequence. As a first step specimens were pre-heated in a forced air circulation furnace maintained at a temperature of 500 ˚C to remove the moisture content for a period of 30 minutes. Later they were transferred to salt bath pre heating furnace maintained at a temperature of 900 ˚C for a period of 7 minutes. Subsequently the lot was transferred to hardening furnace maintained at 1200˚C for austenitization to occur for a period of 2 minutes. The specimens were swiftly transferred and quenched in salt bath furnace maintained at 560˚C for a period of 15 minutes for stabilisation to occur and finally the lot was air cooled up to room temperature. After that the speciman is followed by shallow cryogenic treatment or deep cryogenic treatment.

Shallow cryogenic treatment has been carried out at -85 ˚C with a soaking time of 8 hours the specimens were very slowly cooled at the rate of -0.5 ˚C /min until they reach the fine soaking temperature of -85˚C CT& in Deep cryogenic treatment has at -195˚C with a soaking time of 24hr. Specimens were cooled at the rate of -0.5 ° C / min until they reach the final soaking temperature of -195 ˚C

Before proceeding for cryogenic treatment the batch of conventionally heat treated specimens was cleaned to re-move the dirt, impurities and traces of salt layer found on their surface.[3]

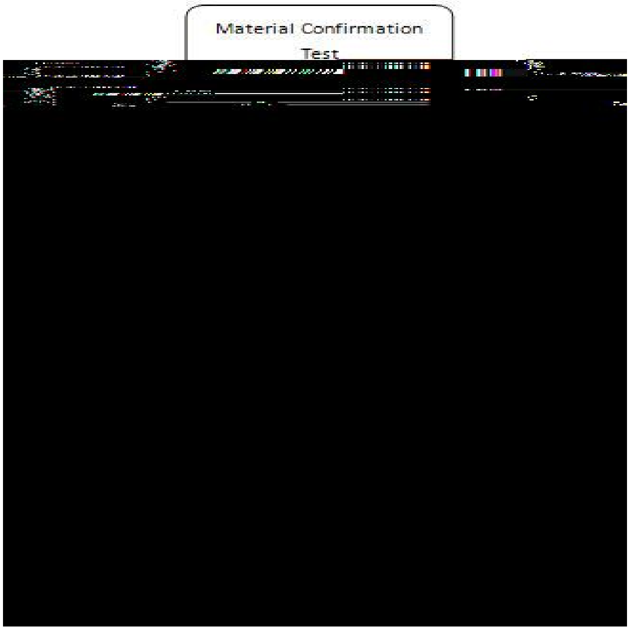

Fig 2-: Flow chart of procedure

3.1.3. HARDNESS TEST

Hardness of specimens subjected to conventional heat treatment (CHT), shallow cryogenic treatment (SCT) and deep cryogenic treatment (DCT) have been presented in the following table. A minimum of four tests were conducted as per IS 1586 – 2000(RA2006) and ASTM E384-10 guide-lines for measurement of bulk hardness and micro hardness respectively. The average value of hardness is presented in Table.1.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.

Table-1: Results of Hardness Test.

SR No.	CHT	SCT	DCT
HRC	64	64.5	65.5
Vickers Hardness	920	934	980

3.1.4. SEM Analysis

In the case of conventional heat treated specimen the presence of primary M6C carbides in the matrix of martensite is very clearly observed Also the presence of micro voids is observed.

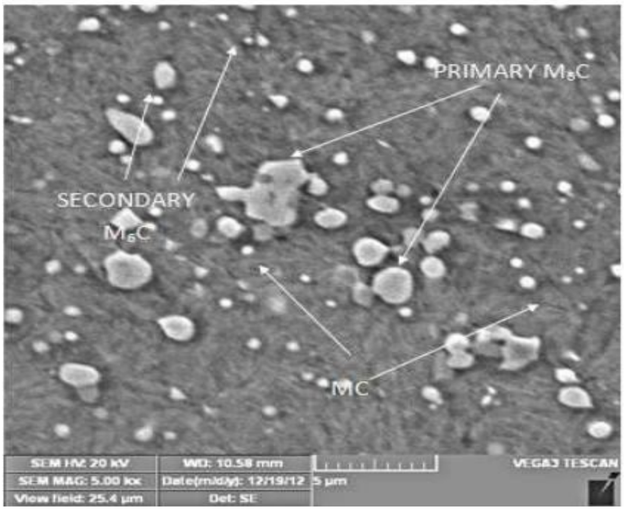

Fig-3: SEM image of Conventionally Heat Treated, AISI M35HSS, Specimen.[3]

The shallow cryogenic treated specimen reveals a homogeneous distribution of primary M6C carbides and precipitation of secondary M6C carbides. Still some micro voids are observed.

