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Cryogenic Treatments Applied to AISI D2 Steel: Effects on Wear, Hardness and Microstructure

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----------*ABSTRACT*----------
Superficial (SCT) and deep (DCT) cryogenic treatments were applied to AISI D2 tool steel, to observe its response regarding wear, hardness and microstructure to these treatments, and its advantage over the conventional quenching-tempering treatment (CHT). For the SCT; after austenitizing, samples were cooled to (-80°C), 6 h. For the DCT, cooling was done at (-196°C), 4h. Cryogenic treatments considerably improve hardness and wear resistance. With the DCT treatment, the highest wear resistance is achieved, due to the refinement and distribution of secondary carbides (SC). The primary carbides (PC) are mainly responsible for the resistance to abrasive wear and the secondary ones (CS), with their fine morphology and distribution, are mainly responsible for adhesive wear. The microstructures of the cryogenic treatments show a martensite matrix with thickened (CP) and secondary (CS) carbides with fine dispersion of the types : $M_{23}C_6$, M_7C_3, Cr_7C_3.
KEYWORDS; *Cryogenics, hardness, types of wear, tool steels, carbide precipitation*

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I. INTRODUCTION

D-series cold work tool steels are high carbon, high chromium steels. They present high resistance to wear and low distortion in heat treatments **[1,2];** Therefore, they are generally used in punches for cutting and stamping, dies for wire drawing, among many other applications **[3].** After applying conventional quenching and tempering (CHT) treatments to these steels, the expected microstructure is tempered martensite with a highamount chromium-rich carbides, called primary carbides **[4].** However, these steels can be very affected by the presence of an undesirable component, retain austenite**[5].**

It is common knowledge that the transformation of austenite into martensite is non-diffusional (it does not depend on time), and depending on the degree of cooling below Ms, a quantity of austenite, which has not been transformed, may remain in the structure after from cooling (retained austenite). This retained austenite is a metastable phase that can transform into secondary martensite during working, and therefore the transformation can cause distortion and residual stresses in the component, leading to the formation of cracks and failures, mainly under dynamic loads **[6].**In this context, treatments at low temperatures called cryogenic treatments are alternative methods to increase the useful life and decrease the residual stresses of tool steels **[7]**. These cryogenic treatments can only be carried out in sub-zero refrigerant media. Sub-zero cryogenic treatment (SCT) uses dry ice (-80°C) as coolant and deep cryogenic treatment (DCT) uses liquid nitrogen (-196°C). Cryogenic treatments are used in this temperature range. .

AISI D2 steel is a ledeburitic steel, that is; It is an alloy steel, within small amounts of ledeburite are present after solidification. The formation of ledeburite, even at low carbon concentrations of ~1.5%, results from the influence of alloying elements in the carbon concentration range where the eutectic reaction takes place **[8].**The ledeburitic carbides play an important role when these steels are subjected to abrasive or adhesive wear.The cryogenic treatment applied to cutting tools, have shown very good results in the machinability and cutting performance **[9–11].** It has also been observed that shallow and deep criogenic treatment of tungsten carbide (WC) inserts, improve surface roughness by 28.3% and 72.3%, respectively, in contrast to untreated conditions during turning. These studies reveal cryogenic treatments improved the wear resistance of inserts in machining **[12].**

Podgornik et al**. [13]**researhed the effect of cryogenic treatments on the fracture toughness, wear resistance and load capacity of tool steels. They found that the effect of cryogenic treatment depended on the type and chemical composition of the tool steel.Priyadashini et al. [14]. report that cryogenic treatment significantly affects the mechanical properties of tool steels, and improves their useful life for industrial applications. These improvements in the mechanical and tribological properties when the SCT and DCT cryogenic treatments are applied, to the microstructural changes after their application such as: redistribution

and more homogeneous precipitation of carbides, as well as the increase of fine submicroscopic carbides (secondary and tertiary), moreover the refinement of the martensite laths **[4].**

The most accepted theories of the influence of DCT on the increase in carbide precipitation establish: (1) the formation of carbides is a consequence of the phase change from retained austenite to martensite **[15,16]**; (2) local plastic deformation during heat treatment causes segregation of carbon atoms through dislocations in local deformed areas during tempering, which acts as nuclei for future carbides **[17]**; (3) the precipitation of carbides is a product of the contraction and expansion of the martensitic network **[18]** and finally (4) the increase in precipitated carbides is a function of the carbides and their location in the matrix **[19].**

The factors that most influence the effectiveness of DCT are the immersion temperature and time, the heating/cooling rate of the DCT, and the location of the DCT in the heat treatment scheme. Moreover, the austenitizing and tempering temperatures also influence the effectiveness of the DCT application in these tool steels **[20-21].**

The purpose of this study is to determine how superficial SCT and deep DCT cryogenic treatments influence hardness, wear resistance and microstructure, when applied to AISI D2 tool steels.

