

The effect of heat treatment on mechanical properties and microstructure of the AISI 422 martensitic stainless steel

H. Babaei*, K. Amini**, A. Shafyei***

*Department of Materials Engineering, Najafabad Branch, Islamic Azad University, Najafabad, Iran

**Department of Mechanical Engineering, Tiran branch, Islamic Azad University, Tiran, Iran

***Department of Materials Engineering, Isfahan University of Technology, Isfahan, Iran

crossref <http://dx.doi.org/10.5755/j01.mech.22.6.13599>

1. Introduction

Martensitic stainless steels have good mechanical properties and good corrosion resistance. For these reasons, they have a wide application in petrochemical, marine, aerospace, and food industries [1-5]. AISI 422 is used in nuclear power plants, dental tools, and surgical knives. Heat treatment plays an important role in this grade. Hence precise control of the heat treatment process to obtain martensite layers, least delta ferrite, and retained austenite is very important [6]. Moreover, carbon and alloy content in austenite have a substantial effect on strength and impact energy. The proposed heat treatment for grade AISI 422 is: a) austenitizing to temperature 1000-1050°C for 60 min and quenching in oil; b) tempering in the temperature range of 200-300°C for 60 min for achieving high tensile strength and fair toughness and second tempering in the range of 600-700°C for 60 min is preferred for getting fair strength with high toughness which should be carried out carefully [7].

Studies by Shafer et al. [8] on Cr-martensitic steels were carried out to find the effect of delta ferrite and dendritic carbides precipitated on the delta ferrite boundaries which caused the drop in impact energy. Wang et al. [9] believed that the presence of delta ferrite in microstructure reduced the impact energy in martensitic steel 13Cr-4Ni. In studies by Calliari et al. [10] on martensitic steel Ni-Mo to find out effect of tempering temperature on hardness, revealed the effect of secondary hardening in temperatures 500-560°C. Type AISI 422 is a hardenable (martensitic) 13% chromium stainless steel that is a modification of type AISI 420. The modification consists of additions of nickel, molybdenum, tungsten and vanadium that improve the elevated temperature strength and resistance to stress corrosion and minimize anisotropic behavior. Good creep-rupture properties of about 480°C and resistance to stress-corrosion cracking in the heat treated condition make this grade suitable for applications in jet engines. Other uses include buckets for steam and gas turbines, discs, bolts, wheel and blades, as well as structural components of aircraft and missiles. This alloy is also used for a number of valve applications. Given the need to obtain high strength and impact energy in grade AISI 422, in present research, the effect of austenitizing temperature and tempering time on strength and impact as well as microstructure has been evaluated.

2. Testing procedures

The composition of studied martensitic stainless steel AISI422 was as follows (in wt%): 0.21 C, 0.29 Si, 0.62

Mn, 0.022 P, 0.006 S, 12 Cr, 0.96 Mo, 0.9 Ni, 0.11 Cu, 0.014 Al, 0.26 V, 1 w. A billet with final diameter 397 mm and length 2000 mm obtained in an electric furnace was forged at 1150-1180°C and annealed at 780°C. The specimens were heat treated at a rate of 8 K/min to 620°C, held for 30 min, continued heating with the same rate to the austenitizing temperature of 1040°C (SA1) and 1070°C (SA2), holding time of 60 min and then quenched in oil at 30°C, and finally tempered at 700°C for 120 min. To study the effect of the tempering temperature on mechanical properties, the specimens were subjected to austenitization at 1040°C for 60 min, quenched in oil and tempered for 2 h at various temperatures, i.e., 300, 400, 500, 600, 700. To study the effect of the duration of the tempering the specimens of the SA1 series were tempered at 700°C for 2 and 5 h. Then they were polished against a No. 1000 (the number of grain per square inch) emery paper to provide a uniform surface roughness.