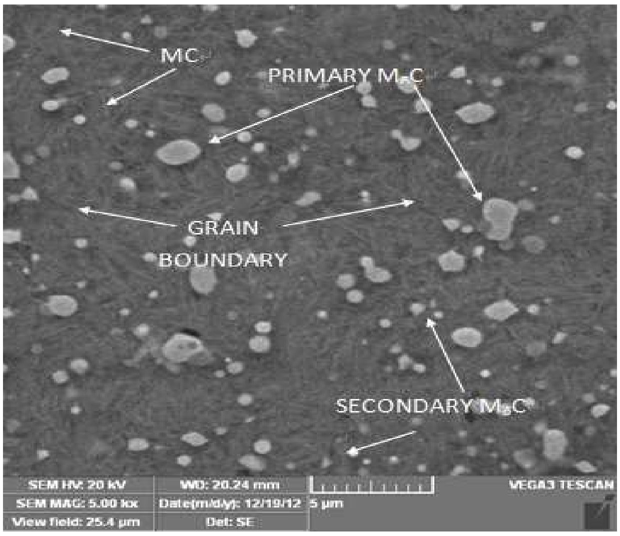

Fig-4: SEM Image Of Conventionally Heat Treated and Shallow Cryogenic Treated AISI M35 HSS Specimen. [3]

The deep cryogenic treated specimen reveals precipitation of more number of secondary M6C carbides and their size refinement. Secondary carbides of size ranging 0.3-0.5 were observed in the deep cryogenic treated specimen. Precipitates of fine carbides are mostly along the grain boundary of the deep cryogenic treated specimen.

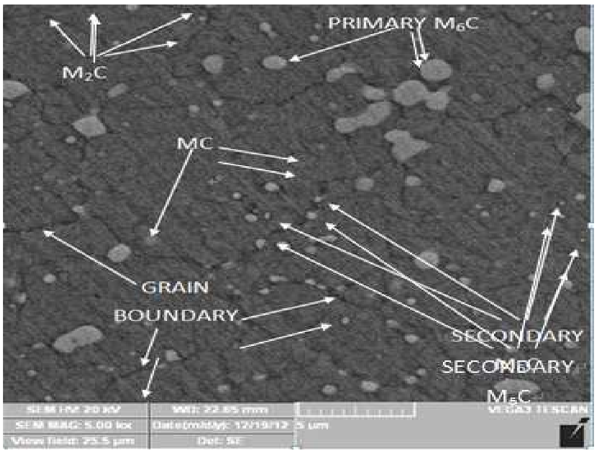

Fig-5: SEM image of Conventionally Heat Treated and Deep Cryogenically Treated AISI M35 HSS Specimen. [3]

3.2. EFFECT OF PROCESS PARAMETER OF CRYOGENIC TREATMEMT OF TOOL STEEL

3.2.1. TEST PROCEDURE

Three tools were investigated in this series of tests, as Steels 1 and 2 will be referred to by their AISI designations, H13 and D2 respectively. Steel 3 will be referred to by its trade name Vanadis 4. The investigation was designed to assess the effectiveness of the following factors on hardness of cryogenically treated steels, with the aim of establishing the optimum heat / cold treatment, with the emphasis on maximizing hardness.[4]

Table-2: Tools used.