II. EXPERIMENTAL METHODOLOGY

2.1 Study material

In the experiments, AISI D2 steel was taken as one of the most representative tool steels of the "D" series, with a high carbon content of 1.5%C, chromium 12% Cr, molybdenum 1% Mo and vanadium0,9% V; moreover, alloyedin small amounts, of tungsten silicon and manganese. It is the most widely used in stamping operations andconformed; it is characterized by:

- High resistance to wear.
- High resistance to compression.
- High hardenability.
- Good dimensional stability during heat treatment.
- Good resistance to tempering.
- Of low tenacity in comparisonwith other steels, which makes it sensitive tochipping or fracture. the chemical composition is shown in table 1.

Table 1. Chemical Composition of AISI D2 Steel

STEEL	C	Si	Mn	W	Cr	Mo	V	Fe
AISI D2	1.5	0.3	0.3	0.02	12.0	1.0	0.90	Balance

2.2.Test and Standard samples.

To measure hardness, 50 mm x 25 mm x 3.5 mm prismatic bars were used; and they were machined in accordance with the ISO 6508 standard, which defines the dimensions of these specimens and the conditions for an accredited test. The readings were made on the HRC scale. To measure the resistance to abrasive wear, test specimens in the form of 76 mm x 25.4 mm x 8 mm prismatic bars were used. The test was performed in accordance with ASTM G65. The measurement of the resistance to adhesive wear was carried out using the "pin on disc" methodology, in which the material and dimensions were classified according to the materials used for the wear test. Specimens in the form of 76mmx25mmx8mm prismatic plates were used. These samples and tests were carried out in accordance with the ASTM G83 – 96, standard.The heat treatments were carried out in an electric muffle furnace. Two austenitizing temperatures 1020°C and 1050°Cwere used. Quenching was done in oil for conventional CHT heat treatment. For the subzero SZC cryogenic treatment, the samples were cooled in a CO_2 bath at (-80°C), for 6 h. For the DCT deep cryogenic treatment, the samples were cooled in a liquid nitrogen bath at (-196°C), for 4h. The tempering temperature was 200°C, for all the samples where it was necessary to apply this treatment.

2.3.Heat treatment program.

It can be seen in the diagram of figure 1. In all treatments the specimens were heated to austenitizing temperature (1020°C - 1050°C) and an electric digital muffle furnace was used. The sequence was the following:

1) Stress annealing at 500°C for 30 minutes with air cooling for 1h.
2) Then the samples were preheated at 500°C for 20 minutes.
3) The specimens were ready and were heated at the austenitizing temperatures: 1020°C - 1050°C, for 30 minutes.

4) Cooling was performed in an isothermal salt bath at 150°C for 30 minutes, with subsequent cooling to room temperature for the CHT treatments. For cryogenic treatments, the previous procedure was the same; but, the cooling for the samples with SCT treatment was in a CO2 refrigerator (-80°C) and for the samples with DCT treatment it was in a liquid nitrogen bath at a temperature of (-196°C). then, samples were subjected to a subsequent tempering at 200°C.

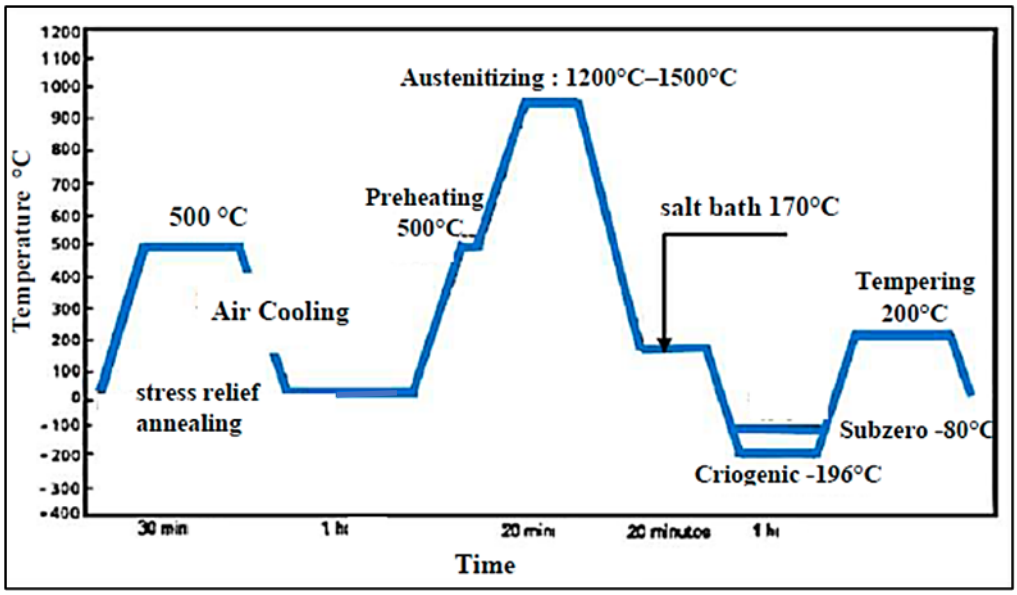

Figure 1.Program of heat treatments followed in the experiment.

