An Investigation on Mechanical Properties of Ultrafine Grained 316 Stainless Steel by Thermomechanical Treatment

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Abstract

In this paper, an advanced thermo-mechanical treatment was conducted on AISI 316 austenitic stainless steel. At first, three samples were rolled at the ambient temperature, the temperature of -20 ºC (dry ice and ethanol) and -196 ºC (liquid nitrogen). Then, the samples were annealed at 800 ºC in the time range of 1 to 15 minutes. In each step, the microhardness values of the samples were measured. Microstructural investigations were conducted using optical and SEM microscopes and Clemex software. Results showed that microhardness of the samples increased due to the formation and precipitation of carbides. The formation of martensitic microstructure after the rolling process was revealed by X-ray diffraction analysis; and by decreasing the rolling temperature, the peak intensity increased. Also, by increasing the annealing time in each step, the volume fraction of the reverted austenite and the intensity of the austenite peaks increased.

Keywords: Austenitic Stainless Steel; Ultrafine Grain; Thermo-Mechanical Processing, X-ray Diffraction Analysis; Mechanical Properties.

1. Introduction

Austenitic stainless steels are widely used in industry due to their excellent ductility and good corrosion resistance [1-4]. Relatively low strength and especially low yield strength of this kind of steels have limited their structural applications. The strength of these steels is equal to mild steels and since they have austenitic microstructure at the ambient temperature, they cannot be transformation hardened [5-7]. There are several ways to increase the strength of stainless steels, including formation of solid solution, work hardening, introducing a second phase into the matrix and grain refining. Among different strengthening mechanisms, grain refinement is the only method which leads to spontaneous improvement of strength and ductility.

In recent years, several methods have been used to produce ultrafine-grain steels such as severe plastic deformation (SPD) subcategories including: equal channel angular extrusion (ECAP), accumulative roll bonding (ARB) and high pressure torsion (HPT) processes. These techniques have been investigated from two different approaches, namely severe plastic

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deformation methods and advanced thermo-mechanical processes. Both of these approaches basically embrace improved processes of rolling steel in large dimensions which are operated easier in different temperatures.

In addition, phase transformation and controlled rolling are used in advanced thermo-mechanical methods, as well [8]. The combination of high strength and ductility is essential for future generations of high-strength steels. The important issue involved in reducing the grain size of stainless steels is their corrosion susceptibility, which is the prime purpose of producing these steels [9]. In some grades such as 300stainless steels, austenite is in meta-stable form that is easily transformed to martensite below the Md temperature by cold-working. However, in more stable alloys, such as 321 and 316 grades, the austenite phase is stable that initial dislocation density and cold-working temperature have critical effects on martensite transformation.

By elevating the temperature of martensite microstructure to annealing temperatures, martensite to austenite transformation occurs that leads to grain refinement [9-11]. Grain refining by thermo-mechanical processes increases performance for high-temperature applications and corrosion resistance for many alloys. This is because of economic considerations plus achieving desired strength and flexibility. In recent years, some studies have been conducted on the grain refining of austenitic stainless steels by this method [12-16]. For example, Schino et al. [17] investigated the effects of martensite formation and austenite reversion on the grain refining of AISI 304 stainless steel and observed that in quenched-rolled samples, almost all of the austenite was transformed to martensite phase in the matrix. They also found that the yield strength and ultimate strength increased and the toughness decreased by reducing the grain size from 20 μm to about 1μm. In this way, several researches on the austenitic stainless steels have been conducted.

In another study, Kumar et al. [18] studied the effect of thermal cycles on heavily cold-deformed AISI 304L austenitic stainless steels. Their results showed that the cycle of annealing process prevented the progressing kinetics in a way that structure grading was modified in comparison with isothermal annealing. This difference could be due to structural strain and non-uniformity which is imposed after each annealing cycle. Momeni and Abbasi [19] carried out an study on the behavior of several austenitic stainless steels and semi-austenitic

microstructures in thermo-mechanical processes. In their study, 304, 304L and 301 stainless steels were compared. They used cold-rolling – anneal to apply thermo-mechanical process. They found out that annealing twining which was formed in 304L steel higher than 304 steel and much higher than 301 alloy. Finally, Kumar et al. [20] investigated the formation of ultrafine grain microstructure in the A316L austenitic stainless steel and its impact on tensile properties. They observed that the annealing time and temperature had a significant effect on the final grain size of the cold-worked stainless steel samples.

In the present investigation, an advanced thermo-mechanical treatment was conducted on an AISI 316 austenitic stainless steel. For this purpose, the steel samples were cold-rolled at different temperatures, annealed at 800 ºC for 1-15 minute(s) and then cooled at the ambient temperature. Then, microhardness and microstructural evaluations were carried out in every step in order to study the effects of rolling time and temperature on martensite formation and carbides precipitation by the facile and cost-effective method of cold-rolling in comparison with other grain-refinement techniques.

