



## Mechanical Properties of Martensitic Stainless Steel (AISI420) Subjected to Conventional and Cryogenic Treatments

Hareer S. Mohamed<sup>a\*</sup>, Ali H. Ataiwi<sup>b</sup>, Jamal J. Dawood<sup>c</sup>

<sup>a</sup> Materials Engineering Department- University of [Technology-Iraq.hareer.salman1@gmail.com](mailto:Technology-Iraq.hareer.salman1@gmail.com)

<sup>b</sup> Materials Engineering Department- University of Technology-Iraq. [130001@uotechnology.edu.iq](mailto:130001@uotechnology.edu.iq)

<sup>c</sup> Materials Engineering Department- University of Technology-Iraq. [130015@uotechnology.edu.iq](mailto:130015@uotechnology.edu.iq)

\*Corresponding author.

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### KEY WORDS

Martensitic Stainless Steel (MSS), Cryogenic treatment, Hardness, Tensile test.

### ABSTRACT

*Martensitic Stainless Steel (AISI420) MSS are vastly used because of their properties conventional which mix good mechanical and corrosion resistance. Cryogenic up to -196°C for different soaking time and heat treatments at (1000,500,200°C) for 15 minutes is one of the ways that used to enhance mechanical properties of these steels by means transformation of retained austenite, deformation regarding martensite then carbide refinement. the result showed an increase in tensile strength of samples that were treated cryogenically and tempered at 500°C was 933 (MPa) compared to samples that just treated conventionally in austenitizing and tempering at the same temperature that was 880 (MPa). The hardness values increased considerably to 414HV and 321 HV for the specimen that tempered at 200°C and 500°C respectively, precipitation of small carbides was observed that this is responsible for the improvement in the mechanical properties of the material.*

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## 1. Introduction

Cryogenic treatments have been employed in the development of the most recent three decades in both high-alloy and tool sheets of steel to increase wear resistance [1]. They are progressively used for steels and another group of alloys for enhancing their tribological performance [2]. Cryogenic Treatments (DCT) that associated with temperatures in around (-196°C) are given by using liquid nitrogen or liquid helium (-296°C) as cooling agents [1].

AISI 420 MSS are alloys that respond to heat treatment unlike furthermore kinds of stainless steel, with chromium contents (11-14 % ) and carbon ( 0.3-0.4% mass fraction ) so are usually used for production parts have superb mechanical properties and adequate corrosion resistance. Also, it can be used for making parts can withstand under low and high temperature and principally used in surgical tools, turbine blades, power generation, compressors, oil extraction, petrochemical, brake discs [3-

5]. The typical heat treatment complement for MSS consists of annealing to soften the steel, austenitizing for construction an austenitic structure and fully or partly dissolve carbide, cool down so, convert the austenite ( $\gamma$ ) to martensite (M), after that tempering of the new structure (M), to upgrade toughness and ductility. Nadig et al. debate the effects of cryogenic treatment on the wear behavior of MSS (SS410). It was found an enrichment of wear properties and hardness after cryogenic treatment [6]. Prieto et al. studied the influence of cryogenic treatment on the fracture toughness of an AISI 420 M.S.S, for both tool and high-alloy steels to develop their wear resistance, mainly within the transformation of retained austenite and the precipitation of fine carbides [2]. Khazaei and mollaahmedi investigated AISI 420 MSS that was exposed to rapid tempering, the influence of the mechanical properties was examined, explained that the precipitation of fine carbides M7C3 type may perhaps be answerable for the secondary hardening effect. In this event, dislocations might be locked by the small carbides, and so, UTS and YS [5]. The main aims of this work are, studying the effect of heat and cryogenic treatment on MSS type AISI 420. A comparison of obtained microstructure and mechanical properties obtained after conventional heat treatment accomplished at different tempering temperatures. Hoping to minimize the retained austenite to lower values and increase car-bides volume fraction percentage by adjusting many variables of the cryogenic treatments like; cryogenic temperature and soaking time.