Sample nomenclature:
M1: Conventional heat treatment (CHT)
M2: Surface Cryogenic Treatment (SCT)
M3: Deep Cryogenic Treatment (DCT)
M4: Treatment (SCT) without tempering
M5: Treatment (DCT) without tempering

III. RESULTS

3.1 Hardness

Table 2 shows a maximum average value of 67.7 HRC, corresponding to sample M5 subjected to DCT cryogenic treatment, austenitized at 1050°C, and a minimum value of 58.8 HRC corresponding to sample M1 subjected to conventional treatment, austenitized at 1020°C, providing an increase of 8.9 HRC, (13% with respect to the CHT). Regarding samples M2 and M4 with SCT cryogenic treatments, values of 64.3 and 67.5HRC are observed, with a difference of Δ= 3.2 HRC. (4.7% increase compared to CHT) For samples M3 and M5 with DCT cryogenic treatments, there are values of 64.9 and 67.7 with a difference Δ= 2.8 HRC, which represents an increase of 4.7% compared to CHT

Tabl2 2. Hardness results (HRC) of all heat treatment samples.

Sample	T_A = 1020°C				T_A = 1050°C			
	Hardness (HRC)							
	1	2	3	Average	1	2	3	Average
M1	58.9	59.9	58.8	**59.2**	61.9	62.8	62.6	**62.4**
M2	64	64.1	64.2	**64.1**	63.3	64.4	65.1	**64.3**
M3	64.1	65.2	64.2	**64.5**	64.4	65.3	64.9	**64.9**
M4	65.1	65	65.2	**65.1**	67.2	67.7	67.6	**67.5**
M5	65.4	65.7	65.3	**65.5**	67.1	68.2	67.8	**67.7**

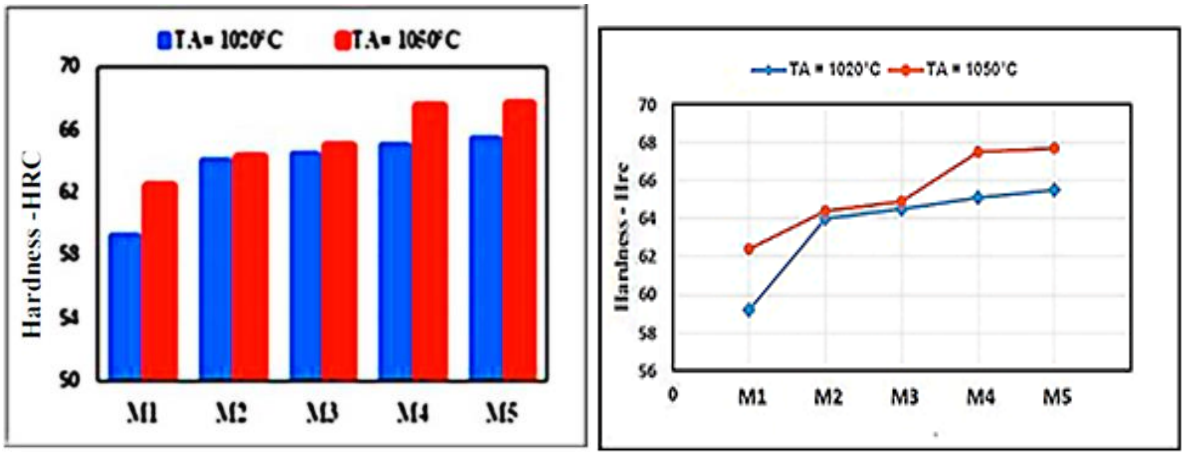

Figure 2. Graph that shows the variation of hardness in the samples subjected to different treatments a) Bar graph, b) Cartesian graph. In both, it is observed that the austenitizing temperature (TA) increases the hardness.

3.2 Mass Lost and resistance due to Adhesive Wear

Table 3 shows a maximum value of mass loss of 3.40 mg corresponding to the M1 samples subjected to conventional CHT heat treatment with an austenitizing temperature of 1020°C. The minimum of 1.40 mg corresponds to sample M3 with DCT treatment with an austenitizing temperature of 1050°C (See table 4).
The graph of Fig. 3 shows us the notable difference that exists in the loss of mass due to adhesive wear, between the cryogenic treatment in its two forms with respect to the conventional CHT treatment. The maximum difference is Δ= 2 mg representing ~ 60% efficiency. There is not much difference between the two types of cryogenic treatments.