2. Materials and Methods

In this study, A316 austenite stainless steel with the chemical composition presented in Table 1 was used. Sample dimensions were 100×30×8 mm and they were homogenized at 1150 ºC for 20 hours prior to use. In order to obtain ultrafine-grained microstructure, advanced thermo-mechanical treatment including cold rolling and annealing were employed with emphasis on the influence of cold-rolling temperature on the microstructure. The processes are described below:

At first, cold rolling was conducted on the samples at three different temperatures: liquid nitrogen (-196 ºC), a mixture of dry ice and ethanol (-20 ºC) and the ambient temperature. Each sample experienced a reduction of thickness up to 90%. In other words, the sample thickness reached about 0.8 mm from 8 mm. Fig. 1 shows a schematic of the performed processes applied on the stainless steel. Then, annealing was performed at the temperature of 800 ºC for 1, 5, 10 and 15 minutes. Hardness was measured by microhardness (Micro

Table 1. Chemical composition of 316 Austenitic stainless steel.

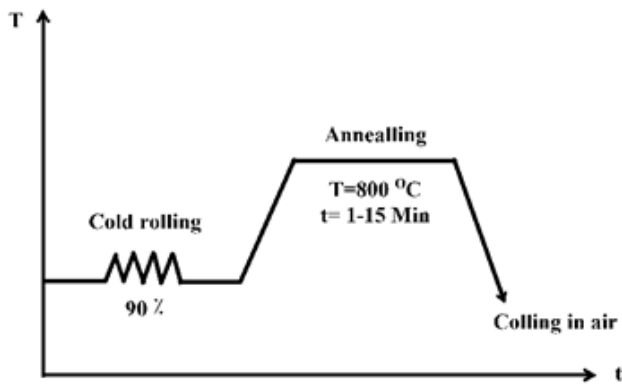| Element	%Fe	%C	%Cr	%Ni	%Mo	%Mn	%Si	%Cu
wt.%	67.9	0.0185	16.3	11.2	2.03	1.34	0.313	0.300

Fig. 1. Thermo-mechanical treatment cycle applied for grain refinement of A316 stainless steel.
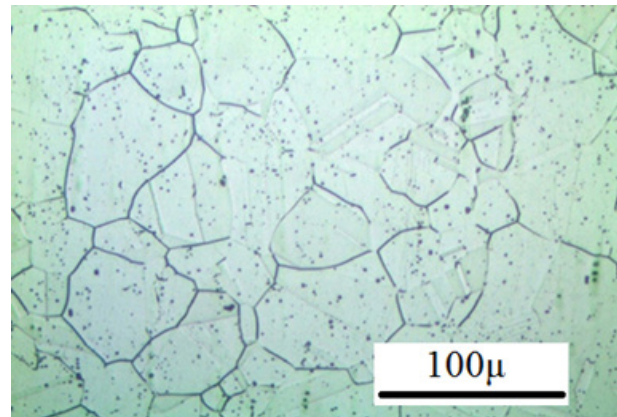

Fig. 2. Optical micrograph of A316 stainless steel base metal.

Vickers model Leitz, England) in all the stages of rolling and annealing at 300 g force at an interval of 15 seconds. Furthermore, the tensile tests were conducted on the base metal and the annealed samples in accordance with ASTM E8-M standard 21) by tensile test machine (INSTRON model H50KS, England). Austenite and martensite phase analysis was done by two methods of X-ray diffraction (XRD) and ferritescopy. To study and determination of different crystalline phases, XRD (PHILIPS model PW3040, Netherlands) was used at CuKα radiation (λ=0.1542 nm) in the range of 2θ=10-100º. For determination of the martensite and austenite volume fraction ferritescope (Fischer model FMP 30, Germany) was utilized at temperatures of 750 and 850 ºC.

Therefore, specimens with proper sizes were prepared by grinding using 80–3000 silicon carbide paper grits, followed by mechanical final polishing using alumina powder. The Surface preparation contributions to the production of martensite on the surface which this martensite phase was created at the early stages of preparation by applying rough grinding. Samples were electro-etched with 60% nitric acid solution at a voltage of 5.1 volts for 90 seconds and then were evaluated by optical microscopy (OM) and SEM (LEO model 435VP, Germany).