## 2. Materials and Methods

### I. Materials

In this investigation, the used material is AISI 420 MSS which is provided from China. Table 1 displays the chemical composition of AISI 420 alloy. Its composition is confirmed with ASM standard in Metal Handbook Volume 3 [1] by using spectroscopy analyses. Figures 1 and 2 display TTT and phase diagram curve of the ternary Fe-Cr-C alloy and AISI 420 MSS. "Fe-Cr-C ternary system" phase diagram that used as a standard to recognize the metallurgy and the phase transformation on cooling and heating in martensitic stainless steel weldment [9]. at hardening temperature of 1000°C, carbides and  $\gamma$  are thermodynamically steady phases. Afterward quenching to room temperature, the  $\gamma$  transforms to M ( $M_s = 302^\circ\text{C}$ ,  $M_f = 204^\circ\text{C}$ ), so, the microstructure has to remain carbides [5].

### II. Abbreviations and Acronyms

DCT: Deep Cryogenic Treatments, CHT: Conventional Heat Treatments, specimen symbol, c=cryogenic, t=tempering, M: Martensite,  $\gamma$ : austenite, TTT: Transformation-Temperature – time, OP: the optical microscope.

### III. Specimens Preparation

The material has been cut by a water jet machine to get the final shape of tensile specimens. The samples were cut with shape and dimensions according to ASTM E8 standards, as shown in figure3.

### IV. Heat Treatment

In this research heat treatments were carried out by using laboratory furnace (type/ Carbolite 1200) in the Department of Materials Engineering, University of Technology.

The conventional heat treatments (CHT) include :-

- 1) Annealing. (An)
- 2) Hardening in air.
- 3) Tempering. (Aht200 tempering at 200°C and

Aht500 is tempering at 500°C).

While Deep Cryogenic treatments (DCT) include:

- 1) Annealing.
- 2) Hardening in air.
- 3) Cryogenic treatments
- 4) Tempering. (Ahct200 is tempering at 200°C and Ahct500 is tempering at 500°C).

All specimens which were annealed by heating to austenitic temperature are (850°C), soaking for 15 min and then cooling in furnace. After that, the samples were hardened by heating to an austenitic

temperature of 1000°C for 15 min cooling in air, and formerly either tempered at 200 °C or 500°C. They are called CHT treatments, or before tempering, they are subject to cryogenic treatment with three different media by gradual decreasing for the temperature to avoid thermal shock. The samples are put, first, in refrigerator at -20°C for 4 hr. After that, they are put in a dry ice at -70°C for 5 hr. Finally, the samples are put in LN at -196°C for 24 hr. This represents a cooling down procedure followed by warm-up in the same manner with reverse sequence ( from LN to dry ice to refrigerator respectively). Then, they are tempered at 200°C or 500°C in this case that is called DCT treatment.

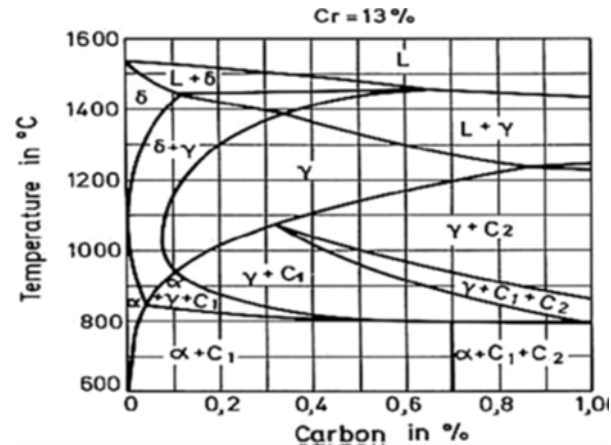


Figure 1: Sections of the binary Fe-C of Fe-Cr-C diagram with Cr = 13%; C1 carbides type (Cr,Fe)23C6, C2 carbides type (Cr,Fe)7C3 [7].

