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Deep Cryogenic Treatment applied to AISI 420 Stainless Steel, with subsequent Tempering or Aging: Evaluation of Hardness, Impact Toughness and Microstructure

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ABSTRACT: *The deep cryogenic treatment DCT (-196°C) was applied, with a subsequent tempering and separately a similar treatment with aging in order to observe the differences in hardness, impact toughness and microstructure of AISI 420 stainless steels. A group of test specimens were austenitized at: 1020-1030-1040-1050 (°C) / 1h; then quenching in oil. After that, they were subjected to cryogenic treatment DCT/4h; and then They were subsequently tempered at: 480- 500- 510- 520- 530- 540 (°C)/ 2h. A second group of samples, after the DCT treatment, were subjected to aging at 150°C, with times: 2.5 - 5- 10 - 20 - 50 – 100 (h). Hardness was measured on rockwell "C" scale; and the Impact Resistance in a Charpy machine, according to ASTM E 23 – 93^a standard; whose measures were taken in Joules (J). The microscopy was taken at optical level. It was found: In the aging range, the samples austenitized at 1050°C have greater hardness than those austenitized at 1040°C. The aged samples exceed the hardness of the tempered samples. In the Treatment: DCT + subsequent aging, the maximum hardness value (58.7 HRC) is combined with an impact toughness value (54J) obtained with an aging time of 30h, which is considered an optimal condition.*

KEYWORDS - *martensitic steel, aged, hardness, precipitation, DCT treatment.*

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I. INTRODUCTION

Cryogenic treatment is a heat treatment that is applied to metallic and in some cases non-metallic materials at temperatures below zero **[1].** During cryogenic treatments, samples are gradually cooled by a control unit, using gases such as nitrogen and helium cooled to temperatures varying between -80 and -196 °C; and then of a waiting period, the samples are gradually going back to the room temperature **[2, 3].** When this treatment is applied at temperatures between −80 and −140 °C it is called superficial cryogenic treatment, while the treatment applied between −140 and −196 °C is called deep cryogenic treatment (DCT) **[4–8].** The peculiarity of the treatment is the result of the conversion of austenite to martensite eliminating almost all the residual austenite during the process, not allowing another transformation that could be harmful to the proposed objectives; Therefore; A microstructure with greater toughness and hardness can be achieved, in addition to a homogeneous distribution of carbides, which will increase its resistance to wear, impact toughness, fracture toughness and others, depending on its application **[9, 10].** The main effect of cryogenic treatment when applied to steels, is the elimination of retained austenite. Another effect that is observed, only in the DCT cryogenic treatment, is the reduction the size of carbides, increasing their percentage and making a more homogenized distribution **[11-15].** In particular, when we focus on the application of stainless steels, the overall picture of DCT effects is not very clear; as when applied to tool steels or carburized steels, where the removal of retained austenite is a consequence of the precipitation of fine and dispersed carbides. Paolo Baldissera **[16]** carried out studies in this regard, finding that the effects of DCT treatment on the tensile mechanical properties of hardened and solubilized AISI 302 austenitic stainless steel did not produce significant changes in the maximum tensile strength UTS and yield strength YS, rather there were a decrease in the elastic modulus "E" in some treatments. When the focus is centered on the application of stainless steels, the overall picture of DCT effects is not very clear; as when applied to tool steels or carburized steels, where the elimination of retained austenite is a consequence of the precipitation of fine and dispersed carbides.

AISI 420 steel is a martensitic stainless steel, with carbon content ranging between (0.15% - 0.40%) (wt), whose corrosion resistance comes from the chromium content (12-14% wt). In these composition ranges lies its balance between mechanical and corrosive properties, which makes it suitable for power generation, turbine blades, compressors, oil extraction, chemical, petrochemical and surgical equipment. It has also been shown that DCT significantly increases its wear resistance, increasing its tribological performance **[17 - 20].** According to

G. Prieto et al, **[21]** By applying cryogenic treatment to the martensitic stainless steels of the 4xxx series, they obtained an increase in the deformation state of the martensite, which led to the precipitation of a greater amount of smaller secondary carbides with a more uniform distribution. Subsequent tempering almost did not alter the amount of retained austenite. These studies show that wear resistance improves with DCT treatment, due to the transformation of residual austenite into martensite and the homogeneous precipitation of carbides. It was also found that when these DCT treatments were applied to AISI 420 steel the wear resistance increased by 35–90% compared to conventionally CHT treated samples.

One of the great applications of DCT treatment is the manufacturing of bearings. The results of many researches show that this treatment can improve the wear resistance of stainless steels using appropriate parameters including: quenching temperature, quenching speed, temperature and soaking time. **[22, 23].** Mechanical properties, and hardness during aging, is an important reference parameter on the stability and reliability of heat-resistant bearings; because the bearings are subjected to high temperatures for a long time in service; Therefore, the performance and stability at high temperatures would be directly related to the service life of the heat-resistant steel **[24].** On this sense, aging would be an alternative and/or complementary and effective treatment in these low and medium carbon stainless steels.

The purpose of this research is to apply the cryogenic DCT + subsequent aging treatment to AISI 420 stainless steel, comparing its benefits with the traditional DCT + Tempering, after evaluating its hardness, mechanical properties, impact toughness and microstructural evolution and to search the optimal parameters for its application on the manufacture of bearings.

II. MATERIAL AND METHODS

The study material was AISI 420 steel. It is a martensitic steel that can be hardened by quenching-and tempering treatment and natural and/or artificial aging. Its chemical composition is shown in table 1.

2.1 Heat treatments.

The study has been carried out under the Experimental Research model. The heat treatments were carried out in a “thermolyne” digital electric oven with a controlled atmosphere. The following parameters were used: Austenitizing Temperatures: 1020-1030-1040-1050 (°C) / 1h; Tempering temperatures: 480- 500- 510- 520- 530- 540 (°C) /2h; Cryogenic temperature: -196°C/ 4h, with aging temperatures: 510°C and aging time: 2.5 -5- 10 - 20 - 50 – 100 (h). The thermal treatment scheme can be seen in fig. 1. Cryogenic treatments were performed at -196°C (DCT deep cryogenic treatment) using liquid nitrogen, inside a muffle furnace shell at room temperature. All cryogenic treatments were carried out after oil quenching.

2.2. Mechanical Tests.

Hardness tests were carried out using a 601RSB digital Rockwell durometer. To measure the hardness of the samples, 10mmx20mmx30mm prismatic bars were used. These samples were machined, ground and polished according to ISO 6508 standards, to guarantee the parallelism of the faces. Three indectations were carried out per sample, taking the arithmetic average to construct the respective graphs.