The tensile test was performed using a ZWICK Z 250 tensile testing machine in accordance with the DIN 250120 standard at room temperature ($30 \pm 5^\circ\text{C}$, moisture content $50 \pm 10\%$) at a deformation rate of 4 mm/min. The impact test was performed by the Charpy method in accordance with EN 100-45 standard. The Rockwell hardness was measured using an EMCO M4U 750 hardness tester in accordance with DIN-EN 6508. The x-ray diffraction studies were performed using a Philips PW37 diffractometer in CuK_α radiation. The grain size was determined according to the ASTM E112 standard. The structure was studied by using an Olympus PMG3 microscope. The structure was etched with a Villela etchant (1 g picric acid, 10 ml hydrochloric acid, 100 ml ethanol). The EDS analysis was utilized to identify the carbides decomposed at different tempering temperature

3. Results and discussion

3.1. The effect of austenitizing temperature

Microstructure of the specimens after treatments SA1 and SA2 is presented in Fig. 1, a, b. When the austenitizing temperature is increased from 1040 to 1070°C, the grain size increases from the initial No. 8 to No. 7 (ASTM), due to the dissolution of the carbides. Fig. 1, c, d shows the tempered martensite layers. Table 1 presents the values of the hardness of the ultimate rupture strength and the impact energy. When the asutenization temperature is increased from 1040 to 1070°C, the hardness and ultimate rupture is increased. Variation of hardness and ultimate rupture

strength can be attributed to the increase of alloying elements such as chromium and carbon in the austenite with the increase of temperature.

The increase of the temperature increases the carbide dissolution in the matrix. Garcia et al. [1] showed that in the AISI 420 stainless steel, the increase of austenitizing temperature decreased carbides from 9.9% to 6.6%. The strength increase is resulted from the dissolution of the $M_{23}C_6$ carbide in the matrix which increases the carbon super-saturation and lattice distortion of the martensite [11] together with the amount of twin boundaries and dislocations in the microstructure [12].

Refinement of the austenite grains causes an increase in the impact energy of specimen SA1 as compared to specimen SA2. In studies by Choi et al. [13] on alloy 03Cr-14Cr-3Mo about effect of austenitizing temperature and primary austenite grain size has been investigated. In this study it is believed that more dissolution of carbides by increasing the austenitizing temperature from 1000 to 1030°C was the

main cause of grain growth.

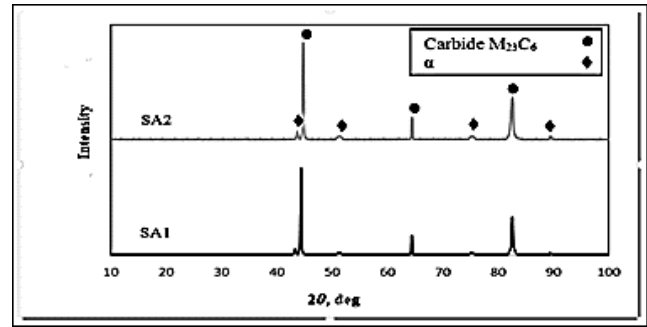


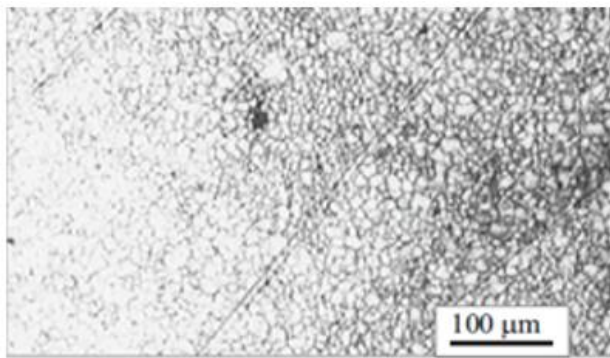
Fig. 2 X-ray diffractogram of specimens SA1 and SA2

The diffractogram of specimens SA1 and SA2 show the presence of an $M_{23}C_6$ carbide in the final structure (Fig. 2).

