Influent of soaking time in liquid nitrogen on microstructure and mechanical properties of AISI 1018 low carbon steel processed by cryorolling

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Abstract. Cryorolling was identified as a new potential route to fabricate ultrafine grained of AISI 1018 low carbon steel plate with less plastic deformation. This work aims to study the influence of soaking time in liquid nitrogen on microstructure and mechanical properties of AISI 1018 carbon steel. AISI 1018 low carbon steel was processed by soaking in liquid nitrogen at various soaking times (5, 10, 20, 30 minutes) and then followed by rolling up to 50% thickness reduction. Microstructure and mechanical properties were investigated using an optical microscope, Vickers microhardness, and tensile test. The hardness and strength were increased up to 10 mins of soaking time and then gradually dropped as the soaking time increases. The highest tensile strength and hardness were 501.75 MPa and 176.04 HV achieved at 10 minutes soaking time.

1. Introduction

Nowadays, low carbon steel has been one of the most used metallic materials in the automotive, railway and construction due to the combination of properties in strength, ductility, good formability and low cost [1, 2]. Although, it has a good mechanical property as mentioned above, its strength is not enough for some engineering application uses. Hence, the improvement in mechanical properties is required through microstructural refinement. Microstructural refinement of steel is usually achieved by alloying and/or thermomechanical treatments accompanies by various types of phase transformation. Recently, severe plastic deformation (SPD) techniques provide another efficient access for grain refinement of metals and alloys. The fabrication of bulk materials with ultrafine grain sizes has attracted a great attention over the past two decades because of the materials’ enhanced properties [3].
The common SPD processes used for bulk materials include such as ARB (Accumulative Roll Bonding), ECAP (Equal Channel Angular Pressing), and simple shear processes (torsion) [4]. However, the current SPD processes may be difficult to apply to practical manufacturing, because of their complicated procedures and/or inapplicability to bulky workpieces. Moreover, the current SPD processes required high strain to achieve ultrafine grained (UFG). To overcome this drawback, cryorolling process was developed to achieve ultrafine grained. Cryorolling is a simple low-processing route which requires a relatively lower load to induce severe strain for producing the ultrafine grain structural features in materials [5]. The cryorolling process involved by soaking the sample in the liquid nitrogen prior to rolling process. During soaking in liquids nitrogen, the material cools, atomic contraction of matrix occurred due to the extremely cold temperature and released great compressive deformation energy that served as the driving force for the enhancement and movement of dislocations during cryorolling. However, for low carbon steel, the extensive research on development of ultrafine grained was investigated using severe plastic deformation (SPD) [6,7] and no work was reported on cryorolling. Therefore, this study was initiated to evaluate the influence of soaking time in liquid nitrogen on the microstructure and mechanical properties of AISI 1018 low carbon steel prior rolling process.

2. Experimental work

Plates of commercial plain AISI 1018 low carbon steel (Fe-0.06wt.% C, 0.14wt.% Mn, 0.01wt.% P, 0.01wt.%) was used. The steel plate was 20 mm in width, 100 mm in length and 5 mm in thickness. The plates were austenitized at 900°C for 1 h then followed by cryorolling. Cryorolling was performed by soaking the plates in liquid nitrogen for 5, 10, 20, and 30 min before each rolling process. The plates then were rolled by 50% thickness reduction (equivalent strain, \( \varepsilon = 0.75 \)). In each rolling pass, the thickness of the samples was reduced by approximately 15%.

The microstructure was observed under optical microscope (OM) attached with image analyzer on the reduction cross section side. Before observed under OM, the samples were prepared by ground with SiC paper up to 2000 grit then followed by diamond suspension polishing of 9 μm to 1 μm. The samples then were etched in 2% nital for 25 seconds. The length and width of the grains were measured and the grain aspect ratio can be achieved by calculating the ratio of the length, \( l \) to the side width, \( w \). The hardness of sample was performed using Microhardness Vickers at ten different location at cross section with load of 300 g for 10 s dwell time. Tensile test was conducted at room temperature with crosshead velocity of 1 mm/min using Instron 5982 digital control testing machine. The strength value was an average from 3 individual tests. The crystallite sizes and lattice strain of the samples were calculated using Scherrer equation as shown in Equation 1 and 2 [8].

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\text{Crystallite size (nm), } \tau = \frac{k\lambda}{\beta \cos \theta} \tag{1}
\]

\[
\text{Lattice strain, } \eta = \frac{\beta}{4 \tan \theta} \tag{2}
\]

3. Results and discussion

3.1 Microstructure

Microstructure of as-received material contains ferrite and pearlite phase. The grains are in the regular shape with the average size of 11.42 μm (ASTM E1382) as shown in Figure 1a. The grain increases after underwent annealing at 900 °C with average size of 83.48 μm as shown in Figure 1b. The grain increases due to annealing temperature fall in the grain growth region. Figure 2 shows the microstructure of the samples after cryorolled at various soaking times in liquid nitrogen. The cryorolled samples exhibited deformed grains which are elongated along the rolling direction at all the soaking times.
The grain aspect ratio increases as soaking time increases up to 10 mins and starts to decrease beyond 20 mins as indicated in Table 1. The optimum soaking time maintain the samples in the cryogenic temperature, ensuring the contraction occurred within structural [9-11]. The contraction enables a greater generation of compression stress and deformation energy in the microstructure. The deformation energy would serve as the driving force for the enhancement and movement of dislocations and increased the deformation rate of the samples. From Table 1, sample soaked at 10 mins showed the highest grain aspect ratio compared to other samples. Thus, sample soaked at 10 mins in liquid has the highest deformation rate during the cryorolling process. Thus, sample with higher soaking time causes higher deformation compared to lower soaking time. However, as the soaking time increases up to 20 mins, the sample might have over shrinkage and the volume of deformation energy might not enough to achieve high deformation rate and not much changes observed in the grain aspect ratio of AISI 1018 low carbon steel.