Trade Name	Ovar Supreme	Sverker 21	Vanadis 4
AISI Designation	H13	D2	None - falls between A7 and D7
General description	Chromium-Molv Vanadium hot work tool steel	High-carbon, high-chromium, moly, vanadium cold work tool steel	Chromium-Molv-Vanadium powder metallurgical cold work tool steel
Typical Analysis	0.38% C, 1.0% Si, 0.4% Mn 5.3% Cr, 1.3% Mo, 0.9% V	1.55% C, 0.3% Si, 0.3% Mn 12.0% Cr, 0.8% Mo	1.5% C, 1.0% Si, 0.4% Mn, 8.0% Cr, 1.5% Mo, 4.0% V

3.2.2. Cryogenic Temperature (Test 1)

Steels austenitised at 1170°C. Samples quenched directly to one of three cryogenic temperatures and held for between 38 minutes and 20 hours.

Analysis

The results were as expected. Hardnesses increased as if retained austenite was continuing to transform to martensite. Although -100°C would appear to be the optimum temperature to achieve maximum hardness it was not possible to say whether complete transformation to martensite had occurred. The increase in hardness of each steel is a reflection of the original quantity of retained austenite in that steel. The greatest increase of over 16 HRc corresponds to a retained austenite content in the D2 steel of well over 50% .[4]

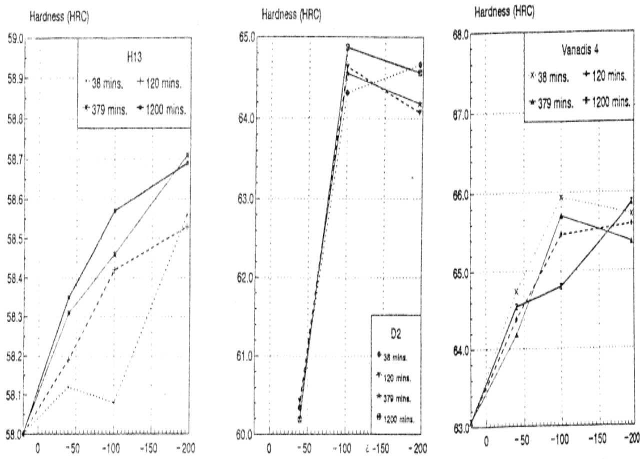

Fig-6: Results of Cryogenic Treated AISI M35HSS (Test 1)[4]

3.2.3. TEMPERING TREATED AND UNTREATED STEELS (TEST 2)

Steels austenitised at 1075°C before treating half at -196°C for 15 minutes. Treated samples were subjected to a one, two hour temper. Untreated samples were tempered twice for two hours.

ANALYSIS

As expected, the graphs of the untreated specimens exhibit secondary hardening characteristic of high alloy tool steels. The treated steels are obviously harder than their untreated counterparts because of the differences in martensite content, but the differences start to decrease after 300°C as the transformation of retained austenite in the untreated steels starts to accelerate, until at about 500°C the untreated steels become harder than the treated ones.

These results have two important implications for the heat/cryo treatment of steels and their application. It is obvious that, if maximum hardness is a priority, then a low temperature temper of a cryogenically treated steel is in order. However, it should also be possible to improve the toughness of some high-alloy tool steels whilst maintaining optimum hardness. These types of steels sometimes exhibit temper embrittlement when tempered in the secondary hardening temperature range, so that hardness is increased at the expense of toughness. It should be possible to achieve this level of hardness by tempering a cryogenically treated steel at a lower temperature, so avoiding temper embrittlement.[4]

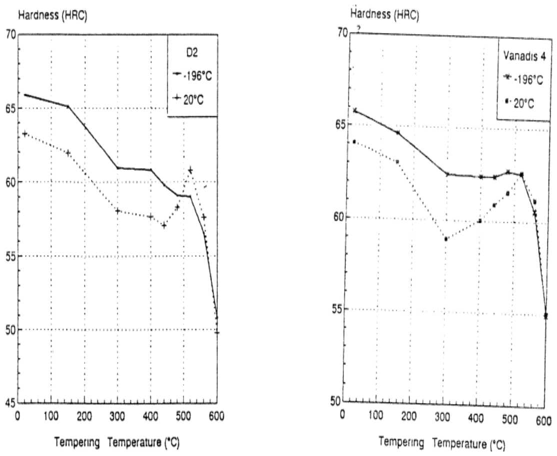

Fig-7: Result of Test 2 [4]