Table 1

Hardness (HRC), Ultimate rupture strength (σ_r), and Impact energy (U_{im}) of stainless Steel AISI 422

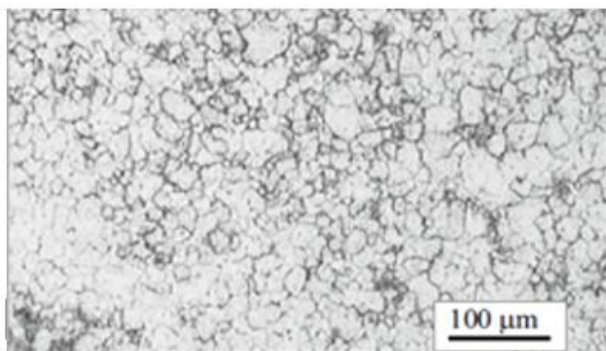
Specimen	HRC	σ_r , MPa	U_{im} , J
SA1	30	1041	27.7
SA2	36	1059	20



a



c



b



d

Fig. 1 Microstructure of steel AISI 422 specimens after different treatments: a - initial austenite grain of SA1; b - initial austenite grain of SA2; c - tempered martensite layer of SA1; d - tempered martensite layer of SA2

3.2. The effect of tempering temperature

The optimum impact energy was observed when austenitizing occurred at 1040°C for 60 min. This cycle was selected for the next tests. To evaluate the effect of temper-

ing temperature on mechanical properties and microstructure, tempering treatments were conducted in the range of 300-700°C for 2 h. In Fig. 3 the hardness value of the specimens quenched and tempered at various temperatures are shown.

As can be seen, hardness remains almost constant

for tempering temperatures ranging from 300°C to 400°C. Increasing hardness during tempering in the range of 400-500°C can be attributed to the secondary hardening phenomenon. This is generally associated with the formation of the M_7C_3 carbides within the martensite lath. It can also be observed in Fig. 3 that hardness decreases when tempering in the range of 500-700°C. This softening occurred when the M_7C_3 carbides started to coarsen and partially transform to the $M_{23}C_6$ carbides. Fig. 5 shows the result of EDS analysis for the specimens that were tempered at 500°C and 700°C respectively. EDS results together with the results reported by Calliari et al. [10] in similar stainless steel showed that the identified carbides at 500°C and 700°C could be M_7C_3 and $M_{23}C_6$ respectively.

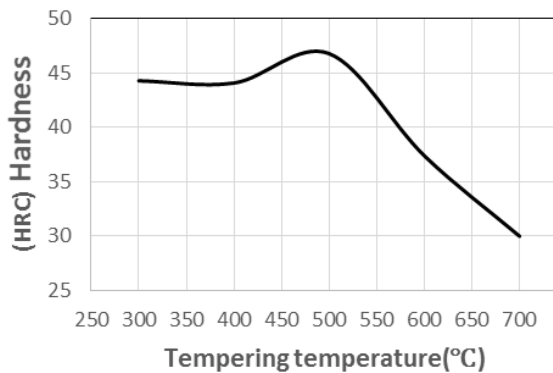


Fig. 3 hardness versus tempering temperatures

Studies by Balan et al. [3] on martensitic stainless steel 16Cr-2Ni shows the secondary hardening in the temperature range of 300–450°C. They believed that the precipitation of the M_7C_3 carbides for secondary hardening and the tempering temperature of 450 – 650°C the transformation of the M_7C_3 carbides to $M_{23}C_6$ as well as grain growth. Amini et al. [14] reported that secondary hardening occurred in martensitic stainless steel DIN 1.4057 in the range of 350-420°C due to precipitation of the M_7C_3 carbides and resulted

decreasing hardness in the range of 450-650°C is due to M_7C_3 to $M_{23}C_6$ transformation. These studies are similar to the result of studies by Naseri et al. [7] on martensitic stainless steel AISI 420 in which the secondary hardening effect was seen at 400 – 500°C.