![Figure 1](image1.png)

**Figure 1.** Microstructure of low carbon steel AISI 1018. (a) as-received and (b) annealing.

![Figure 2](image2.png)

**Figure 2.** Microstructure of cryorolled low carbon steel AISI 1018 at various soaking times. (a) 5, (b) 10, (c) 20, and (d) 30 minutes.
Table 1. The average grain aspect ratio at various soaking time

| Soaking time (min) | Grain Aspect Ratio |
|-------------------|-------------------|
| 5                 | 4.53 ± 1.47       |
| 10                | 5.27 ± 1.94       |
| 20                | 4.33 ± 0.89       |
| 30                | 4.13 ± 1.97       |

3.2 Crystallite Size and Lattice Strain

The variations of lattice strain ($\langle \varepsilon^2 \rangle^{1/2}$) and crystallite size against soaking time were determined qualitatively from the XRD peak broadening using Scherrer equations and is shown in Table 2. The result showed that the lattice strain for all the samples slightly increased meanwhile the crystallite size decreased as the soaking time was prolonged to 10 minutes. As reported by [12], the cryorolled Al–Cu alloy samples exhibited reduced crystallite size along with enhanced lattice strain and dislocation density compared to room temperature rolled samples. The factors that contribute to the high value of lattice strain are due to the high amount of large deformation strain achieved and the maximum contraction of Feum matrix. The soaking time of 10 minutes able to introduce contraction in Fe matrix which suppressed the atoms inside the sample.

Table 2. Variation of crystallite size and lattice strain of cryorolled AISI 1018 at different soaking times

| Soaking time (min) | Crystallite size (nm) | Lattice strain  |
|--------------------|-----------------------|-----------------|
| 5                  | 42.1                  | 6.14 x 10^{-4}  |
| 10                 | 30.9                  | 9.67 x 10^{-4}  |
| 20                 | 50.9                  | 5.34 x 10^{-4}  |
| 30                 | 34.0                  | 8.56 x 10^{-4}  |

3.3 Mechanical Properties

The hardness and tensile strength of as-received material and cryorolled samples at different dipping time is presented in Figure 3. The as-received material recorded hardness, yield strength and tensile strength of 132.27 HV and 380.43 MPa. The highest hardness (176.04 HV) achieved for sample soaked at 10 mins with increment of 33.09% compared to as-received. The hardness increases as the soaking time increases and then dropped beyond 20 mins. The yield strength and the tensile strength also displayed the same trend as hardness. The highest tensile strength was 501.75 MPa at 10 minutes soaking time. The increase in both hardness and strength are due to increment of plastic deformation as can be observed in high grain aspect ratio.

The cryogenic temperature enables the suppression of dynamic recovery. The suppression of dynamic recovery retains the high strain (high dislocation density) in the sample and thus increased the hardness and strength of the samples. The high dislocation density impedes the propagation of dislocation in the slip system which lead to increase in hardness and strength.
Figure 3. Strength and hardness of cryorolled low carbon AISI 1018 at various soaking times

4. Conclusion

The variation of soaking times affect the microstructure, hardness and strength of cryorolled AISI 1018 low carbon steel. The microstructure showed the highest elongated grain at 10 minutes soaking times with the highest grain aspect ratio of 3.16 µm. The hardness and tensile strength of low carbon steel showed improvements of 33.09% and 31.89% at 10 minutes soaking time compared to as-received. Thus, the suitable soaking time in liquid nitrogen prior rolling for AISI 1018 prior is 10 minutes due to the excellent mechanical properties.

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References

[1] Phoumiphon N, Radzali O, and Ismail A B 2015 Procedia Chemistry 19 822.
[2] Meng Q, Li J, and Zheng H 2014 journal of Materials & Design 58 194.
[3] Zrnik J, Dobatkin S V, Raab G, Fujda M and Kraus L 2010 Revista Matéria 15 240.
[4] Zrnika J, Dobatkin S V and Ondrej S 2008 Metal 13 1.
[5] Yuan C, Wang Y, Sang D, Li Y, Jing L, Fu R and Zhang X 2015 Journal of Alloys and Compounds, 619 513.
[6] Shin D H, Kim B C, Kim Y S and Park K T 2000 Acta Materialia 48 2247.
[7] Park K T, Kim Y S and Shin D H 2001 Metallurgical and Materials Transactions A: Physical Metallurgy and Materials Science 32 2373.
[8] Mahato DN 2009 State University of New York at Albany.
[9] Shi J T, Hou L G, Zuo J R, Zhuang L Z, and Zhang J S 2017 Journal of Minerals, Metallurgy and Materials 24 638.
[10] Siti A Z, Zuhailawati H, and Anasyida A S 2007 AIP Conference Proceedings 1865.
[11] Anas N M, Quah W L, Zuhailawati H and Anasyida A S 2015 Procedia Chemistry 19 241.
[12] Krishna N, Tejas R, Sivaprasad K and Venkateswarlu K 2013 Materials and Design 52 785.