For the Charpy impact tests, specimens were prepared according to the ASTM E 23 – 93. The sketches for its preparation are shown in fig. 2. All the microstructures of the samples were revealed at the optical level, using the Zeiss 1000X Microscope. To reveal the microstructure, 3% Nital reagent was used for 60 s, with prior encapsulation, grinding and very fine polishing.

Table 1 - Chemical composition of martensitic stainless steel AISI 420 (w%)

%C	%Si	%Mn	%Cr	%Ni	%Mo	%Fe
0.28	0.45	0.30	10.69	0.11	0.02	Balance

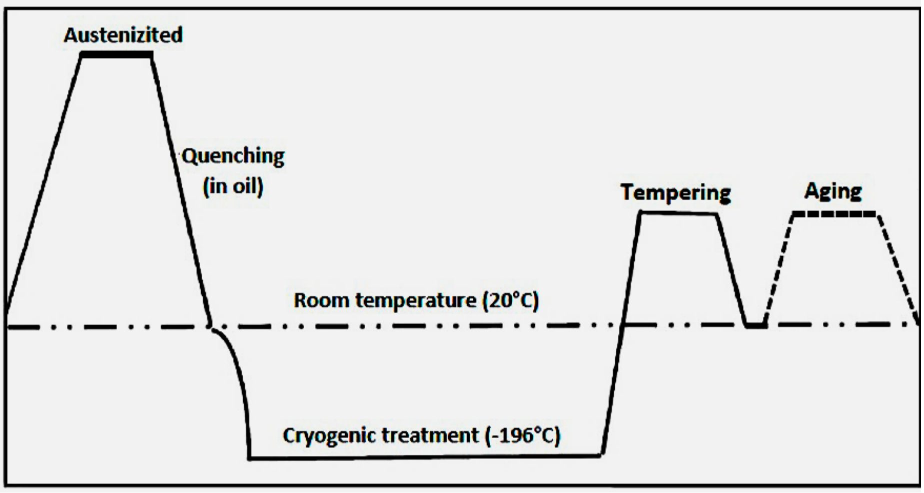
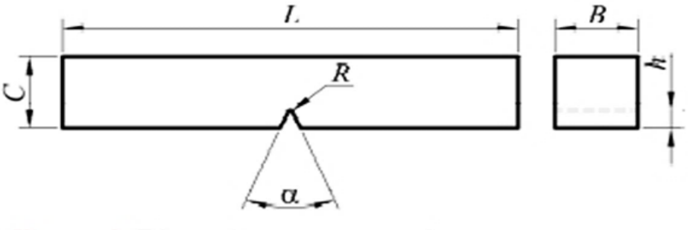


Figure 1. Scheme of the heat treatment cycle including cryogenic DCT (-196°C) with subsequent tempering or aging, performed after the austenitizing treatment.



Dimension	*Sample standard*
L [mm]	55
B [mm]	10
C [mm]	10
α [°]	45
h [mm]	2
R [mm]	0,25

Figure 2. Dimensions of the Charpy specimen according to ASTM E 23 – 93^a standard.

III. RESULTS AND DISCUSSION

3.1. I Influence of Austenitizing Temperature on Hardness on Conventional DCT Treatment

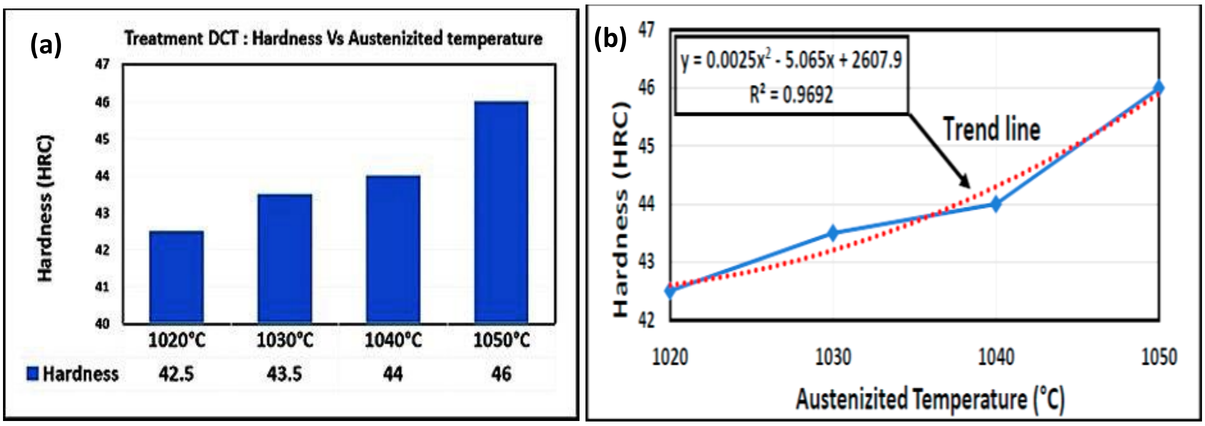


Figure 3. Hardness after applying conventional cryogenic treatment for different austenitizing temperatures: a) bar graph; b) cartesian graph with its trend line. These results led to selecting the austenitizing temperatures: 1040°C and 1050°C, for the tests.

The austenitizing temperature-hardness relationship is shown in Fig.3. The hardness increases as the austenitizing temperature increases. The hardness values are in the range [42.5 – 46.0 HRC] and within an

austenitizing temperature range [1020°C -1050 °C], which shows that an increase of 30°C in the hardness temperature Austenitizing produces a 35 HRC increase in hardness. This increase in hardness can be explained by the fact that at a higher austenitizing temperature the cooling rate is much greater, eliminating the untransformed residual austenite more efficiently. On the other hand, at high temperatures all the alloyed carbides that come with the delivery state (annealed) must have completely dissolved. Furthermore, DCT treatment increases the diffusion driving force of atoms (especially carbon atoms) that promote the formation of fine carbides in the tempering process; and carbides are more likely to grow or roughen during aging. Likewise, is suggested that the austenitizing temperature of the tested steel does not exceed 1050°C to avoid the formation of ferrite δ during the austenitizing process. **[25].** This compound is harmful and promotes corrosion and fracture toughness. This is the reason why it has been deemed convenient to select only the temperatures 1040°C and 1050°C for all the respective tests.