3. Results and Discussion
3. 1. Microstructural observations

The microstructure of the A316 stainless steel base metal in rolling direction which contains coaxial austenitic grains is presented in Fig. 2. In this case, the grain size was 36 µm. Fig. 3 shows the microstructure of the annealed samples at the temperature of 800 ºC for 1 and 10 minute(s). Figure 3(a) represents the microstructure of the annealed samples after 1 minute. The results showed that after 1 minute the austenitic grains nucleate in the cold-worked structure while the effects of the cold-work in the microstructure of the samples were still evident.
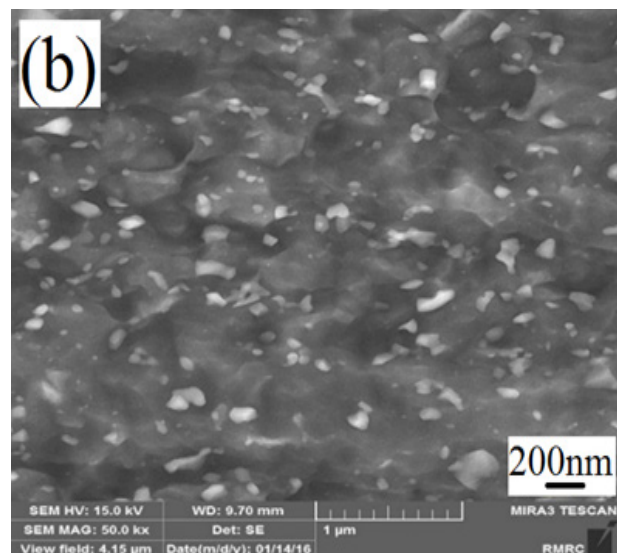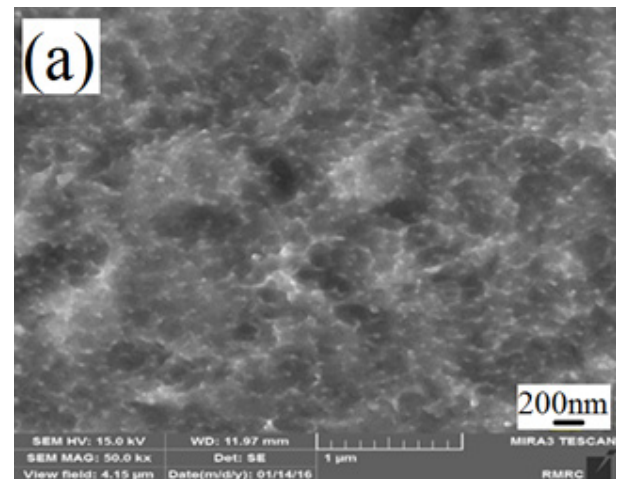

Fig. 3. SEM micrograph of the samples which were cold-rolled, annealed at 800 ºC for: (a) 1 minute, (b) 10 minutes and cooled in air (rolling direction is left to right)

direction and only a few of them had coaxial nucleation. Therefore, the austenite to martensite transformation was not completed and there was so much martensite existed in the microstructure. Annealing at 800 ºC for 1 minute rendered a grain size of about 200 nm. Due to the very low amount of carbon, very few carbide particles and late formation of the carbides, these nuclei grew rapidly. Fig. 3(b) represents the microstructure of the annealed samples after a 10-minute interval. As it is evident, by increasing the annealing time the ultrafine coaxial austenite grains nucleated and the average size of the grains was about 200 nm but more carbides formed in comparison with the 1-minute annealed sample.
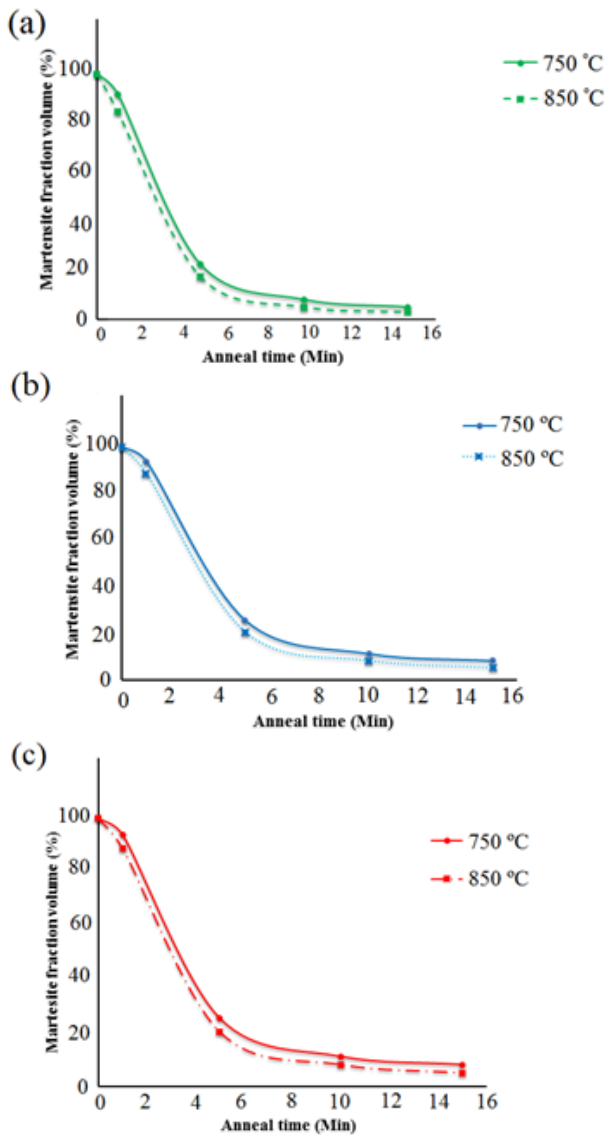
3. 2. Ferritescopy result