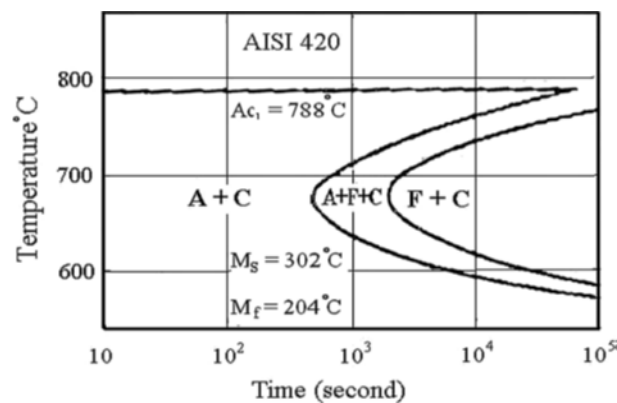


Figure 2: TTT curves for AISI 420 MSS [5].

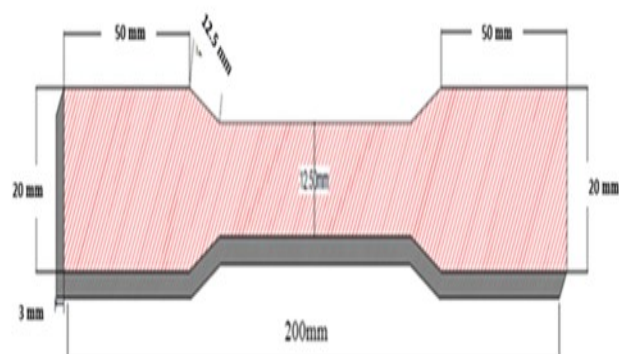


Figure3: The tensile specimens dimensions

Table 1: AISI 420 chemical composition

| Element | AISI 420 datasheet (wt%0) | Actual chemical composition of used steel (wt %) |
|---------|---------------------------|--|
| C       | $\geq 0.15$               | 0.347  |
| Si      | $\leq 1.00$               | 0.422  |
| Mn      | $\leq 1.0$                | 0.332  |
| S       | $\leq 0.03$               | 0.03   |
| P       | $\leq 0.04$               | 0.0156   |
| Cr      | 12-14                     | 14.11  |
| Fe      | Balance                   | Balance  |

### V. Metallography

After the former treatments, all specimens that are required for metallographic examination were prepared by using ASTM E3-11 standards ; sectioned, mounted, grounded with abrasive Sic papers maximum 2000 grit and refined to a shiny finish. The refined samples were etched with (HCl10 ml, and 100 mL ethanol) to reveal the microstructure. The finished specimens were inspected using OP and EDS.

### VI. Mechanical Testing

Tensile and micro hardness tests were done on treated samples. Vickers hardness device with a 98 N applied force and loading time was 15 S.

## 3. Results and discussion

### I. Microstructural Characterization of Base Alloy

#### 1) Microscopic Examination

The microstructure of AISI 420 MSS in the annealed condition, includes ferrite and M<sub>23</sub>C<sub>6</sub> (where M=Fe or Cr ) spheroid carbides, as shown in Figure 4. For hardened and tempered specimens at (200°C or 500°C), the microstructure involve retained austenite and undissolved dispersed fine carbides at 200 °C and coarse carbides at 500°C carbides dispersed in a tempered lath martensitic matrix as shown in Figure 5. At 500 °C, coarser M<sub>23</sub>C<sub>6</sub> and M<sub>7</sub>C<sub>3</sub> begin to grow at grain boundaries. Figure 6 shows microstructures of hardened cryogenic treated and tempering at 200°C (Ahct200°C) and 500°C (Ahct500°C). As compared to these structures with those of Figure 5, it reveals that in cryogenic treatment at (-80 °C and -196 °C) the microstructures comprise fine dispersed carbides and a fraction of retained  $\gamma$  and yields extra identical structure [9]. Prieto et .al [1] obtained the same results. The microstructure consists of martensitic matrix and carbides and very fine lenticular martensitic matrix and evenly distributed precipitated globular carbides.

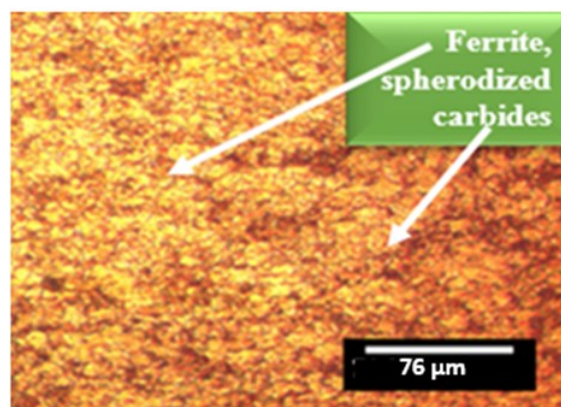


Figure 4: microstructure of the AISI 420 MSS in the annealed condition.