3.1.1. Hardness with Cryogenic Treatment and subsequent aging.

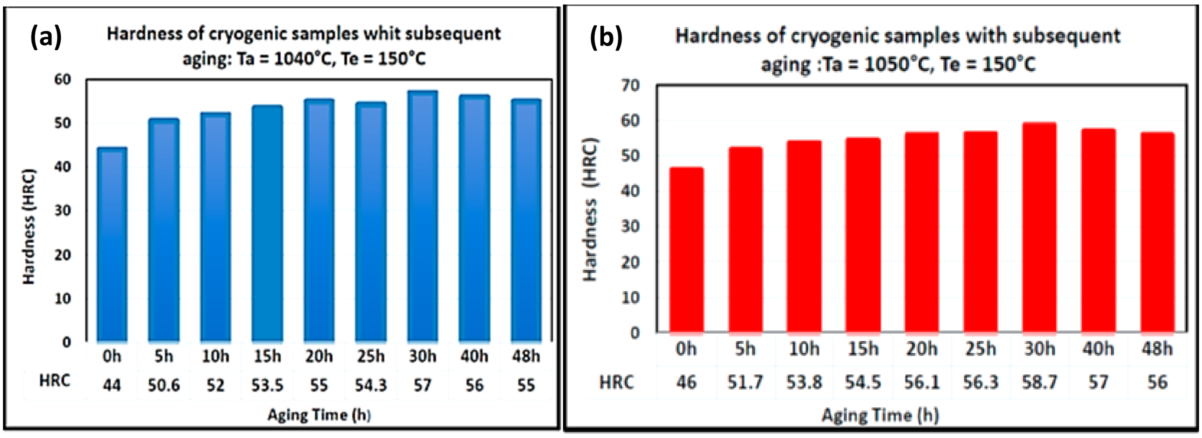


Figure 4. Graphs showing the variation in Hardness obtained in the samples after applying cryogenic treatment with subsequent aging. a) Ta= 1040°C; b) Ta = 1050°C.

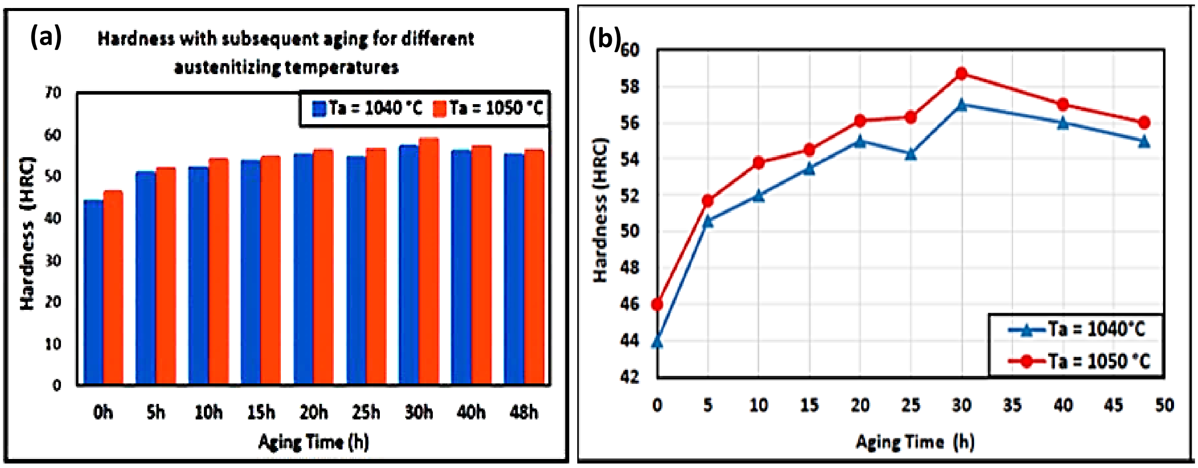


Figure 5. Comparative graphs of the hardness obtained after applying the DCT treatment with subsequent aging, for the austenitizing temperatures: 1040 -1050°C. a) Bar graph; b) Cartesian graph.

The hardness of the samples aged with austenitizing at 1040°C is observed in Fig. 4a). The initial hardness value with DCT is 44 HRC, and then the effect produced by subsequent aging is shown. In the aging time interval [5h – 48h] the hardness has increased; but not at a constant rate, but with certain oscillating intervals. The maximum value was 57 HRC for an aging time of 30h, corresponding to an increase of 13 HRC (30% more compared to the initial value). The minimum value was 50.60 HRC, corresponding to the lowest temperature of 5h, corresponding to an increase of 6.6 HRC (15% more compared to the initial value). The fact that the hardness increases to a maximum peak of (30h) and then becomes decreasing is explained by the same aging process; Thus, the microstructure evolves over time and it is possible that after 30 h the microstructure precipitates softer phases or depresses the carbides of the hard phases.

In the case of the samples austenitized at 1050°C, the results can be seen in figs. 4b). The initial starting value was 46 HRC. In the aging interval [5h – 48h] the hardness presents two zones; an increasing aging (5h - 30h) and a decreasing one (30h – 48h). The peak value was 58.7 HRC for aging of 30h, corresponding to a 28% increase compared to the initial value. The minimum value was 51.70 HRC, corresponding to the aging of 5h, showing an increase of 12.3% compared to the initial value. The results increase almost exponentially until 30 hours and then decrease until 48 hours; As seen in the comparisons shown in Figure Fig.5a and Fig.5b. In both graphs it is observed that for the entire aging time interval, the samples austenitized at 1050°C have greater hardness than those austenitized at 1040°C, showing coincidence in their maximum peaks at 30h of aging with a difference of 1.7 HRC.

3.1.2. Hardness with Cryogenic Treatment and subsequent Tempering.

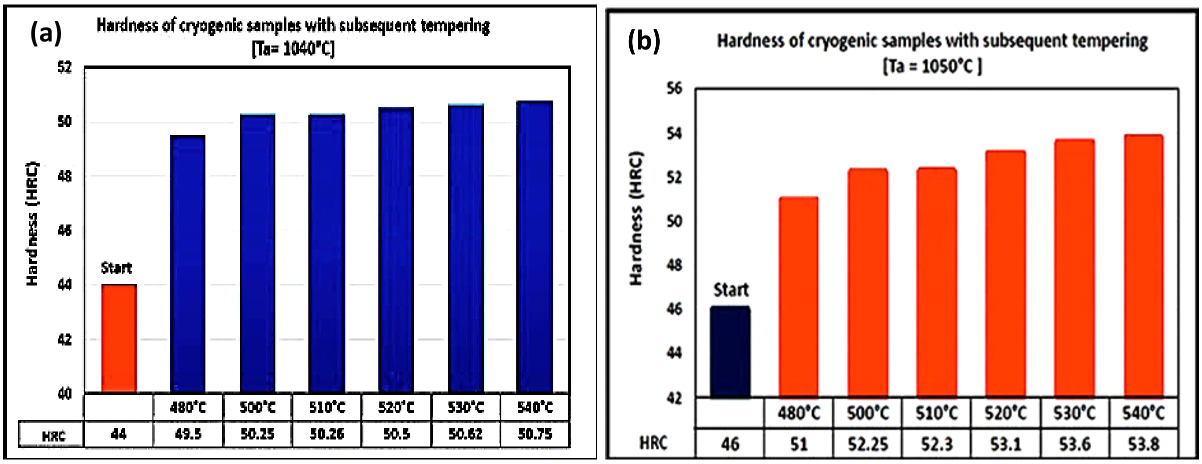


Figure 6. Graphs showing the variation on Hardness obtained in the samples after applying cryogenic treatment with subsequent tempering. a) Ta= 1040°C; b) Ta = 1050°C.