Fig. 4 shows the martensite variations versus annealing time for 90% cold-rolled samples at 750 ºC and 850 ºC temperatures. 800 ± 50 ºC temperature range was selected in order to precisely observe the martensite variation around 800 ºC. It can be observed that the slope of Fig. 4b and 4c curves are slightly lower than Fig. 4a, even though in all three conditions there is still residual martensite in the structure after 15 minutes. This is because of low kinetic of martensite to austenite transformation at low temperatures. In addition, it is possible for the chromium carbides to precipitate. This phenomenon might lead to the formation of martensite during cooling. It was shown that the martensite value was higher than returned process of martensite. Figure 4 shows that annealing for 15 minutes at 850 ºC reduced martensite. Resilience rate increased at this temperature, but an increase in carbide precipitation was also expected.

As a result, by an increase in the annealing time when the resilience occurrs, decarburization occurs more quickly than it did at lower temperatures. Martensite resilience is the dominant process until 15 minutes, after that martensite formation become dominant during cooling. As shown in Fig. 4(c), the sample which was rolled in liquid nitrogen had a lower slope than the sample which was rolled at the ambient temperature. In this situation it is expected that by increasing the annealing time and temperature, martensite fraction reduces. In other words, by increasing the annealing time and temperature, reduction of martensite fraction for the ambient sample is more than that of dry ice and ethanol mixture; and for dry ice, it is more than liquid nitrogen. Also, the highest martensite resilience fraction takes place at 850 ºC.

Fig. 5 shows the martensite volume fraction vs annealing time measured by ferritescopy and XRD methods. A great compromise was observed in both measuring methods at all annealing times. Using four peaks, Dickson [22] calculated the austenite and martensite

Fig. 4. Martensite variations vs. annealing time of 90% cold-rolled in:
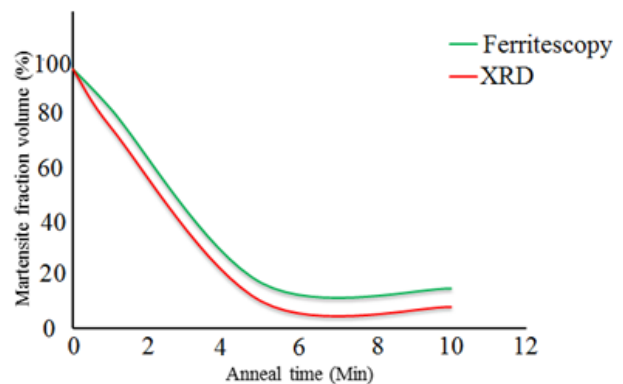(a) air (ambient temperature), (b) dry ice and ethanol mixture (-20 ºC), (c) liquid nitrogen (-196 ºC).

Fig. 5. Comparison of the two methods used for the calculation of martensite fraction.

phases to reduce the errors and remove the effects of preferred orientation. Latanision et al. 23) calculated the martensite percent by XRD and other methods, and observed a good consistency. In this study, three peaks were used to calculate the phase amount. As it can be seen, there is a little difference between the methods of measuring martensite but in high martensite fractions, an increased difference at the saturated strain was obtained by the XRD which was a bit higher than ferritescopy. This is because of using conversion coefficient for ferritescopy to converse ferrite to martensite that caused a slight difference in high amounts. Hausild et al. 24) also reported a good consistency between XRD and magnetic measurements.

Fig. 6. Vickers microhardness versus annealing time.

3. 3. Microhardness evaluations

To investigate the effect of annealing time on microhardness of the samples, all of the three samples were annealed at the temperature of 800 ºC and in four different annealing times of 1, 5, 10 and 15 minutes. The microhardness measurement results of the samples are presented in Fig. 6. As it can be seen in the figure, for the annealing time of one minute, the microhardness of the samples increased which was attributed to the deposition and formation of Cr_7C_3 carbides. Furthermore, by increasing the annealing time, the microhardness of the samples decreased. In other words, within the time interval of 5 to 15 minutes, the microhardness of the samples decreased. In this time range, the microhardness of samples decreased due to the dissolution of carbides, transformation of Cr_7C_3 to $Cr_{23}C_6$ and grain coarsening. As it is evident in Fig. 6, three slope zones are visible for all of the three samples. In the first zone the slope is positive and the hardness is increasing. In this case it seems that the austenite resilience rate is the dominant mechanism. In the second zone, the slope is reversed and the hardness decreases severely which might be due to the notion that
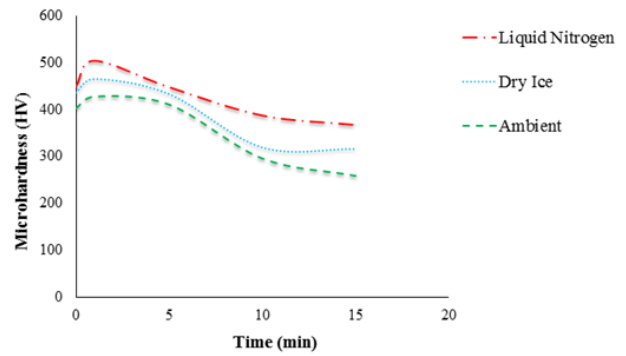
the grain growth rate mechanism is dominant. Third zone has a different situation from the two previous zones, the curve slope is zero, the hardness remains constant and the grains growth and austenite resilience rate are equal. In other words, along with coarse grains and its effect on the hardness, the new austenite grains that have high dislocation density are crystallized and compensated the hardness reduction.