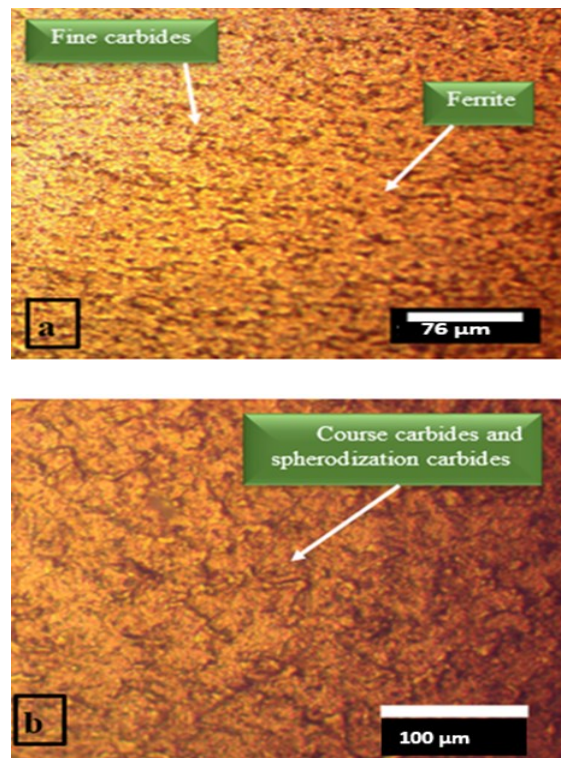


Figure 5: Microstructure of the AISI 420 in the condition of hardening and tempering (a) at 200 °C, (b) at 500°C.