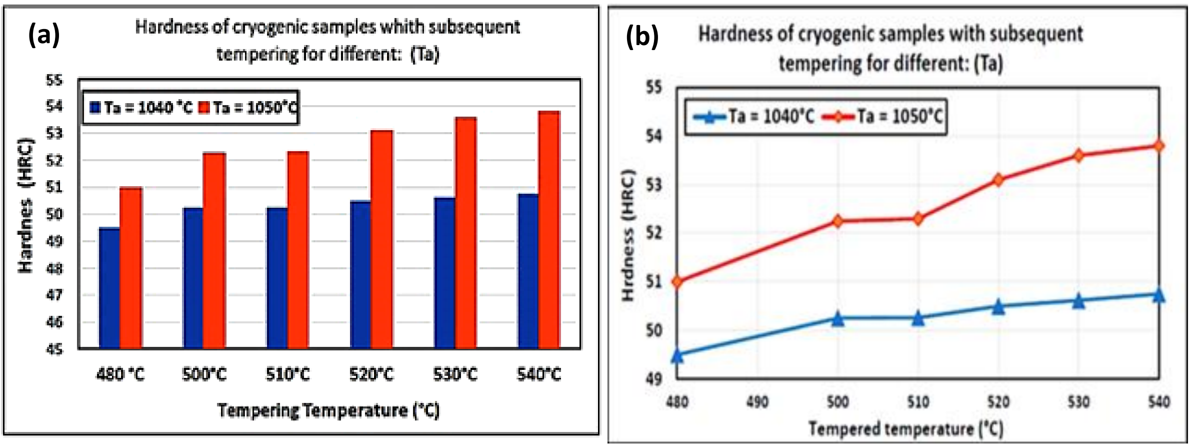


Figure 7. Comparative graphs of the hardness obtained after applying the DCT treatment with subsequent tempering, for the austenitizing temperatures: 1040°C -1050°C. a) Bar graph; b) Cartesian graph.

The results can be seen in Figure 6a and 6b. For samples austenitized at 1040°C, the initial hardness value is 44 HRC. It is observed that in the temperature range [480°C – 540°C] the hardness does not present much variation, with its maximum and minimum values being 50.75 HRC and 49.50 HRC; with a difference of Δ= 1.25 HRC, (2.8% more, compared to the initial value); demonstrating the benefit of subsequent aging in hardening.

For this austenitizing temperature, although the trend is increasing; The increase in hardness is slight, with the peak hardness at 540°C at the highest tempering temperature. The explanation of why tempering slightly increases hardness and does not decrease it as in the case of simple carbon steels. In this study it is a stainless steel that contains alloys such as Chromium (Cr) that are strongly carbide-forming elements, and these nucleate and precipitate as the tempering temperature increases.

For the samples austenitized at 1050°C, the results can be seen in fig. 6b. The initial value of hardness obtained by DCT treatment is 46 HRC. In the temperature range [480°C – 540°C] the hardness presents a slightly greater variation than for the previous case, with its maximum and minimum values being 53.80 HRC and 51.00 HRC; with a difference of Δ= 2.8 HRC, (6% more compared to the initial value). In Figure 7, we have comparative hardness diagrams for the two austenitizing temperatures, where it can be seen that the hardness trend is linear. In fig. 7b, it can be observed that the hardness of the samples austenitized at 1050°C suffers a considerable increase, when compared to the samples austenitized at 1040°C, where the change in hardness due to subsequent tempering is almost negligible.

3.1.2. Comparative analysis of hardness results.

The comparative analysis is clearly observed in the graphs of figures 8a) and 8b). The first graph shows the hardness of the two treatments sequentially for the samples previously austenitized at 1040°C. where it is observed that in all cases the aged samples exceed the hardness of the tempered samples, even in their minimum values. In the second graph the same trend is observed with slightly higher values. It can be inferred for this case study that the subsequent DCT + Aging treatment is intended to increase the hardness of the material; while the DCT + subsequent tempering acts as a treatment aimed at reducing residual stresses with a very slight increase in hardness compared to the initial value.

Graph 8a) shows that for an austenitizing of 1040°C, the DCT + subsequent aging treatment provides much higher hardness results than those found for DCT + tempering. Furthermore, during tempering the hardness hardly changes. It can be said that tempering acts as a residual stress relief treatment. The case of samples austenitized at 1050°C is very different, where it is observed that the subsequent tempering does have a significant variation.

The fact that in both cases, the aging treatment causes a much higher hardness is related to the microstructural evolution of the aging treatment; Therefore, it is a phase precipitation treatment, which are generally precipitates of alloyed carbides and intermetallic compounds, which evolve as time passes, depending on the aging temperature chosen. In our case; For an aging temperature of 510°C, for both austenitizing temperatures, the hardness peak is found for 30 h of immersion. From then on the samples decrease their hardness values.

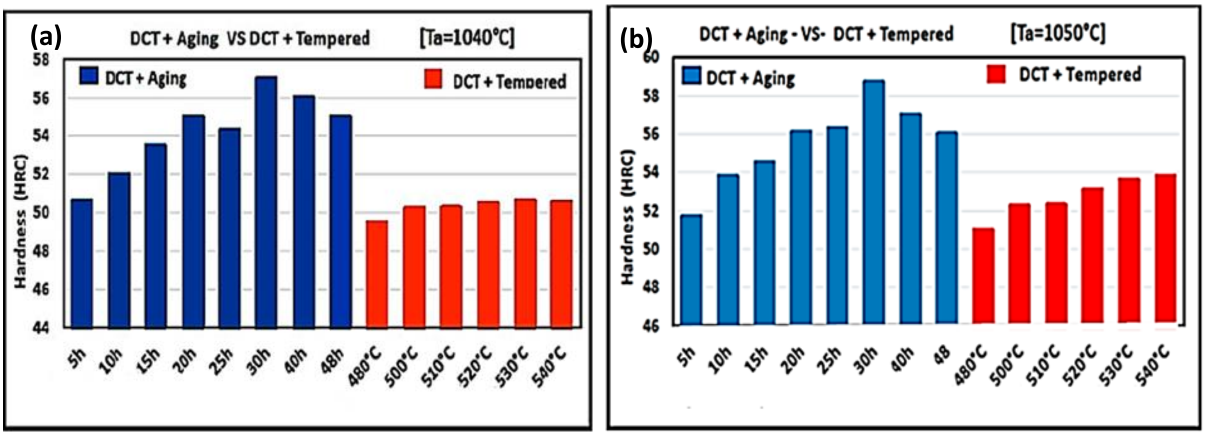


Figure 8. Comparative graphs of the hardness obtained in the samples after applying after applying both cryogenic treatments; a) Ta =1040°C; b) Ta= 1050°C