3. 4. XRD results

The XRD patterns of untreated AISI A316 stainless steel (base metal) is shown in Fig. 7. As it can be observed in the figure, the only visible phase in the microstructure is austenite. Fig. 8 shows that, the intensity of the diffraction peaks of austenite has significantly decreased and instead the intensity of the martensite diffraction peaks increased significantly during the rolling process. Reducing the cold-rolling temperature has a great impact on the intensity of the diffraction peaks of the martensite as a result of increasing the martensite phase in the sample. Fig. 9 shows the XRD patterns of the samples during annealing process at a temperature of
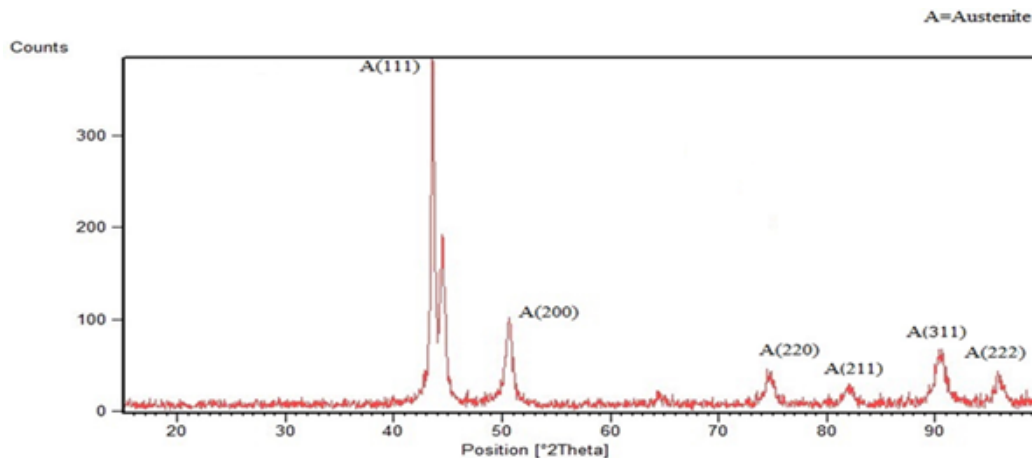

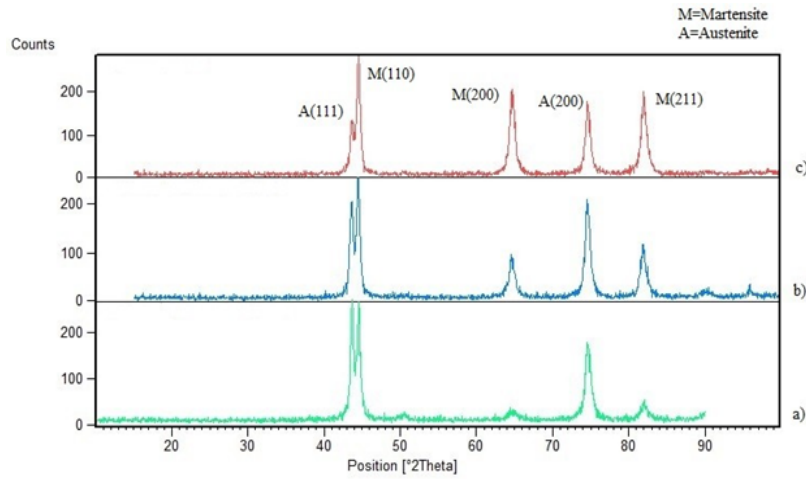Fig. 7. X-ray diffraction pattern of the base metal (untreated sample).