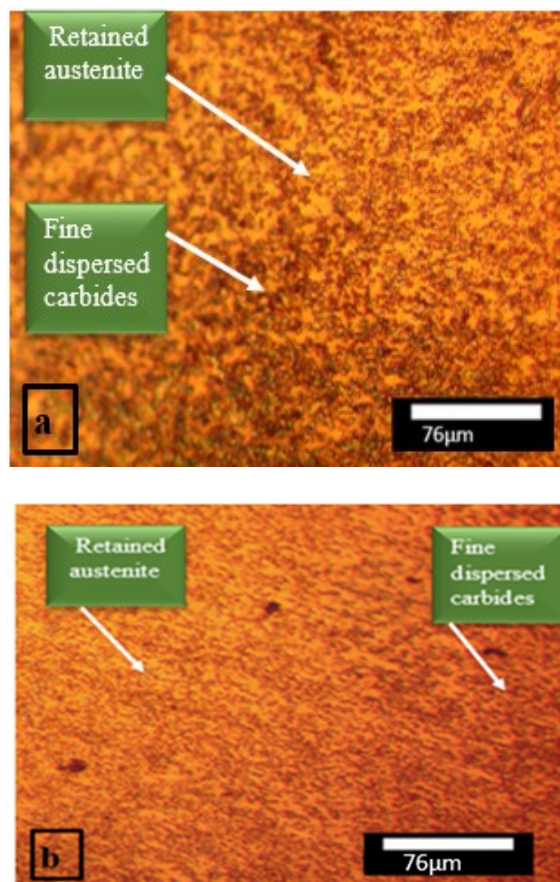


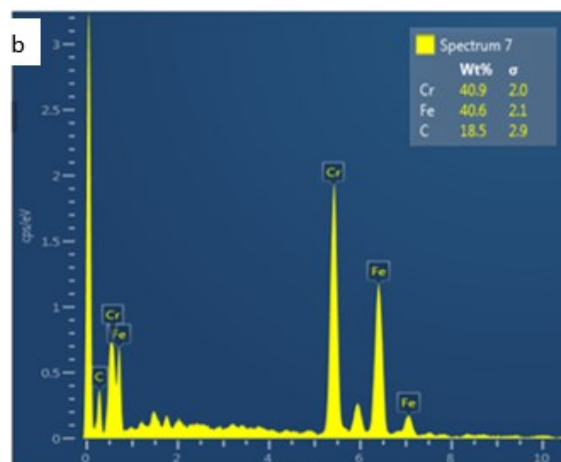
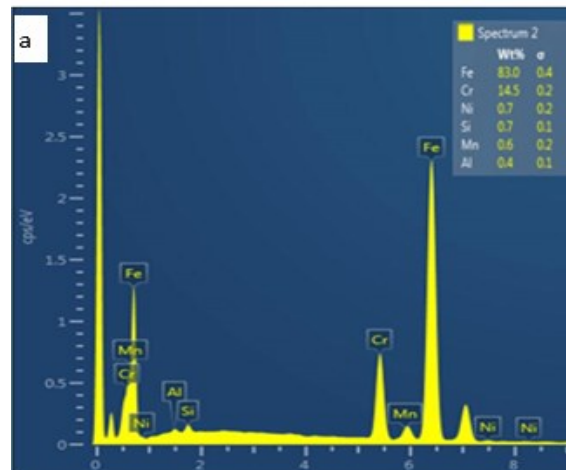
Figure 6: Microstructure of the AISI 420 in the condition of hardening, cryogenic and tempering (a) at 200 °C, (b) at 500°C.

4. Conclusions



## 2) Energy Dispersive (EDS) Spectroscopy

EDS analysis of hardened and tempered specimens at 200°C is shown in (Figure 6 a, b, c). EDS test was done at three locations. Figure (6a) represents elements of content in the base material. Also, revealed the carbides which are not dissolved and minor precipitated. Also, no Cr depletion is in the matrix (Cr 14%). The carbon contents are not observed in the matrix because they are very low percentage (0.34%). These findings have also been reported by [2]. Figure 7 (b, d) revealed chromium and iron content in the carbide particle and represent that there is chromium depletion for carbide particles, because of M<sub>23</sub>C<sub>6</sub> or M<sub>7</sub>C<sub>3</sub> carbides formation. EDS analysis of hardened, cryogenic treated and tempered specimens at 500°C are shown in Figure 7(d, e). They also revealed that Cr and Fe amount in the carbide particle, shown in Figure d, in the matrix (in Figure e). From this Figure, it is clear that there is more carbon in the carbides particle of samples that treated cryogenically than in conventional heat treatment.