The impact strength (Fig. 4) decreased when tempering temperature increased from 300-500°C so that the least impact strength was observed at 500°C. Reduction of impact energy with increasing the tempering temperature to 500°C could have been resulted from reducing the retained austenite volume fraction, increasing carbide particle and also re-precipitation of phosphorous at grain boundaries [2]. Impact strength increased again at 700°C, because martensite tempered and a recovery took place at this temperature.

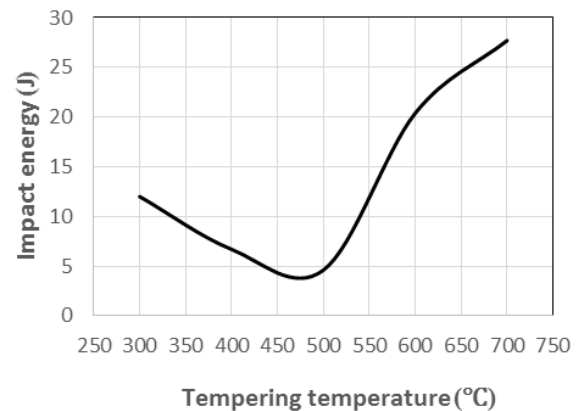


Fig. 4 Impact energy versus tempering temperatures

In Table 2 the behavior of yield strength and tensile strength by tempering temperature is shown. As can be seen, with increasing the tempering temperature, in the beginning the yield and tensile strength change inconsiderably but in the range 400-500°C, both properties increase and again in the range 500 – 700°C the yield and tensile strength decrease.

Table 2

Ultimate rupture strength and yield strength of steel ASI 422 as a function of the tempering temperature

Tempering temperature, °C	Yield strength, MPa	Ultimate rupture strength, MPa
300	1359	1579
400	1366	1598
500	1394	1620
600	1130	1270
700	883	1041

3.3. The effect of the tempering time

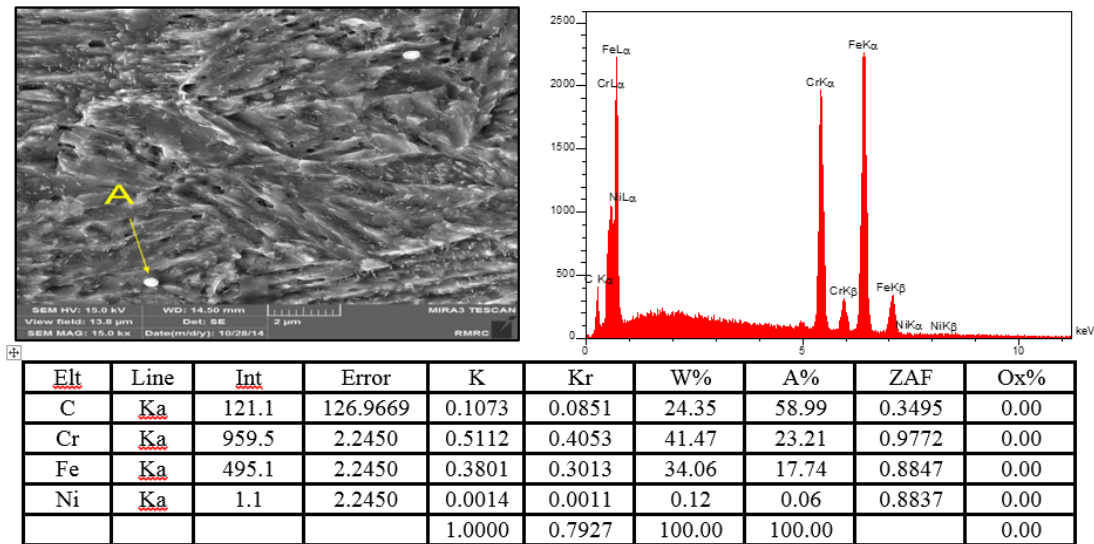
To evaluate the effect of the tempering time on specimen (austenitized at 1040°C), it was tempered at 700°C for 2 and 5 hours. Then the impact test was carried out on the specimens. The result is shown in Table 3.