3.3.2. MACHINING & TOOL WEAR MEASUREMENT

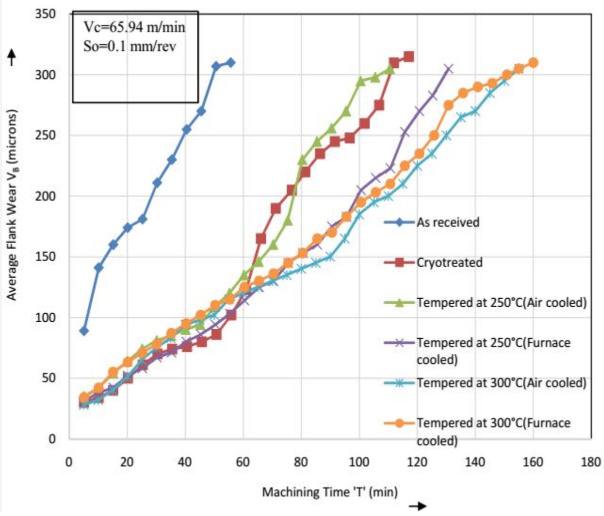

Fig-8: Machining & Tool wear. [1]

It is clearly observed from the experiment that in all the machining trials the growth of flank wear more or less showed the established pattern. Under all cutting velocities, Cryotreated and tempered inserts provided the highest tool life followed by Cryotreated tool inserts. Initially flank wear of both types of inserts is same but with consequent machining, cryogenically treated inserts showed less flank wear compared to untreated inserts. Also the tool life is reduced at high cutting speed. There was a gradual improvement in tool life observed in samples after treatment. The maximum tool life was shown by Cryotreated and tempered and 300 ^{0}C followed by furnace cooling. Higher wear rate of untreated inserts during the machining can be attributed to coarse carbide structure.

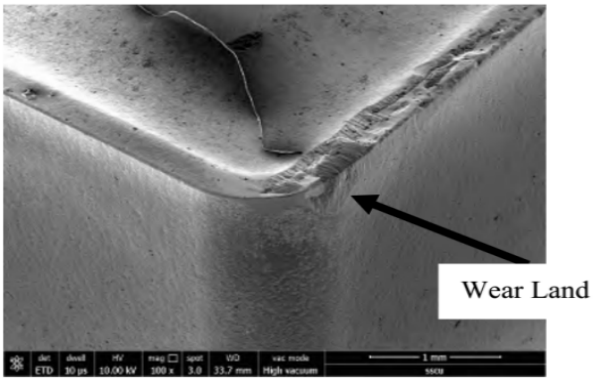

Fig-9: As received tool

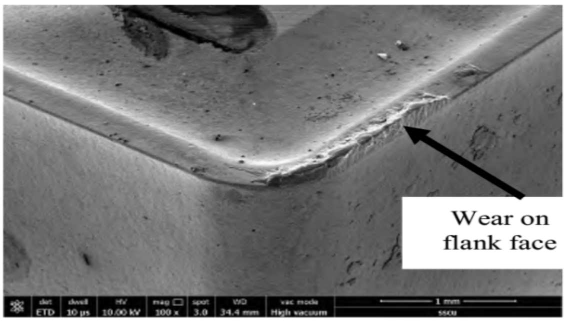

Fig-10: Cryogenically treated tool steel

3.4. EFFECT OF CRYOGRNIC TREATMENT ON CORROSION OF AISI D3 STEEL

Table-3: Compostion of D3 Tool Steel [2]

Element	Amount (wt %)
Carbon	2.0-2.3
Manganese	0.6
Silicon	0.6
Nickel	0.30
Chromium	11.0-13.5
Sulphur	0.03
Phosphorus	0.03
Vanadium	1.0
Ferrous	Remainder
Total	100

3.4.1. Method

Electrochemical measurements were carried out in a three-electrode type cell with separate compartments for the reference electrode (Ag/AgCl), the counter (Pt) and the working (AISI D3 steel) electrodes having an area of 0.5 cm^2. The surfaces of the working electrode were prepared by grinding with 400-1800grade abrasive paper, rinsing with distilled water and then degreasing with acetone. During the measurements, the solution was stirred with a magnetic stirrer . The composition of AISI D3 steel is given in Table 3.