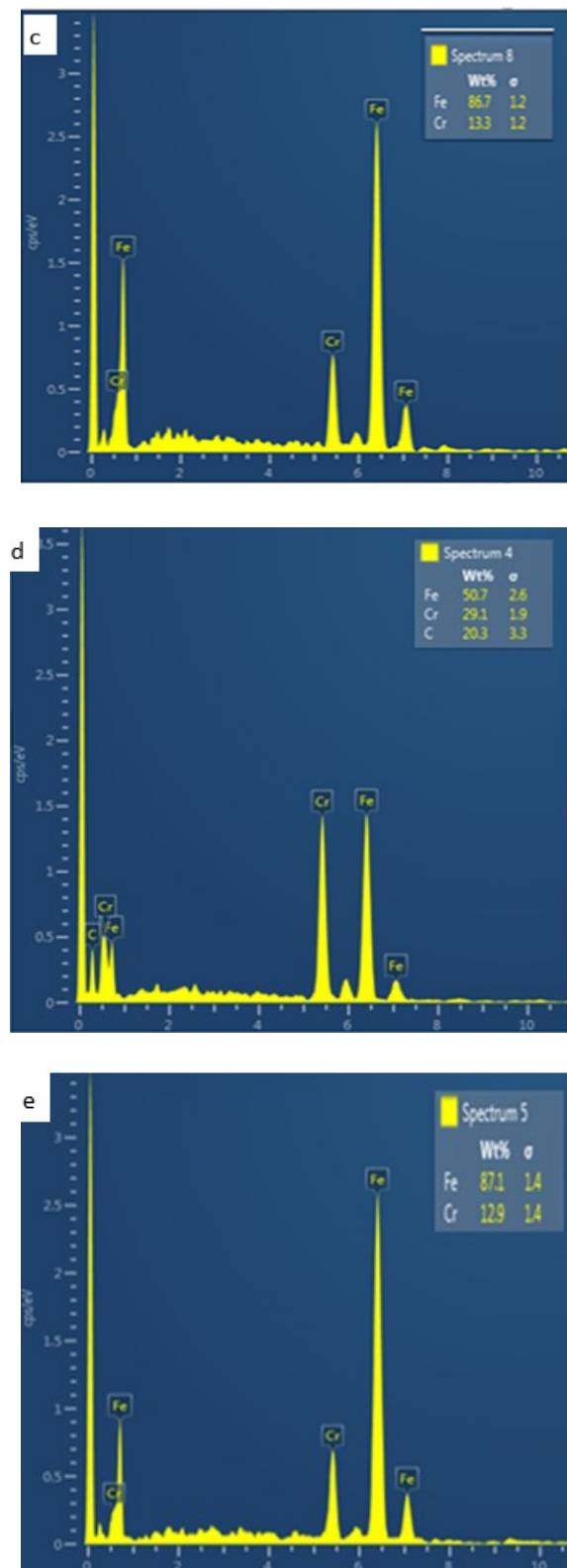


Figure 7: EDS analysis of specimens tempered at 200°C (a) Base metal (b) Carbides particle (c) point in matrix, (d) carbide particle of specimen cryogenically treated and tempered at 500 °C (e) point in matrix of specimen cryogenically treated and tempered at 500 °C.

#### 4. Mechanical Properties

##### 4.1. Hardness Evaluations

The hardness values after the heat and cryogenic treatments are shown in figure (8). This figure shows clearly that the heat treatment ways considerably affect the hardness of an AISI 420 MSS. The hardness value is 222 HV after annealing treatment. This lowest hardness value confirms the there is

no M and increased retained  $\gamma$ . To put it another way, the softening happened as soon as the M7C3 carbides start to coarsen, spheroidized and distributed homogenously in a ferrite matrix in its microstructure. The hardness increases after hardening and tempering at different temperatures (200 °C and 500 °C). This is because of the precipitation of carbide when the  $\gamma$  transforms to M during tempering. The hardness values increased considerably to 414HV and 321 HV for the specimen that tempered at 200°C and 500°C respectively. These high values can be recognized due to Cr carbides that dissolved gradually so the content of C and alloying element rises. The highest value of 414HV was gained at 200 °C (Aht200) that revealed in Figure 8 this is also due to the transformation of  $\gamma$  to M. The matrix contains a fine M and minimum amount of retained austenite ( $\gamma$ ) that was retained and more carbide, so these phases are the reason of high hardness values. Also, the C content in M is increased so, it increasing hardness. Because of the dissolution of C., therefore, the M becomes harder.

The hardness increment at a tempering temperature of 500°C which is called secondary hardening was stated by [4]. It is supposed that the formation of carbides cause of this occurrence. At this temperature, the hardness profile was noticeably decreased. Decreasing in the hardness was because of M recovery and carbide coarsening and also spheroidization of the carbides. The creation of M7C3 inside lath M is the reason for this effect. The M7C3 converts to coarsen and some of them change to M23C6, so be softer [4]. The great hardness is shown because of the homogenous distribution of lath M in the material microstructure. Also it is due to dissolved Cr and C in the M, which is increases lattice super saturation of the M [8]. The dissolution of the M23C6 carbide makes carbon super saturation and the lattice residual stress increases. Therefore, hardness increase is a consequence of that increases of the M. It is expected that the carbides will be dissolved in more amount due to the quicker cooling rate from the hardening to the room temperature, greater is the dissolution [8].

Hardness increment, for samples of AISI420 that treated cryogenically, can be attributed to the precipitation and distribution of finer small carbides, within the bulk of the material and the bigger strain state in the martensite, as reported by [1].