The resistance to wear is a parameter whose relationship is inverse to the loss of mass. These values and trends are observed in table 4 and figure 4. This parameter tells us that the maximum resistance to wear is found in the M3 samples and the minimum in the M1 samples, the opposite of the previous case. The effect of subsequent tempering on adhesive wear resistance is almost negligible;

Table 3. Mass loss due to Adhesive wear .**Table4.**Adhesive wear resistance

	T_A = 1020 ° C	T_A= 1050 ° C
Sample	**Massloss(mg)**	
M1	3.40	3.20
M2	1.85	1.65
M3	1.80	1.40

	1020 °C	1050 °C
Sample	**Adhesive Wear Resistance (mg/m)$^{-1}$**	
M1	393.53	418.13
M2	723.27	810.96
M3	743.38	955.75

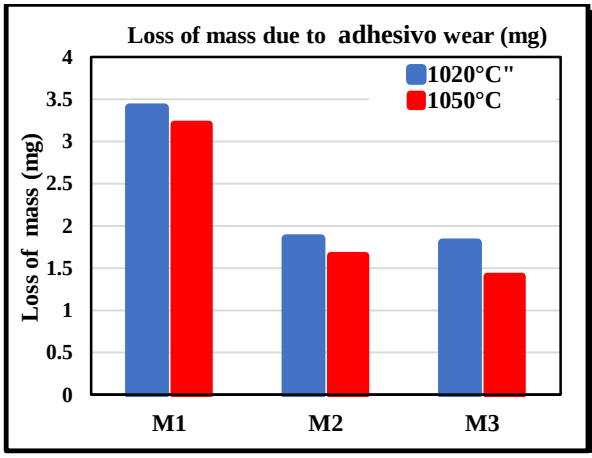

Figure 3. Graph showing mass loss of samples due to adhesive wear.

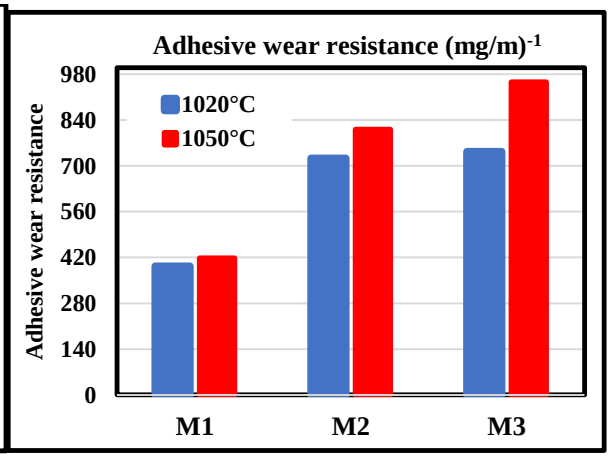

Figure 4.Graph showing the resistance of the samples due to adhesive wear.

3.3Mass lossand resistance to abrasive wear

The results are found in tables 5 and 6 and their trend graphs in figures 5 and 6.

Table 5. .Mass lost due to abrasive wear **Table 6.** Abrasive wear resistance

Loss of mass due to abrasive wear (mg)		
	1020°C	**1050°C**
M1	46.95	46.40
M2	42.40	37.50
M3	33.00	31.40

Abrasive wearresistance(mg/m)$^{-1}$		
	1020 °C	**1050 °C**
M1	91.78	92.87
M2	101.63	114.91
M3	130.58	137.23

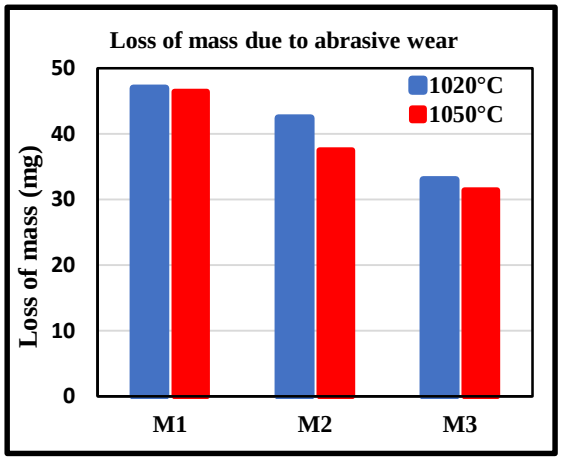

Figure5.Graph showing the mass loss due to abrasive wear of samples subjected to conventional and cryogenic treatment.

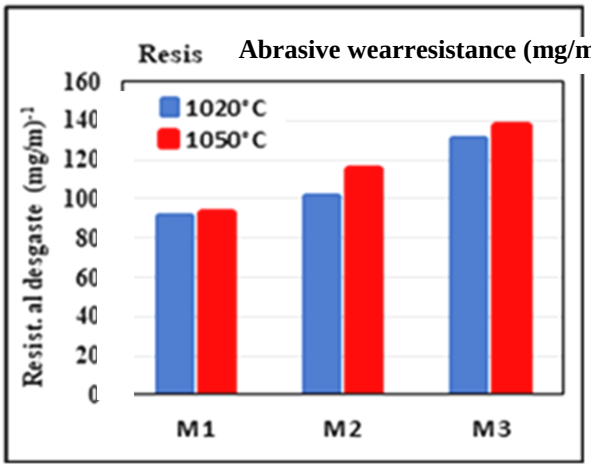

Figure 6.Graph showing the resistance to abrasive wear of samples subjected to conventional and cryogenic treatment.