Fig. 8. X-ray diffraction patterns after rolling (a) at the ambient temperature, (b) in a mixture of dry ice and ethanol and (c) in liquid nitrogen.
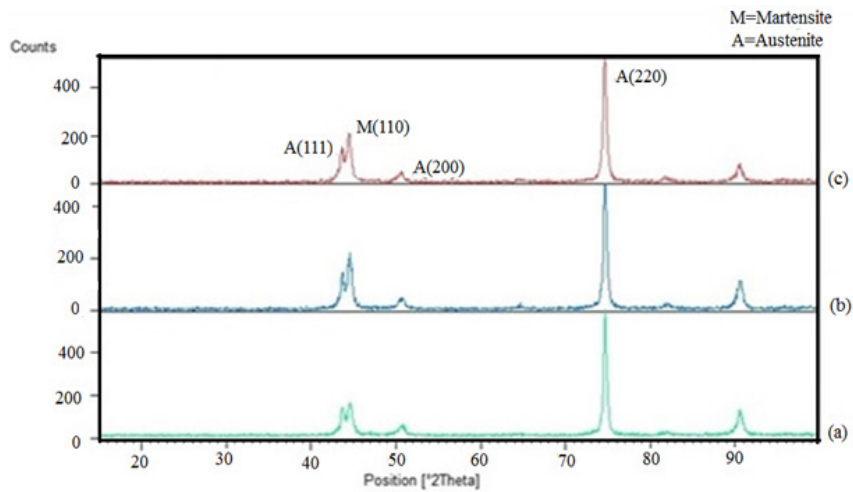

Fig. 9. X-ray diffraction patterns of samples which were annealed at 800 °C for 1 minute after rolling: (a) at the ambient temperature, (b) in mixture of dry ice and ethanol and (c) in liquid nitrogen.
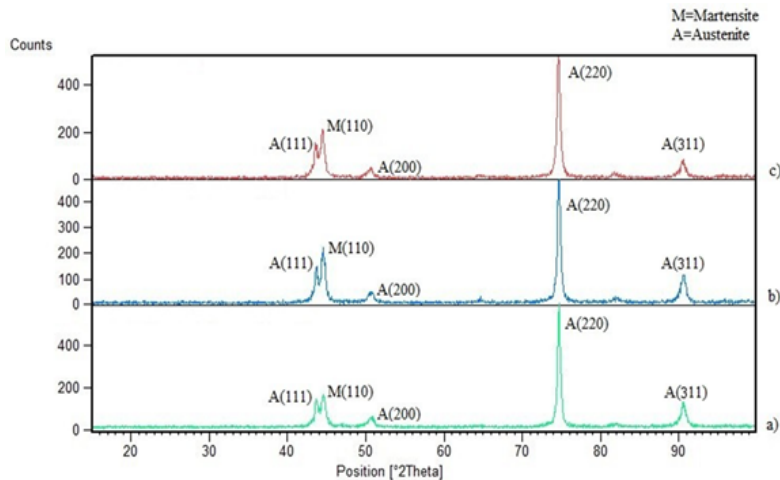

Fig. 10. X-ray diffraction patterns of samples which were annealed at 800 °C for 10 minutes after rolling: (a) at the ambient temperature, (b) in dry ice and ethanol mixture and (c) in liquid nitrogen.

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800 ºC for 1 minute. As it is shown, the intensity of the austenite diffraction peaks increased significantly and the martensite diffraction peaks substantially reduced or are eliminated. In other terms, it can be said that the desired stable austenite phase has increased during the annealing treatment. The XRD pattern of the 90% cold-rolled samples which were annealed at 800 ºC for 10 minutes are illustrated in Fig. 10. As shown, there are no martensite in the structure after 10 minutes which caused stress relieving in austenite/martensite interface after a short time of annealing. It can probably be carbide precipitation which causes the decarburization and locally increases the Ms temperature, which results in the formation of martensite after annealing and cooling.

3. 5. Effect of Temperature on the Strain-induced Martensitic Transformation

The martensite fraction volume versus reduction percent diagram was drawn at the ambient tempture, mixed dry ice and ethanol, and liquid nitrogen during rolling (Fig. 11). The saturation strain is decreased by decreasing the cold-rolling temperature. The motive force and the required energy for the martensite formation increased by decreasing the temperature. It means that its reduction might be due to the contribution of mechanical work of martensite formation. Therefore, decreasing temperature can increase the martensite percentage. Since formation of shear bands and production of martensite nucleation sites are strongly dependent on stacking fault energy (SFE), by increasing the temperature, the stacking fault energy increased as the result of the martensite

transformation induced by strain delay. Talonen et al. [25] aimed to compare different methods for measuring strain induced martensite content in austenitic steels and found that reducing the temperature had stronger effects on the transformation rate rather than reducing the SFE. Therefore, the driving force is not high at the ambient temperature. The martensite formation rate is sensitive to factors such as strain rate and austenite grain size. Sensitivity to strain rate and austenite grain size decreased by increasing driving force and decreasing temperature so that the strain rate and the austenite grain size effects can be completely ignored. This is because almost all the structure is talented to martensite germinate by sufficient driving force.