3.2. Impact Tenacity.

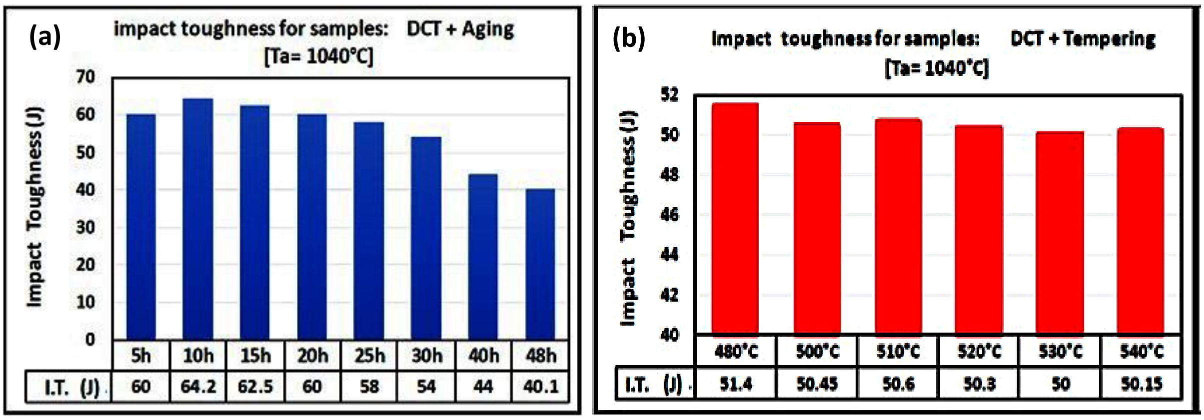


Figure 9. Graphs showing the impact toughness obtained in the samples after applying: a) DCT+ Aging; b) DCT + tempered; for an austenitizing temperature: Ta = 1040°C

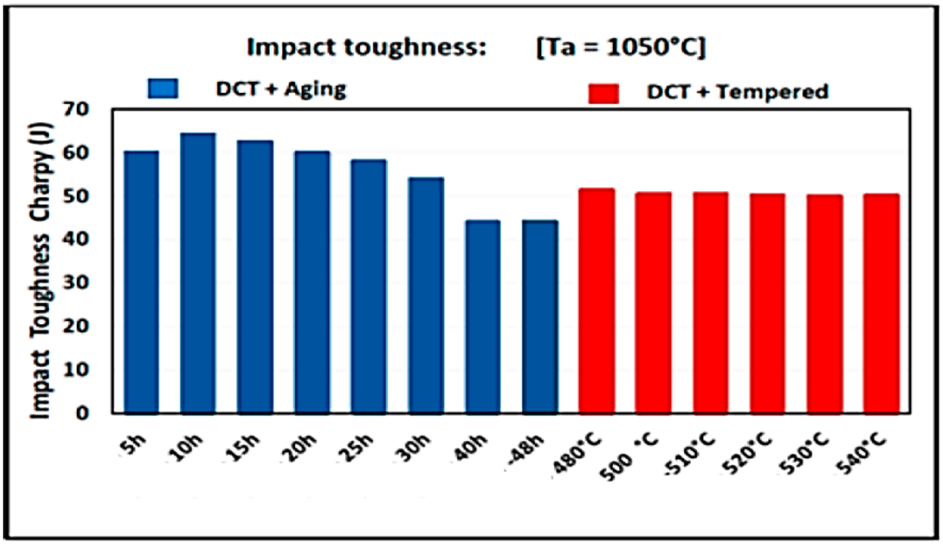


Figure 10. Comparative graph of impact toughness after applying the DCT + Aging and DCT + Tempered cryogenic treatment for an austenitizing temperature: Ta = 1050°C

3.2.1. Hardness-Tenacity: Relationship

Figure 11a) shows the Hardness-Toughness relationship for samples treated with Cryogenic Treatment: DCT + subsequent aging. The maximum hardness value (58.7 HRC) is combined with an impact toughness value (54J) obtained with aging time of 30h. On the other hand, the maximum value of toughness (64.2 J) is linked to a hardness value of (53.8 HRC), for 10 hours of aging.

Figure 11b) shows the Hardness-Toughness relationship for samples treated with Cryogenic Treatment: DCT + subsequent tempering. The maximum hardness value (53.8 HRC) is combined with an impact toughness value (50.15J) obtained with a tempering temperature of 540°C. On the other hand, the maximum toughness value (51.4 J) is linked to a hardness value of (51 HRC), for a tempering temperature of 480°C. The graphs show an inverse relationship between hardness and impact toughness in both types of treatment.

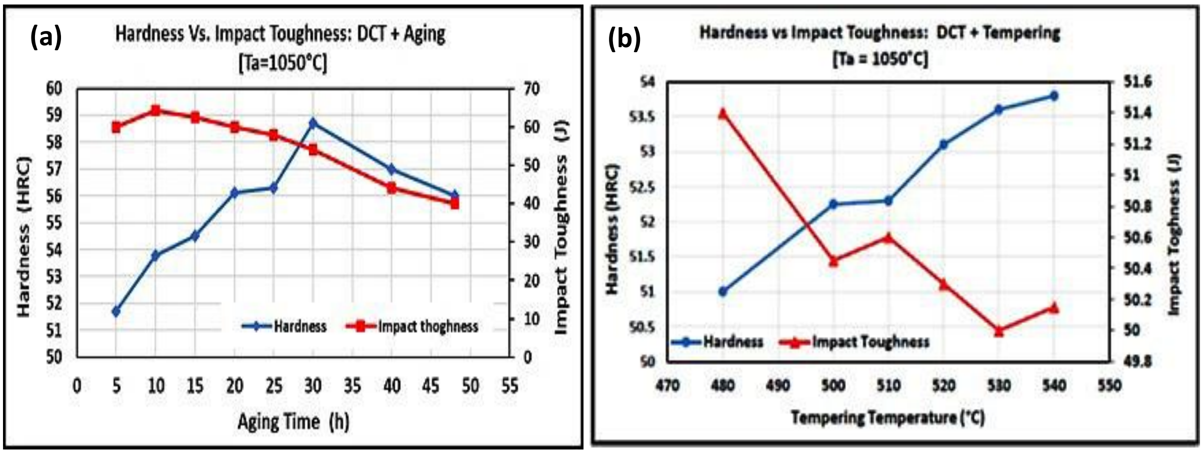


Figure 11. Graphs that compare Hardness with Impact Toughness: a) DCT + Aging; b) DCT + Tempering (Ta = 1050°C).