Four types of uncoated AISI D3 steel samples were used: conventionally heat treated, 24 h cryogenically treated, 36 h cryogenically treated and 36 h cryogenically treated and then 2 h tempered at 150 °C. The cryogenic treatment for the AISI D3 samples was achieved by gradually lowering them from room temperature to -145 °C at the cooling rate of about 5 °C/min and holding at this cryogenic temperature for 24 h or 36 h, then gradually bringing them back to room temperature at the heating rate of 5 °C/min. The tempering of the AISI D3 steel was performed in a muffle furnace with the capacity of 9kW, 380 V and 1200 °C. After the AISI D3 steel samples were prepared , their corrosion behaviour in a 3.5% NaCl medium was then examined.[2]

Table-4: Investigated AISI D3 steel sample codes and qualifications.

Sample (AISI D3) Code	Qualifications
Q	Without pre-treatment
QH	Heat treatment
QH24cry	Heat treatment + 24 h cryogenic treatment
QH36cry	Heat treatment + 36 h cryogenic treatment
QH36cry2temp	Heat treatment + 36 h cryogenic treatment +2 h tempered

3.4.2. Morphological measurements

Table 5 shows the general EDS analysis results of the samples and their concentrations (wt%). Based on the corresponding EDS results, it can be concluded that all AISI D3 steel samples were corroded in the 3.5% NaCl solution. These findings validate the SEM and EIS results. While the concentration of C was mostly constant, O, Cr and Mn concentrations increased depending on the type of pre-treatment. The reduction in the amount of Fe and the increase of Cr has also been reported in previous studies.

Table-5: General EDS results of concentration (wt%) of samples (a-e)

Element	General EDS results of samples (a-e) conc. (wt%)				
	Q	QH	QH24cry	QH36cry	QH36cry2temp
c	3.19	2.67	3.12	3.49	3.75
O	25.28	31.67	35.47	41.06	45.52
Cr	7.47	10.01	10.45	10.92	11.7
Mn	0.3	0.36	0.34	0.35	0.42
Fe	63.76	55.29	50.62	44.18	38.61

EDS results were obtained from averages of the entire surface areas of the presented SEM images. The SEM micrographs are shown in Figure 11a-e. Table 5 gives the corresponding EDS spectra of the surface of the AISI D3 steel specimens after conducting electrochemical tests (EIS) on the different pre-treated samples in 3.5% NaCl solution. Figure 11a-e reveals that the pre-treatment of AISI D3 steel had a significant effect on the corrosion process. It is clear from Figure 11 that the surface of (a) was damaged less than the others (b-e). The surface of sample (e), with heat treatment + 36 h cryogenic treatment + 2 h tempering, was damaged by pitted areas in the typical form of pitting corrosion. The surface roughness of samples (d) and (c) seemed to be greater than that in the other SEM images. This study also suggests that initial surface roughness does not have a predominant effect on corrosion resistance. Rather, corrosion resistance is associated with both compressive and residual stresses.

Figure-11: SEM images for different pre-treatment AISI D3 steel samples in 3.5% NaCl solution: (a) Q, (b) QH, (c) QH24cry, (d) QH36cry,(e) QH36cry2temp[2]

IV. CONCLUSIONS

1. There is a marginal improvement in hardness from 64HRC to 64.5HRC for shallow cryogenic treated specimens. And it improved further after DCT to 65.5HRC.
2. Also the micro hardness measured in Vickers scale shows an increase in hardness value from 920 to 934 in case of shallow cryogenic treatment and it was 980 in case of DCT.
3. In case of H13 Tool steel its hardness value goes on increasing upto -196^{0}C, while we get optimum hardness value for D2 and Vanadis4 at -100^{0}C cryogenic temperature.
4. Tempering cryogenically treated D2 or Vanadis 4 steels at their secondary hardening temperature should be avoided. Actual hardness will not be better than conventionally treated steel. By tempering the treated steels at lower temperatures the user should benefit from the higher hardness due to the cryogenic treatment.
5. Cryogenic Treatment of Tungsten Carbide tool inserts result in improved wear resistance.
6. All investigated samples were corroded in 3.5% NaCl. Furthermore, the corrosion resistance of these samples can be ranked as follows: Q > QH > QH24cry > QH36cry > QH36cry2temp.

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