Table 3

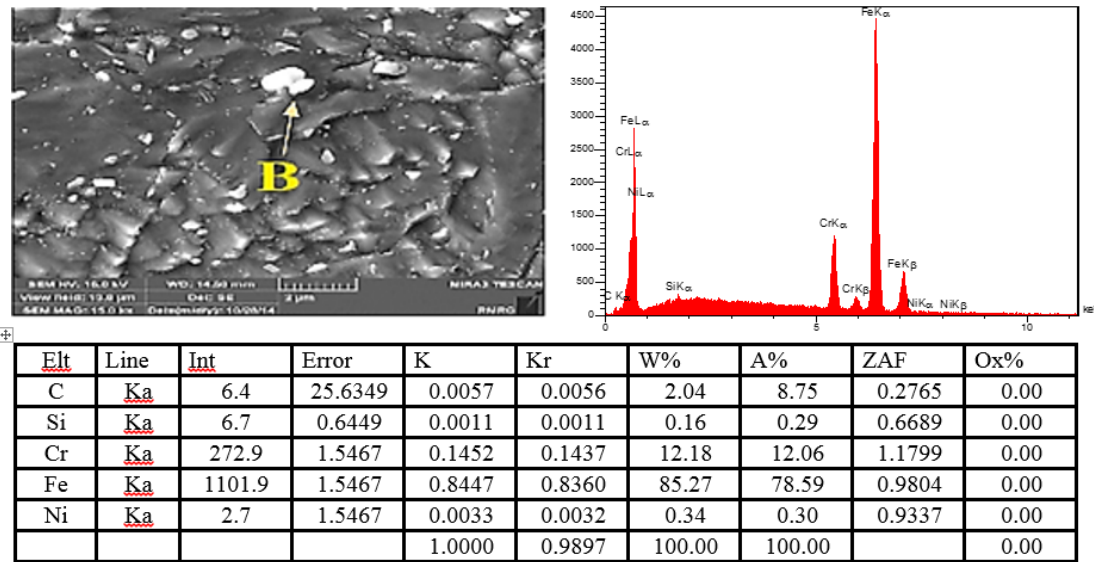
Impact energy of the AISI 422 steel as a function of the duration of tempering

Specimen	Tempering time, h	Impact energy, J
SA1-2h	2	27.7
SA1-5h	5	35.1

Also, the microstructure of these specimens (SA1-2h&SA1-5h) was studied by SEM (Fig. 6). By increasing the tempering time from 2 to 5 hours, the size of the precipitated carbides increased from 0.59 μm to 0.74 μm . According to Table 3, the impact energy increases from 27.7 to 35.1 Joules. The studies by Ning et al. [15] on hyper martensitic stainless steel at the tempering temperature of 600°C for 6 and 12 hours showed that with increasing the tempering time, the retained austenite grains are elongated and the percentage of retained austenite increases and they inferred that the percentage of retained austenite is the cause of increase in the impact energy.