3.3. Microstructure

3.3.1. Delivery Status.

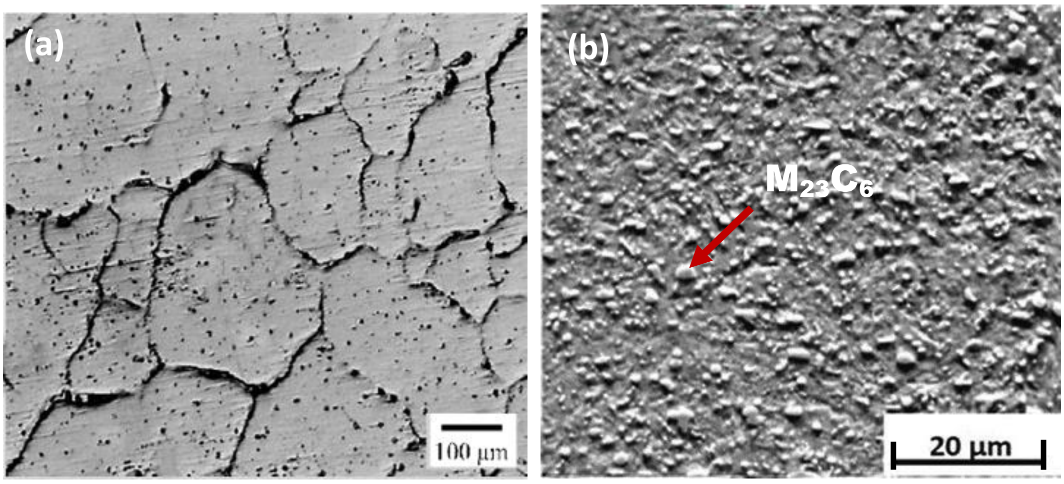


Figure 12. Microstructure of AISI 420 steel on delivery condition (annealed). It can be seen that it consists of a ferritic matrix with a homogeneous dispersion of spheroidized secondary carbides. The average hardness of the material is 34 HRC. (a) Optical level; (b) SEM electronic level, taken in an intergranular zone.

Figure 12 shows the structure of the supplied material. Microscopy reports that AISI 420 stainless steel has been previously laminated and has a two-phase structure made up of ferrite (α) and cementite carbides (F_3C). The constituents of the microstructure: the carbides appear in strong contrast with the matrix **[26].** An agglomeration of irregularly shaped carbides is observed in the continuous phase, while the nucleation of large cementite phases is heterogeneous with the main structure **[27].** On the other hand, a large precipitation of carbides is shown at the grain boundaries.

According to the specialized literature, heat treatments decompose large, hard and brittle cementite particles into small spheroidal particles isolated from each other **[28].** We also observe that the carbides precipitate in a ferritic matrix. According to J.S. Dubey, microstructure of martensitic stainless steel AISI 420 in the annealed condition includes ferrite and carbides of the $M_{23}C_6$ type **[29].** Depending on the austenitizing temperature, the carbides amount on the matrix is variable.

3.3.2. Samples quenching at different "Ta" without DCT.

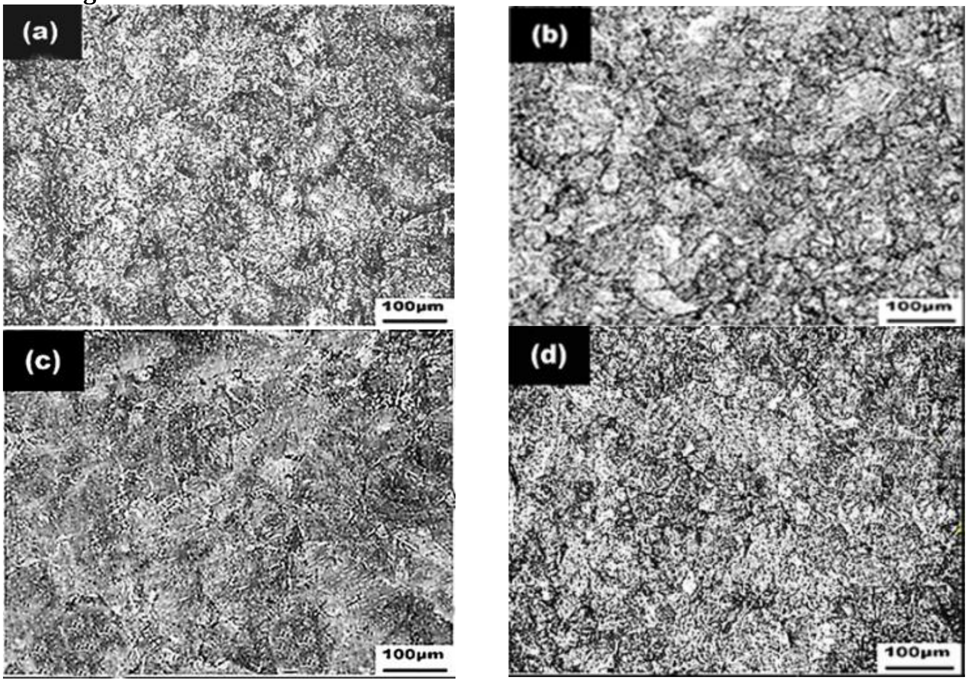


Figure 13. Microstructures of samples after quenching at different austenitizing temperatures: (a) 1020°C; (b) 1030 °C; (c)1040°C; (d) 1050°C.