3. 6. Mechanical test results

Fig. 12 shows the engineering stress-strain curves obtained in this research before thermo-mechanical treatment and after 90% cold rolling in three different temperatures and annealing treatment at 800 ºC for 10 minutes in rolling direction. In Table 2 it can be observed that after the thermo-mechanical treatment in optimum conditions, the yield strength of the steel increased about 2 times in liquid nitrogen. Also, it increased about 148 MPa and 117 MPa in a mixture of dry ice and ethanol and at the ambient temperature, respectively. Finally, an increase in the tensile strength of steel is about 410 MPa in liquid nitrogen and about 290 MPa and 385 MPa in a mixture of dry ice and ethanol and ambient temperature, respectively.
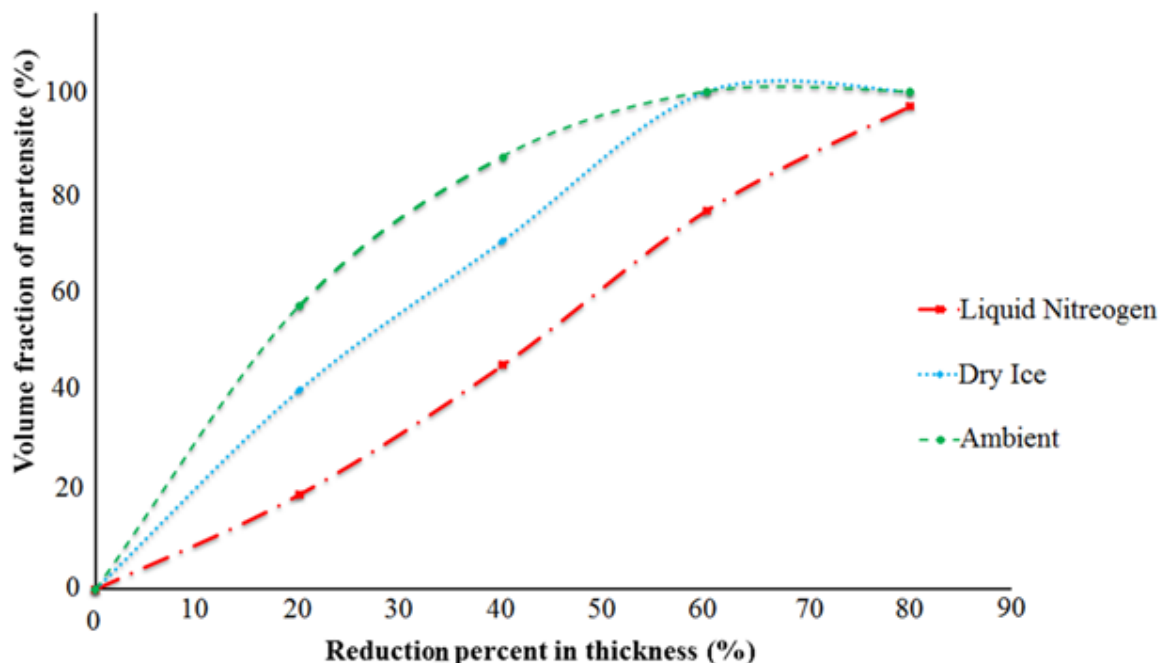

Fig. 11. The effect of cold rolling temperature on the martensite formation and thickness reduction.
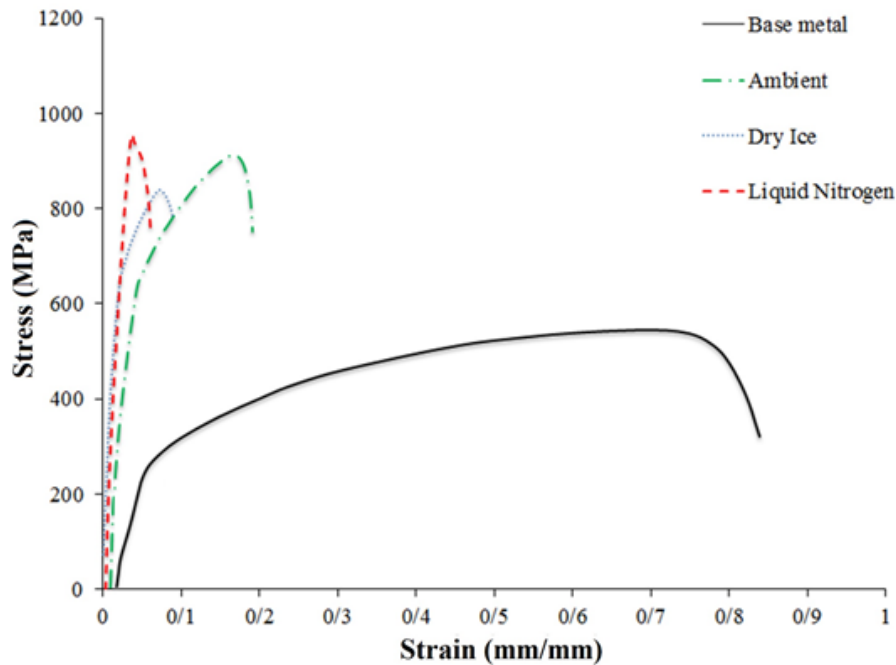
Fig. 12. Engineering stress-strain curve of base metal (untreated sample).