3.4. Microstructure in as-delivered state.

The supply sample was delivered in the annealed state (Figure 7). A mixture of various types of carbides is observed on a ferritic type matrix. Primary carbides (PC) that did not dissolve during austenitizing are observed. Primary equiaxed carbides indicated with a yellow arrow have a size of 11.4 µm and 5.5 µm. An elongated (PC) of 16.4 µm in length is also observed. In addition, there is a fine dispersion of secondary carbides (SC) precipitated during annealing; as the one indicated with a size of 2.5 µm in length. All observed carbides are micrometer in size.

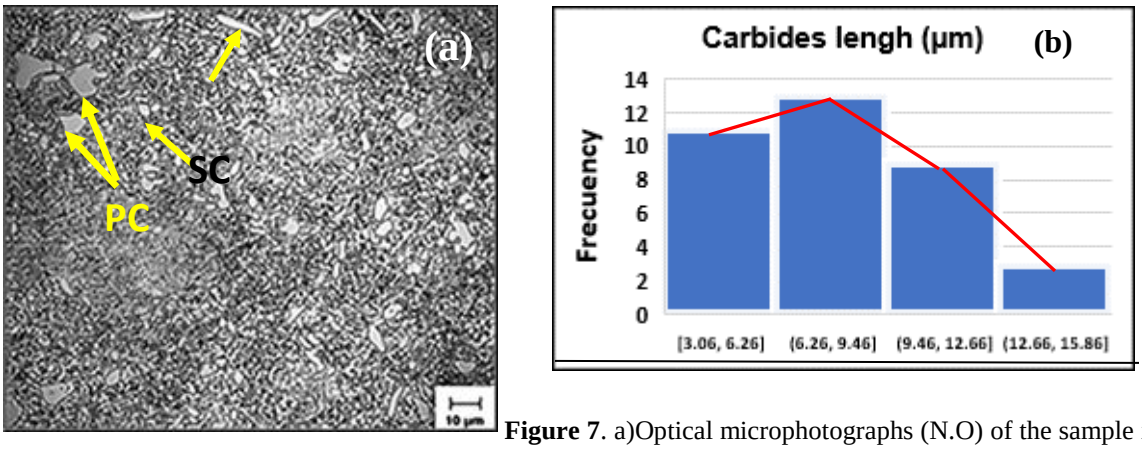

Figure 7. a)Optical microphotographs (N.O) of the sample in the state of supply; b) Carbide distribution histogram