3.3.3. Samples with DCT cryogenic treatment.

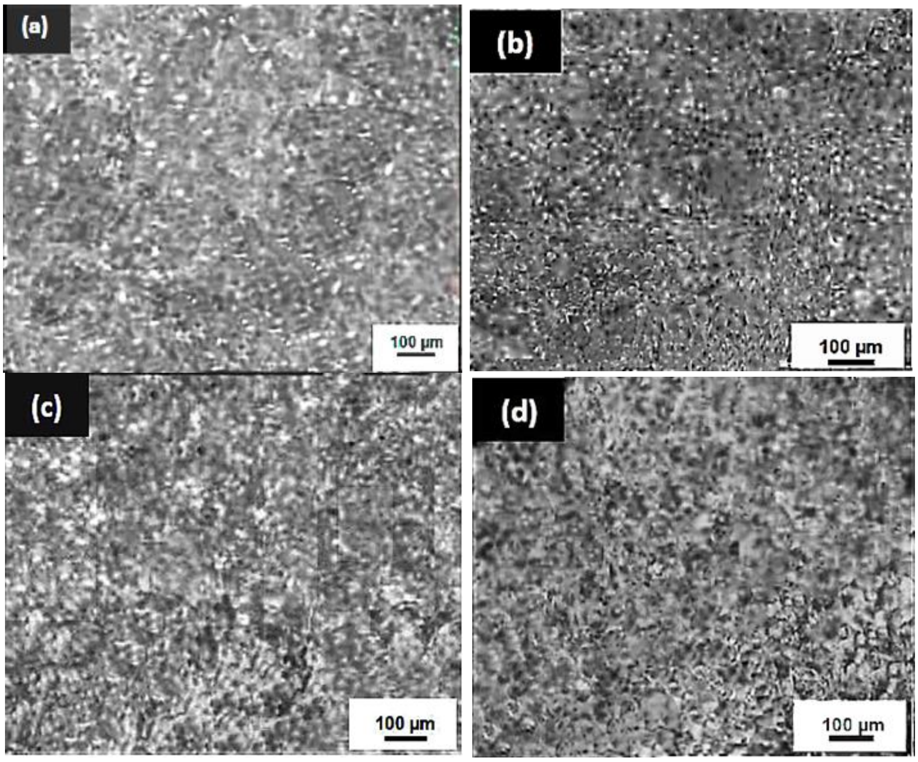
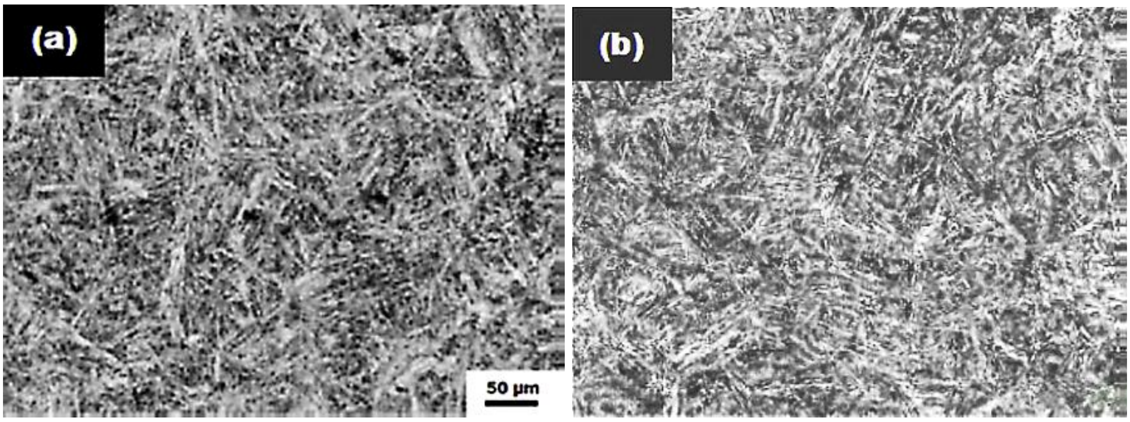


Figure 14. DCT/24h samples, with different austenitizing temperatures: (a) DCT/24h, 1020°C/30 min; (b) DCT/24, 1030°C/30 min; (c) DCT/24h, 1040°C, d) DCT/24h, 1050°C.

3.3.4. DCT Cryogenic treatment Samples + Post Tempering [Ta= 1050°C]



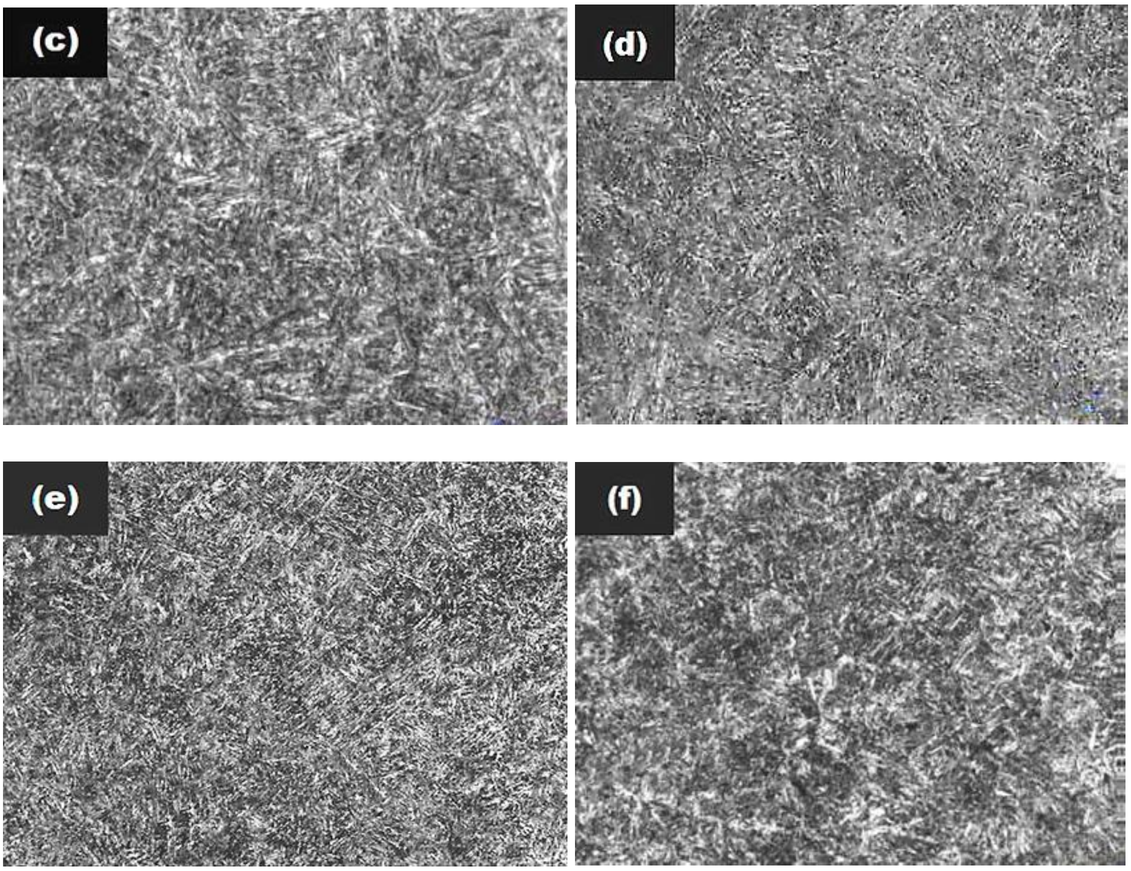


Figure. 15. Microstructures of the samples subjected to DCT + Post tempering: (a) 480°C; (b) 500°C; (c) 510°C; (d) 520°C; (e) 530°C; (f) 540°C. The micrographs have the same magnification (50μm). [Ta = 1050°C]

Figure 13 shows the microstructure after quenching, at different austenitizing temperatures. It is composed of lath martensite and the original grain size of the austenite can be clearly seen. The effect of austenitizing temperature on the microstructure and properties of martensitic stainless steels has been the subject of much research. Calliari et al**. [30]** reported that the maximum hardness upon cooling in martensitic stainless steel AISI 420 is found at an austenitizing temperature of 1050°C.