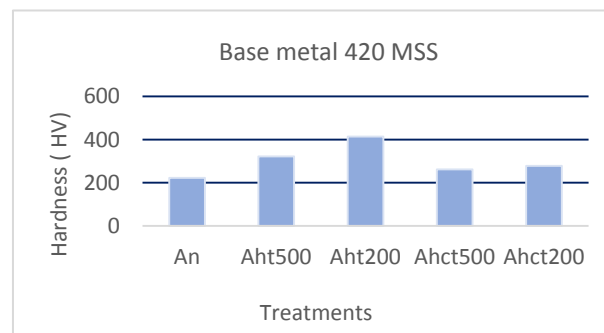


Figure 8: Hardness values after the heat and cryogenic treatments.

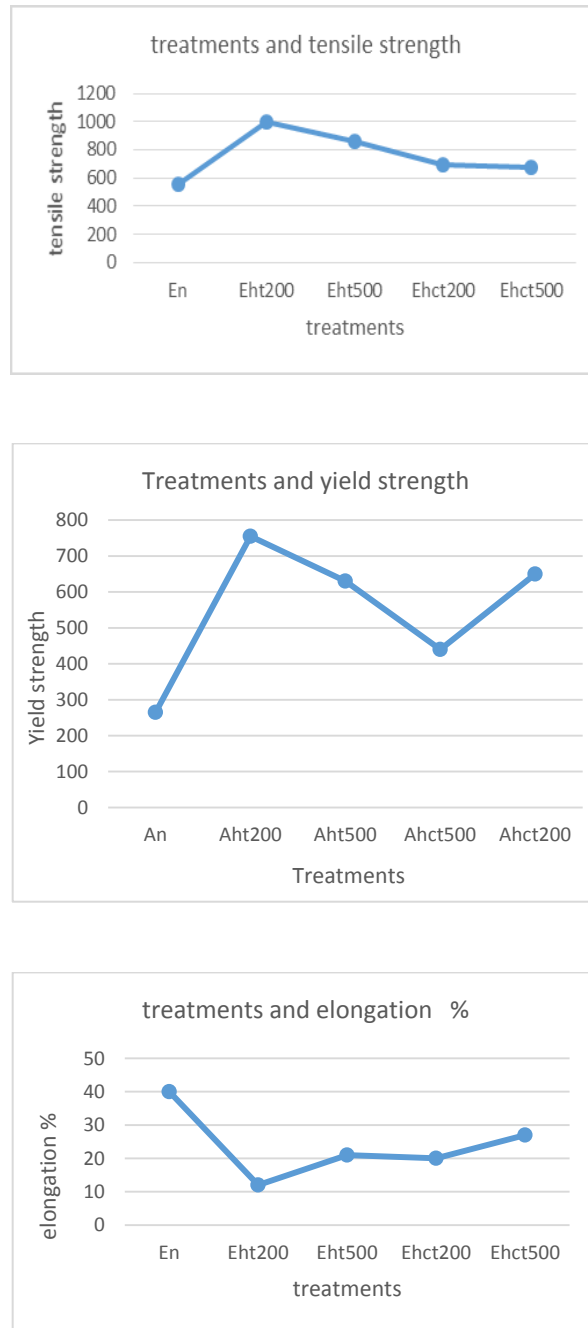
## II. Tensile Properties Evaluation

The tensile properties of treated specimens have been carried out at room temperature. Tensile strength, offset yield strength and percent elongation are shown in Table 2. Yield strength (YS), ultimate tensile strength (UTS) and the fraction of elongation (%EL) values were determined in Figure 9 as a function of different treatment. The maximum strength was acquired, for the specimen austenitic at 1000 °C and tempered at 200°C, is 989 MPa, the maximum value of yield strength is 755 MPa, while the elongation is 13.7%. Variations of tensile and yield strengths as a function of tempering temperature are as follows: Tensile strength have minimum value in annealing treatment after that it is increased and reach the highest amount at tempering at 200°C, it is then decreased in small amount and has a minimum value in cryogenic and tempering at 500 °C. The behavior of yield strength is similar to tensile strength. Elongation is at maximum values in annealing treatment, then is reduced when hardening and tempering at 500°C, further reduce when tempering at 200°C, increment again when cryogenic and tempering at 500°C.