3.6. Optical microstructures with treatments: CHT SCT and DCT Austenitizingat 1020°C.

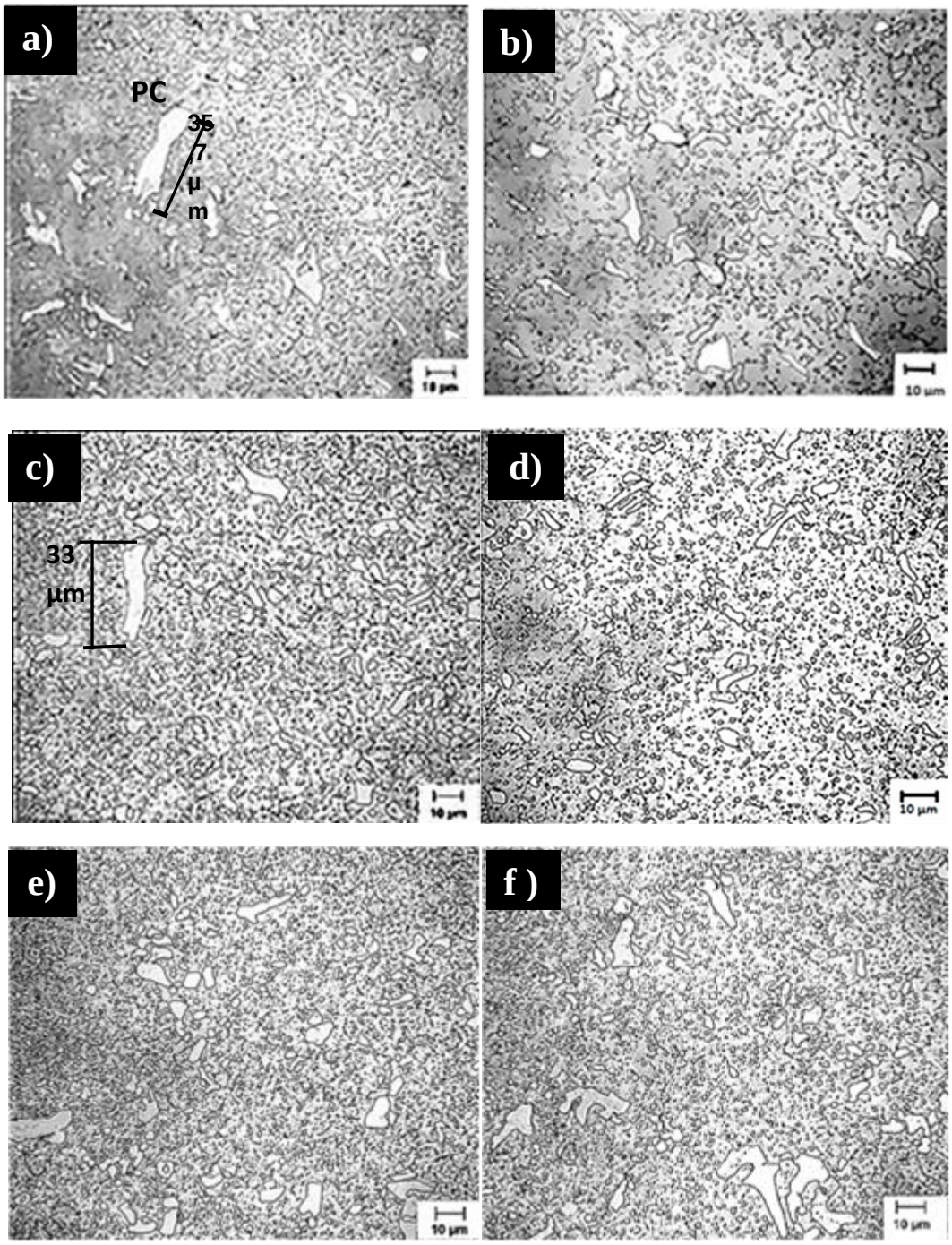

Figure 8. Photomicrographs (N.O) showing the microstructures of the samples subjected to conventional and cryogenic treatments, at austenitizing temperatures of 1020°C: a) CHT cooled in air and tempered b) CHT cooled in oil) c) SCT without tempering; d) SCT tempering treatment; e) DCT without tempering; f) DCT with tempering.

3.7.SEM, cryogenic SCT and DCT microstructures Austenitizing at 1050°C.