a



b

Fig. 5 The EDS analysis of specimens tempered at (a) 500°C and (b) 700°C

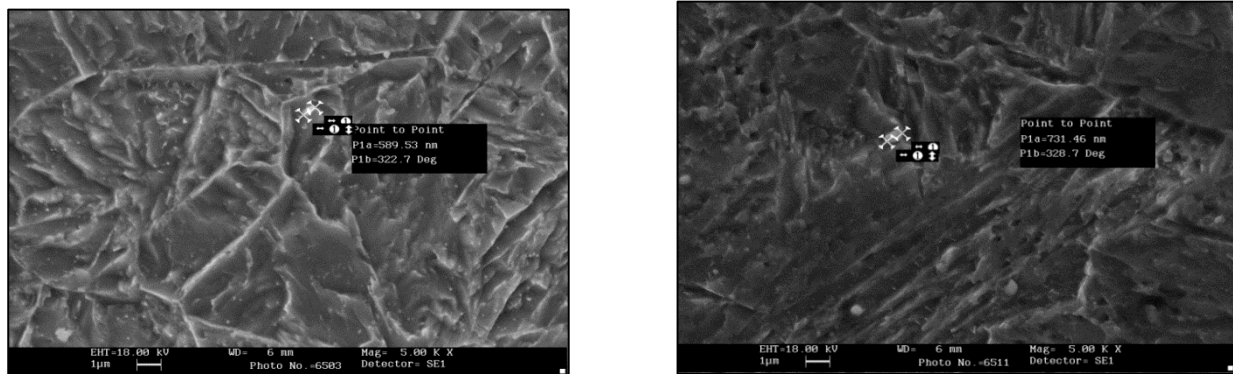


Fig. 6 SEM micrographs: austenitizing at 1040°C for 60 min and tempered at 700°C for: a - 2 h; b - 5 h

4. Conclusions

1. In the range of austenitizing temperature from 1040 to 1070°C, the austenite grain size increases and the

impact energy decreases from 27.7 to 20 J.

2. Secondary hardening took place at the tempering temperature in the range of 400-500°C due to the for-

mation of M_7C_3 within the martensite lathes. Softening occurred when the M_7C_3 carbides start to coarsen and partially transform to the $M_{23}C_6$ carbides.

3. A good combination of mechanical properties can be achieved by the austenitizing at 1040°C and tempering at 700°C for 2 h.

4. With increasing the tempering time from 2 to 5 h, the impact energy increases from 27.7 to 35.1 J, and the cause is understood as the increase of the precipitated carbides size.