Table 2. Tensile properties of steel samples before and after thermo-mechanical treatment.

Materials	Yield strength (MPa)	Ultimate tensile strength(MPa)	Elongation(%)
Base metal (untreated)	270	540	87.5
Rolled at ambient temperature	387	925	20.88
Rolled at -20 °C	418	830	9.68
Rolled at -196 °C	630	950	6.25

4. Conclusions

In current paper, the effects of rolling temperature and annealing heat treatment time on the AISI 316 stainless steel were investigated and the following conclusions have been obtained:
• The microhardness of the samples increased significantly with decreasing the rolling temperature, owing to this fact, the microhardness of the sample which was rolled in liquid nitrogen was higher than the sample rolled at ambient temperature.
• Lower annealing times led to the precipitation and formation of Cr_7C_3 carbides which finally caused an increase in the microhardness. However, increasing the annealing time resulted in grain coarsening.
• The intensity of the X-ray diffraction peak of martensite increased by decreasing the rolling temperature. Also, with an increase in the annealing time, the diffraction peaks of martensite significantly decreased and the diffraction peaks of austenite dramatically increased.

• The saturation strain decreased by decreasing the cold-rolling temperature, because the motive force and the required energy to form martensite increased by decreasing the temperature.
• By applying thermo-mechanical treatment in optimum conditions, the yield strength of the steel increased about 2 times by cold working in liquid nitrogen.

References

[1] V. F. Zackay, E. R. Parker, D. Fahr, R. Busch: Trans. Am. Soc. Met., 60(1967), 252.
[2] M. F. McGuire: Stainless Steels for Design Engineers, Materials Park, Ohio: ASM International, (2008).
[3] G. E. Totten: Steel Heat Treatment, Taylor and Francis Group, Portland, Oregon, U.S.A, (2007).
[4] M. E. Bajgholi, R. Soltani Tashi, A. A. Akbari Mousavi, E. Heshmat Dehkordi: Adv. Mater. Processes., 1(2013), 47.
[5] J. C. Lippold, D. J. Kotecki: Welding Metallurgy and Weldability of Stainless Steels, New Jersey, Wiley Inter-

science, (2005).

[6] J. W. Elmer, S. M. Allen, T. W. Eager: Metall. Mater. Trans. A., 20A(1988), 1989.

[7] J. Talonen, H. Hänninen: Metall. Mater. Trans. A., 35(2004), 2401.

[8] J. W. Fu, , Y. S. Yang, , J. J. Guo, , J. C. Ma, W. H. Tong: J. Cryst. Growth., 311(2008), 132.

[9] K. H. Lo, C. H. Shek, J. K. L. Lai: Mater. Sci. Eng., 65(2009), 39.

[10] D. Fabr, Metall. Mater. Trans. A., 2(1971), 1883.

[11] J. Speer, D. Matlock, D. C. Murdock: Metall. Mater. Trans. A., 37(2006), 1875.

[12] K. Tomimura, S. Tukaki, S. Tuminutu, Y. Tokunaga: ISIJ Int., 31(1991), 721.

[13] S. Takaki, K. Tomimura, S. Ueda: ISIJ Int., 34(1994), 522.

[14] R. Misra, B. R. Kumar, M. Somani, P. Karjalainen: Scripta Mater., 59(2008), 79.

[15] N. Tsuji: Production of Bulk Nono-structured Metals by Accumulative Roll Bonding (ARB), Nova Science Pub-

lisher, Chapter 7.4, (2005), 543-564.

[16] R. Song, D. Ponge, D. Raabe, J. G. Speer, D. K. Matlock: Mater. Sci. Eng. A., 441(2006), 1.

[17] A. D. Schino, I.Salvatori, J. Kenny: J. Mater. Sci., 37(2002), 4561.

[18] B. R. Kumar, S. Das, S. Sharma, J. Sahu: Mater. Sci. Eng. A., 527(2010), 875.

[19] A. Momeni, S. Abbasi: J. Mater. Sci. Techn., 27(2011), 338.

[20] B. R. Kumar, S. Sharma, B. Mahato: Mater. Sci. Eng. A., 528(2011), 2209.

[21] ASTM E8 Standard Test Methods for Tension Testing of Metallic Materials.

[22] M. J. Dickson: J. Appl. Crystallogr., 2(1969), 176.

[23] R. M. Latanision, A. W. Jr Ruff: Metall. Mater. Trans. A., 2(1970), 505.

[24] P. Haušild, V. Davydov, J. Drahokoupil, M. Landa, P. Pilvin: Mater. Des., 31(2010), 1821.

[25] J. Talonen, P. Aspegren, H.Hänninen: Mater. Sci. Techn., 20(2004), 1506.