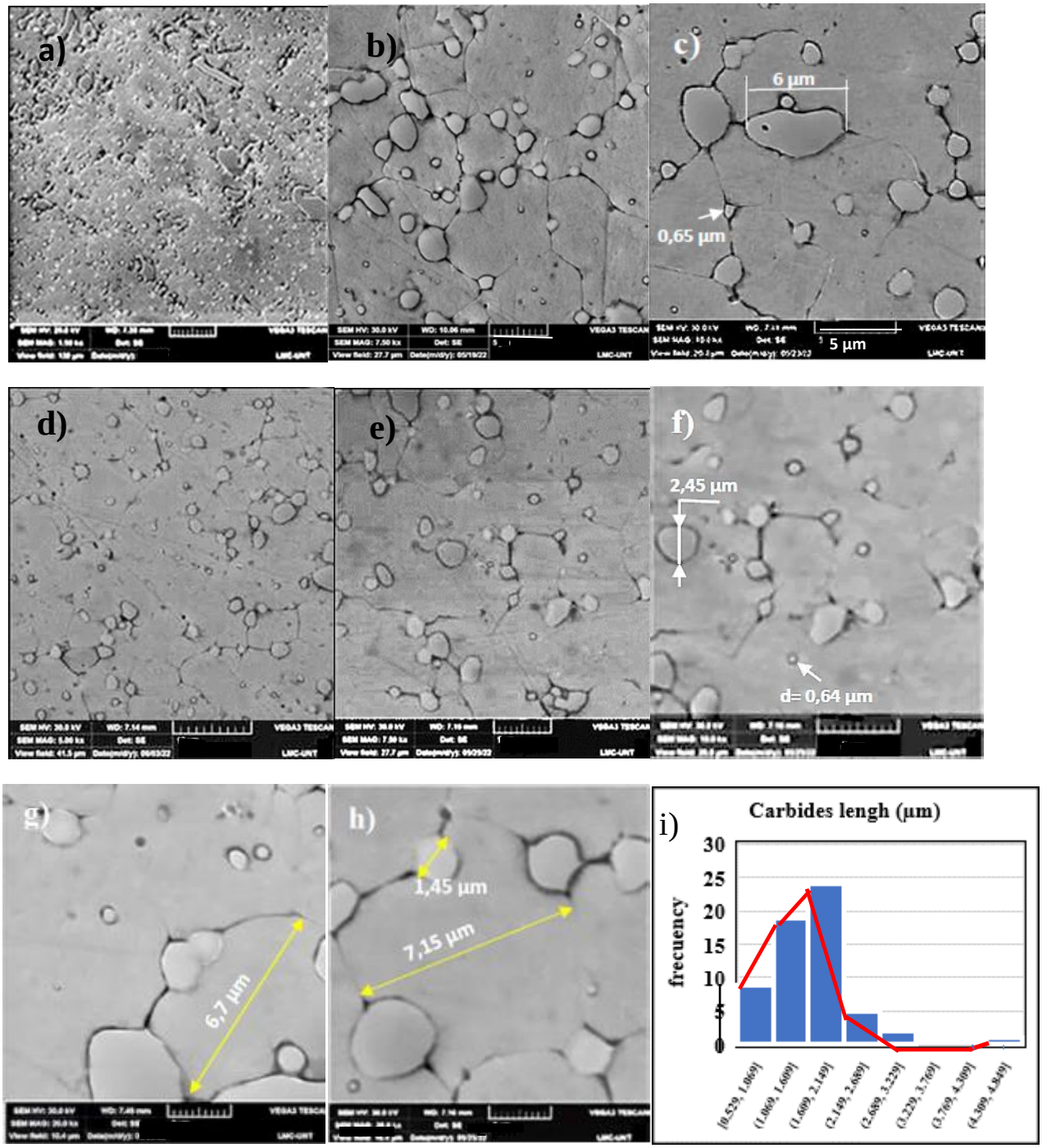

Figure9. Microstructures of cryogenic SCT and DCT samples: a),b),c) samples treated with SCT seen at different scales; d), e), f) samples treated with DCT seen on different scales; g) and h) samples treated with SCT and DCT respectively with the same scales and with a magnificence of 20.Kx, showing grain and carbide dimensions.i)Histogram of the size of carbides present in the sample d) subjected to DCT

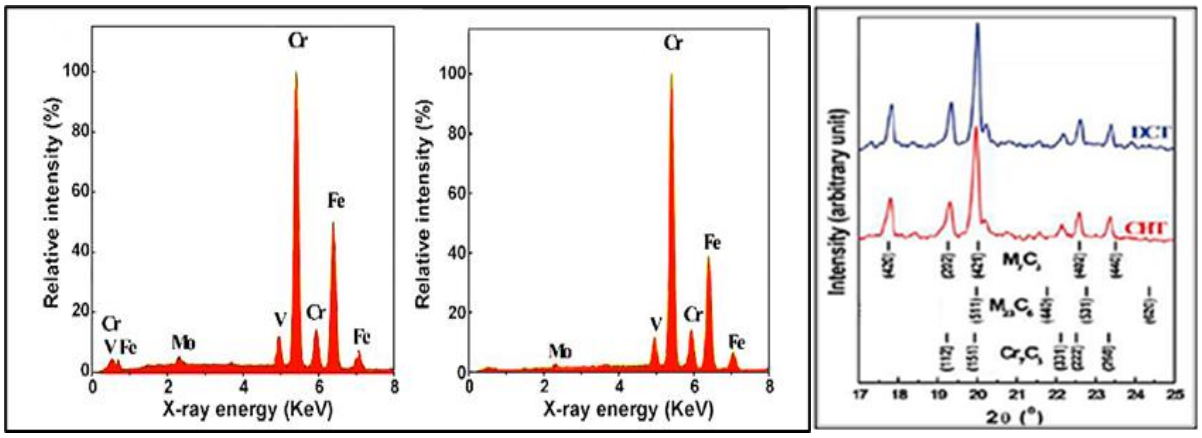

Figure 10. a) EDX analysis of primary carbides in DCT treatment: b) EDX analysis of secondary carbides in DCT; c) XRD diffraction analysis applied to the carbides extracted from the samples with CHT treatment and cryogenic DCT samples. The analyzes indicate the following types of carbides: M_7C_3; $M_{23}C_6$; Cr_7C_3.

IV.DISCUSION

For the two austenitizing temperatures; The maximum Hardness (67 HRC) was obtained with the DCT cryogenic treatment without tempering and the Minimum (59 HRC) with the conventional CHT treatment; the difference is 8 HRC. It is observed that a lower cryogenic temperature increases the hardness. On the other hand, a higher austenitizing temperature produces an increase in hardness. The increase in hardness due to a lower cooling temperature means that there has been a greater transformation of retained austenite into Martensite, getting closer to the end point "Mf" of the transformation.

In cryogenic treatments as the residence time increases, the hardness does not increase suddenly since the martensite transforms almost spontaneously (non-diffusional transformation). However, the change of microconstituents and the formation of carbides requires a transformation time (diffusional transformation). It has been observed that the phase transformations in cryogenic treatments can reach a limit of ~36h **[9]**, where diffusional processes should occur.

The results indicate that cryogenic samples without subsequent tempering have higher wear resistance than non-tempered samples, demonstrating that subsequent tempering can decrease hardness depending on the tempering temperature, but has little effect on wear. In cryogenic treatments, it is the abrasive wear that presents the greatest loss of mass, but when compared with the CHT treatment, the loss of mass is significant. On the other hand, both in cryogenic and conventional CHT treatments, when the austenitizing temperature is raised there is a tendency to reduce the loss of mass due to wear.

Cryogenic treatments show very little mass loss in adhesive wear. Likewise, it is observed that the loss of mass due to abrasive wear is much greater with respect to adhesive wear; this is because the mechanisms of adhesive and abrasive wear are very different. The mechanism of abrasive wear is very complex and is not by itself a property of the material; since this type of wear is directly related to the mechanism on which it depends. In general, abrasive wear obeys the three-body wear scheme and is related to the environment in which the material that has formed the tool in service operates **[10, 11].** On the other hand, hardness itself is not an indicator of the increase or decrease in resistance to adhesive or abrasive wear**. [12, 14].** An exact relationship between hardness and wear in steels or metallic materials has not yet been found. In some cases, there is a direct correlation and in others an inverse correlation.