References

- Garcia de Andres, C.; Alvarez, L.F.** 1993. Optimization of properties obtained by quenching martensitic stainless steel X30-40Cr13 and X40-60CrMoV14, *Journal of Materials Science* 28: 1264-1268. <http://dx.doi.org/10.1007/BF01191962>.
- Bhambri, S.K.** 1986. Intergranular fracture in 13 wt.% chromium martensitic stainless steel, *Journal of Materials Science* 21: 1741-1746. <http://dx.doi.org/10.1007/BF01114734>.
- Balan, K.P.; Venugopal Reddy, A.; Sarma, D.S.** 1999. Effect of single and double austenitization treatments on the microstructure and mechanical properties of 16Cr-2Ni steel, *Journal of Materials Engineering and Performance* 8: 385-393. <http://dx.doi.org/10.1361/105994999770346963>.
- Porter, D.A.** 1981. Phase Transformation in Metals and Alloys, Von Nostrand Reinhold.
- Garcia de Andres, C.; Alvarez, L.F.; Lopez, V.** 1998. Effects of carbide-forming elements on the response to thermal treatment of the X45Cr13 martensitic stainless steel, *Journal of Materials Science* 33: 4095-4100. <http://dx.doi.org/10.1023/A:1004424329556>.
- Manova, D.; Mandl, S.M.** 2005. Wear behavior of martensitic stainless steel after PIII surface treatment, *Journal of Surface and Coating Technology* 200(1-4): 137-140. <http://dx.doi.org/10.1016/j.surfcoat.2005.02.085>.
- NaseryIsfahany, A.; Saghafian, H.; Borhani, Gh.** 2011. The effect of heat treatment on mechanical properties and corrosion behavior of AISI420 martensitic stainless steel, *Journal of Alloys and Compounds* 509: 3931-3936. <http://dx.doi.org/10.1016/j.jallcom.2010.12.174>.
- Schafer, L.** 1998. Influence of delta ferrite and dendritic carbides on the impact and tensile properties of a martensitic chromium steel, *Journal of Nuclear Materials* 258-263: 1336-1339. [http://dx.doi.org/10.1016/S0022-3115\(98\)00200-1](http://dx.doi.org/10.1016/S0022-3115(98)00200-1).
- Wang, P.; Lu, S.P.; Xiao, N.M.; Li, D.Z.; Li, Y.Y.** 2010. Effect of delta ferrite on impact properties of low carbon 13Cr-4Ni martensitic stainless steel, *Journal of Materials Science and Engineering: A* 527: 3210-3216. <http://dx.doi.org/10.1016/j.msea.2010.01.085>.
- Calliari, I.; Zanesco, M.; Dabala, M.; Brunelli, K.; Ramous, E.** 2008. Investigation of microstructure and properties of a Ni-Mo martensitic stainless steel, *Materials and Design* 29: 246-250. <http://dx.doi.org/10.1016/j.matdes.2006.11.020>.
- Candelaria, A.F.; Pinedo, C.E.** 2003. Influence of the heat treatment on the corrosion resistance of the martensitic stainless steel type AISI 420, *Journal of Material Science Letters* 22: 1151-1153. <http://dx.doi.org/10.1023/A:1025179128333>.
- Gonzalez-Rodriguez, J.G.; Bahena-Martinez, G. Salinas-Bravo, V.M.** 2000. Effect of heat treatment on the stress corrosion cracking behaviour of 403 stainless steel in NaCl at 95°C, *Materials Letters* 43: 208-214. [http://dx.doi.org/10.1016/S0167-577X\(99\)00261-X](http://dx.doi.org/10.1016/S0167-577X(99)00261-X).
- Choi, Y.S.; Kim, J.G.; Park, Y.S.; Park, J.Y.** 2007. Austenitizing treatment influence on the electrochemical corrosion behavior of 0.3C-14Cr-3Mo martensitic stainless steel, *Materials Letters* 61: 244-247. <http://dx.doi.org/10.1016/j.matlet.2006.04.041>.
- Amini, K.; Hoda, M.R.; Shafyei, A.** 2014. Investigation of the effect of heat treatment on the mechanical properties and microstructure of DIN 1.4057 martensitic stainless steel, *Journal of Metal Science and Heat Treatment* 55: 499-503. <http://dx.doi.org/10.1007/s11041-014-9661-9>.
- De-Ning, Z.; Ying, H.; Wei, Zh.; Xu-Dong, F.** 2010. Influence of tempering process on mechanical properties of 00Cr13Ni4Mo super martensitic stainless steel, *Journal of Iron and Steel Research, International* 17(8): 50-54. [http://dx.doi.org/10.1016/S1006-706X\(10\)60128-8](http://dx.doi.org/10.1016/S1006-706X(10)60128-8).

H. Babaei, K. Amini, A. Shafyei

THE EFFECT OF HEAT TREATMENT ON MECHANICAL PROPERTIES AND MICROSTRUCTURE OF THE AISI 422 MARTENSITIC STAINLESS STEEL

S u m m a r y

Martensitic stainless steel has a wide range of applications in steam generators and food industries like mixing blades due to high mechanical and good corrosion resistance. In this research, the effect of austenitizing temperatures (1040°C -1070°C), tempering temperatures (300, 400, 500, 600 and 700°C) and also the tempering times of (2 and 5 h) on microstructure and mechanical properties of martensitic stainless steel X20CrMoWV121 (AISI 422) has been investigated. The results of the experiments showed that with increasing the austenitizing temperature from 1040 to 1070°C the impact energy is reduced, and the hardness and yield strength are increased. These changes are due to the increase in the austenite grain size. The secondary hardness occurs in the tempering temperature in range of 400-500°C. The results revealed that after tempering, an increase in hardness and strength and a reduction in toughness occurred in the samples. Also, with increase in the tempering time from 2 to 5 hours at 700°C, the toughness increased and hardness as well as strength were reduced due to the precipitation of the carbides.

Keywords: Martensitic stainless steel AISI 422, mechanical properties, heat treatment.

Received November 23, 2015

Accepted November 25, 2016