**Table 2: Tensile properties of AISI 420 MSS**

| Material | Tensile strengths (MPa) | Yield strengths(MPa ) | Elongation % |
|----------|-------------------------|-----------------------|--------------|
| An       | 530                     | 265                   | 13.7         |
| Aht200   | 989                     | 755                   | 15           |
| Aht500   | 880                     | 630                   | 18.9         |
| Ahct200  | 664                     | 440                   | 15.2         |
| Ahct500  | 933                     | 650                   | 40           |

**Figure 9: Tensile strength, offset yield strength and elongation with treatments**

## 5. Conclusions

The effect of cryogenic and conventional heat treatments on the characteristic of AISI 420 MSS was investigated and the conclusions can be drawn:

1- When treated at 850 °C, the microstructure is composed of a ferrite matrix and a dispersion of M<sub>23</sub>C<sub>6</sub>.

- 2- The hardness (414 HV) and Tensile strength (989 MPa) are resulted from conventional austenitizing at 1000 °C and tempering at 200 °C.
- 3- Secondary hardening took place at tempering temperature 500 °C, the creation of M<sub>7</sub>C<sub>3</sub> inside the lath M is the reason for this effect. The M<sub>7</sub>C<sub>3</sub> convert to coarsen and some of them change to M<sub>23</sub>C<sub>6</sub>, so be softer
- 4- A good combination of mechanical properties done by the hardening at 1000 °C and tempering at 200 °C for 15 min.
- 5- DCT has enhanced the tensile and hardness properties, by the way of increasing precipitation of small size carbides.

## References

- [1] G. Prieto, J. Ipiña and W. Tuckart, "Cryogenic treatments on AISI 420 stainless steel: microstructure and mechanical properties," *Materials Science and Engineering*, Vol. 605, pp.236-243, 2014.
- [2] G. Prieto, W. Tuckart and J. Ipiña, "Influence of a cryogenic treatment on the fracture toughness of an AISI 420 Martensitic," Vol. 51, No. 4, pp. 591, 2016.
- [3] L. Barlow and M. Du Toit, "Effect of austenitizing heat treatment on the microstructure and hardness of martensitic stainless steel AISI 420," *Journal of Materials Engineering and Performance*, Vol. 21, No. 7, pp. 1327–1336, 2011
- [4] A. Isfahany, H. Saghafian, and G. Borhani, "The effect of heat treatment on mechanical properties and corrosion behavior of AISI420 martensitic stainless steel," *Journal of Alloys and Compounds*, Vol. 509, No. 9, pp.3931–3936, 2011.
- [5] A. Khazaei and A. Mollaahmadi, "Rapid tempering of martensitic stainless steel AISI420: microstructure, mechanical and corrosion properties," *Journal of Materials Engineering and Performance*, Vol. 26, No. 4, pp. 1626–1633, 2017.
- [6] D. Nadig, P. Shivakumar, S. Anoop, K. Chinmay, P. Divine, and H. Harsha, "Effects of cryogenic treatment on the wear properties of brake discs," *IOP Conference Series: Materials Science and Engineering*, *Materials Science and Engineering*. Vol. 171, 2017.
- [7] V. Macro and C. Andrea, "Stainless steels," *Lucefin S.p.A.* 1-25040 Italy, 2014.
- [8] C. Scheuera, R. Fraga, R. Cardoso, S. Brunatto, "Effects of heat treatment conditions on microstructure and mechanical properties of AISI 420 steel," 21° CBECIMAT - Congresso Brasileiro de Engenharia e Ciência dos Materiais 09 a 13, Cuiabá, MT, Brasil, 2014.
- [9] L. Ramos, L. Simoni, R. Mielczarski, M. Vega, R. Schroeder, and C. Malfatti, "Tribocorrosion and electrochemical behavior of DIN 1.4110 martensitic stainless steels after cryogenic heat treatment," *Materials Research*, Vol. 20, No. 2, pp.460–468, 2017.
- [10] E. E. Reber, R. L. Michell, and C. J. Carter, "Oxygen absorption in the earth's atmosphere," *Aerospace Corp.*, Los Angeles, CA, Tech. Rep. TR-0200 (4230-46-)-3, 1988.