The loss of mass due to wear is related to the microstructure obtained. However, the exact relationship Microstructure → type of wear is still under study. For example, cases of steels with different microstructures that have the same resistance to abrasive wear have been found **[15].**

In the results you can see the benefits of applying Cryogenic Treatments to increase resistance to wear; especially the abrasive, being used as tool steels for both hot and cold work **[16, 20].**

To state that the improvement in wear resistance is explained by the transformation of retained austenite into martensite is still a simple explanation. However, this transformation is a key feature in increasing hardness and wear; both in conventional heat treatments and in cryogenics.For example, the improvement of the wear resistance of tool steels, although it depends on the minimization of retained austenite; It also depends on the refinement of carbides, especially secondary carbides (SC) **[21- 24].**

In some cases, it is observed that an increase in hardness is directly correlated with an increase in wear resistance. It has been shown that this correlation is valid only in the case of pure metals and not in alloy steels

[25]. The relationship hardness → wear rate has been studied, both abrasive and adhesive. It has been experimented with various tool steels **[26].**

The results indicate that the conventionally treated (CHT) samples present a microstructure with a tempered martensite matrix with a uniform dispersion of large primary carbides (PC), which are the ones that did not dissolve during austenitizing, and secondary carbides (SC) more small precipitates during the treatment as shown in Fig.8. The samples with cryogenic treatment exhibit a martensite matrix with a dispersion of primary carbides (PC) of various sizes, fine secondary carbides (SC). of micrometric sizes. The carbides (SC) are of various sizes, medium and small, of micrometric sizes, and are found in the range <0.65- 6> µm as observed in fig. 9c). These carbides precipitate as large secondary carbides (LSC) and small secondary carbides (SSC).

The primary carbides (CP) are responsible for the good resistance to abrasive wear and the secondary (SC) are more related to adhesive wear. Carbides add to the strengthening of tool steels.Large carbides ~20µm provide great resistance to abrasive wear, as long as they are evenly distributed **[27].**Primary carbides (PC) are mostly responsible for resistance to abrasive wear. Secondary carbides (SC), with their fine morphology and greater distribution, are more related to resistance to adhesive wear.

During cryogenic treatments (DCT) applied to tool steels, in addition to the formation of secondary carbides of different sizes, it is possible, in addition to increasing their content, to promote a more homogeneous distribution; which is essential to increase wear resistance **[27].** This considerable improvement in wear resistance depends on minimizing retained austenite, as long as it is accompanied by refinement and distribution of carbides, especially SC secondary carbides.

The microstructures of the CHT treatments show primary carbide particles of 35.7µm and the secondary ones are found within a tempered martensite matrix and are on average ~2.5µm in size. The microstructure of the samples with SCT and DCT treatments show grains of ~7µm and primary carbides (CP) of large sizes of ~6µm and medium, together with secondary carbides (SC) whose sizes are within the range <0.65-6> µm. Nanometric carbides have not been observed **[28].**The carbides found, are of the type: M_7C3 ; $M_{23}C_6$; Cr_7C_3 .

The results indicate that the DCT treatment, compared to the conventional CHT, increases the hardness by 12%; adhesive wear in 58.8%, and abrasive wear in 30%, when applied to AISI D2 steels, under the established conditions.

V. CONCLUSION

For the two austenitizing temperatures, the samples subjected to cryogenic treatments indicate a greater hardness with respect to the conventional CHT treatment. The maximum Hardness ~67 HRC was obtained with the DCT cryogenic treatment without tempering and the minimum ~59 HRC was obtained with the CHT treatment, almost a 12% increase. The increase in hardness occurs as the cryogenic temperature decreases.

Applying the cryogenic treatment to the samples, the wear resistance is considerably improved, which depends on minimizing the retained austenite, as long as they go together with a refinement and distribution of carbides, especially SC secondary carbides. Primary carbides (PC) are mostly responsible for the resistance to abrasive wear. Secondary carbides (SC), with their fine morphology and greater distribution, are more related to resistance to adhesive wear.

In the treatments where subsequent tempering is applied, the results show little change in hardness and wear resistance. The tempering applied at 200°C had the purpose of eliminating residual stresses.

The microstructures of the CHT treatments show primary carbide particles ~ 35.7µm and the secondary ones are found within a tempered martensite matrix and are on average ~2.5µm in size. The microstructure of the samples with SCT and DCT treatments show grains of ~7µm and primary carbides (PC) of large sizes of ~6µm and medium, together with secondary carbides (SC) whose sizes are within the range <0.65-6> µm. Nanometer carbides have not been observed. The carbides found are of the type: M_7C_3 ; $M_{23}C_6$; Cr_7C_3.

Finally, the results indicate that the DCT treatment, compared to the conventional CHT, produces an increase in hardness of 12%; an increase in adhesive wear of 58.8%, and an increase in abrasive wear of 30%.

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