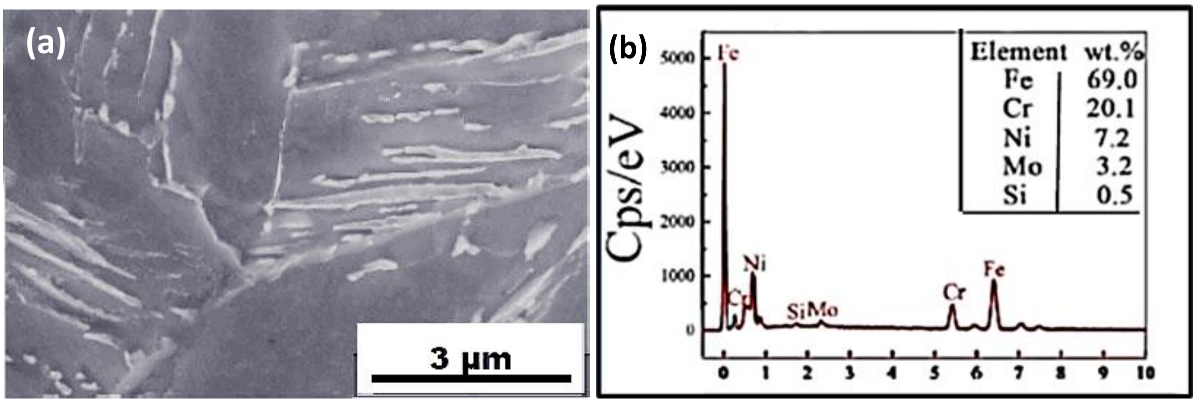


Figure 16. a) SEM photomicrograph of Figure (f) DCT + tempering at 540°C; b) EDS analysis of the sample. A high content of Cr, Ni and Mo is observed, elements that are strong carbide formers (white lines and dots)

It is assumed that at this temperature the complete dissolution of the carbides occurred. Tavares et al. **[31]** proposed the austenitizing temperatures in the range: 980-1100 °C for AISI 420 medium-carbon martensitic steel. This range is considered too wide to guarantee consistent hardness values in the quenched state. Latrobe **[32]** reported for stainless steel LSS 420 HC that it is completely austenitic after heating above 860 °C, with a peak hardness of 660 HV and minimally retained austenite after air quenching from 1020°C. The results are often contradictory, but in our case the best hardness and impact results have been achieved with an austenitizing temperature of 1050°C; being the starting point for a DCT treatment.

Fig. 14 shows the microstructures of the samples subjected to DCT/24h, with different austenitizing temperatures without subsequent treatment. We observed a carbide structure in a Lath martensite matrix. Fine carbides precipitate during deep cryogenic treatment. These newly formed fine carbides plus the larger original carbides form a denser, more coherent and much stronger matrix. Fine carbides are those that significantly improve hardness and wear resistance without significantly affecting impact toughness **[33].** It has been confirmed that DCT treatments facilitate the decomposition of retained austenite **[34, 35],** and that DCT treatments are the driving force for the transformation of retained austenite to martensite. However, the retained austenite cannot be completely converted to martensite; that is; DCT treatment, apart from minimizing the retained austenite, what it mainly does is give greater stability to the retained austenite.

In Fig. 15, the microstructures of the samples subjected to DCT + subsequent tempering are observed; For all tempering temperatures, a lath martensite structure is observed with thick primary carbides and very small fine secondary carbides and traces of retained austenite. As the tempering temperature increases, variations are observed in the martensite grains with very little variation in the shape, size and distribution of fine carbides, which is in accordance with the changes in hardness observed in Fig. 10.

Figure 16 is an amplified SEM microphotograph of a sample treated with DCT + tempered at 540°C, together with its corresponding EDS Analysis, which shows a high presence of Fe and Cr, indicating they are carbides enriched in (Cr and Fe). According to the studies of P. Jovicevic-Klug et al., these carbides of the investigated stainless steel must be of the types: $M_{23}C_6$, M_7C_3 and M_3C_2 **[36].**

In both treatment: DCT + Tempering and DCT + aging, there are variations; both in hardness and impact toughness, depending on the aging time or tempering temperature. The variations in properties observed in the present investigation are closely related to the variation in microstructure, due to the precipitated carbides that are formed, whose sizes are refined or thickened, depending on the process. It has been observed that the hardness and impact properties improve when these carbides are refined. For example: D. Das et al., studied the principles of microstructure formation in tool steels under cryogenic treatments found, that reducing the size of carbides and improving their spatial distribution are key factors to avoid loss of impact toughness when cryogenic treatments are applied **[37].** Da Silva et al., reported a 43% improvement in the impact toughness of high-speed steels when DCT treatment is applied, compared to conventionally treated samples. These results were attributed to both the transformation of the retained austenite and the precipitation of ultrafine carbides, the latter being considered the key factor for the changes in this property **[38]**

IV. CONCLUSIONS

In this work, the DCT + tempering and DCT + aging treatment at different austenitizing temperatures, and aging times, is analyzed in the effects on the Hardness, Impact Toughness and microstructure of AISI 420 martensitic stainless steel. The conclusions drawn are:

1. For all treatments; the hardness of the samples austenitized at 1050°C are considerably higher than the samples austenitized at 1040°C, which indicates the important role that the austenitizing temperature plays in cryogenic treatments.
2. The aged samples exceed the hardness of the tempered samples, obtaining the maximum hardness peak after 30 hours of aging.
3. For the DCT samples with subsequent aging, the impact toughness decreases with time, achieving a maximum value of 64.20J for 10h of aging; and a minimum of 40.10 J for 48 hours.
4. The DCT treatment with subsequent tempering almost does not affect the impact resistance of the material.
5. For austenitizing at 1050°C, the samples aged for 30 h show a maximum hardness value (58.7 HRC), and it is coupled with an impact toughness value (54J). On the other hand, the maximum value of toughness (64.2 J) is linked to a hardness value of (53.8 HRC), for 10 hours of aging.
6. If the service conditions require high hardness, it is recommended to carry out the DCT + aged treatment with 30 hours of immersion, to achieve the properties (58.7 HRC, 54J); But, if the requirement is to achieve high impact toughness, it is recommended to use the same treatment with 10 hours of immersion to achieve the properties (53.8 HRC; 64.2 J) with 10 hours of aging. Intermediate combinations are determined by the service conditions of the material.
7. In all microstructures, a lath martensite structure is observed surrounded by few primary carbides and abundant fine and ultrafine secondary carbides, even nanometric ones. The retained austenite is almost not observed. The fine and ultrafine carbides are those that improve the mechanical properties studied, and it has been possible to distinguish the $M_{23}C_6$ carbide.

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