Metalurgi (2022) 1: 15 - 20

METALURGI
Majalah Ilmu dan Teknologi
p-ISSN 0126-3188 | e-ISSN 2443-3926
ejurnalmaterialmetalurgi.lipi.go.id

GRAIN GROWTH KINETICS OF AUSTENITIC STAINLESS STEEL 316L AND THE RELATIONS BETWEEN GRAIN SIZES AND HARDNESS UNDER ISOTHERMAL CONDITIONS

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Received : 16-02-2022, Revised : 02-06-2022, Accepted : 23-06-2022

Abstract

The 316L austenitic stainless steel is usually used in nuclear power plant. This steel has an austenitic phase at room temperature, and it can change grain size after being exposed at high temperatures. This study aims to investigate grain growth behavior and hardness of 316L austenitic stainless steel after cold-rolled and annealing to 1100 °C with holding times of 0, 900, 1800, 2700, 3600 s. The result showed that the grain growth of 316L austenitic stainless steel usually occurs. Austenite grain size of 316L increased with increasing holding time, resulting in hardness decreases. Experimental grain growth of 316L austenitic stainless steel shows no significant difference from the prediction, with an error of about 0.7. The highest Micro Vickers hardness is found at a grain size of 14.93 µm.

Keywords: *Austenitic stainless steel 316L, grain growth kinetics, hardness, modeling*

1. INTRODUCTION

Due to its mechanical properties such as high tensile strength, good ductility, and high corrosion resistance, 316L austenitic stainless steel has many applications in industry and is commonly used as a structural material in nuclear power plants [1]. Austenite grain size and microstructure grain growth play an important role in determining mechanical properties [2]. For example, the hardness of steel decreases with increasing grain size. To increase the material's hardness, the grain boundaries must be increased to make the grains as small as possible. Grain boundaries prevents movement of dislocations due to lack of continuity of the slip plane from one grain to another, and dislocations propagating to other grains must change the direction of movement [3].

Previous studies have been conducted on grain growth in austenitic stainless steels. Järvenpaa et al., [4] found that the finer the grain size of austenitic stainless steels, the higher the yield strength. Li et al., [5] studied the effect of cold rolling on the mechanical properties of 304N stainless steel. They found that the higher the cold reduction, the more strained martensite was formed, which increased strength but decreased elongation. Bedjati et al., [6] studied the effect of annealing temperature on the grain size of austenitic stainless steel Ni-free. They used cold rolling with a high rolling reduction of about 80% and then reverse annealed at 900 °C for 100 seconds to obtain ultra-fine and nano-sized austenite grain. Nano/ultra-fine grain size provides ultra-high-strength and excellent elongation. Jiang et al., [7] investigated the

DOI : 10.14203/metalurgi.v37i1.629

microstructure and mechanical properties of high nitrogen austenitic stainless steel after aging at 900 °C for 0 to 50 hours. They stated that the presence of intergranular Cr_2N precipitation caused the Vickers hardness value to decrease and the Vickers hardness value to increase due to increased Cr_2N precipitation, the formation of cellular Cr_2N, and the intermetallic phase. Thikonova et al., [8] stated that the strength of metallic materials increases with ductility decreases. The controlled thermomechanical treatment includes post-deformation annealing resulting in a beneficial effect on the microstructure. Wang et al., [9] presented an air-coupled probe for grain size characterization of 316L stainless steel using Rayleigh wave attenuation. Matt et al., [10] stated that the Arrhenius equation could predict the grain size of austenitic stainless steels by examining the behavior of materials at high temperatures. It was argued that the appearance of abnormal grain in the high-temperature region would absorb the surrounding grain, and the sediments generated at the grain boundary could be realized through the modified Arrhenius equation.

However, to the best of the author's knowledge, studies on grain growth of 316L austenitic stainless steels due to cold rolling under low reduction have not yet been conducted. This study was designed to predict austenite grain size using the grain growth equation and the relationship between hardness and grain size.

2. Materials And Methods

In this experiment, Steel SS316L was used. Table 1 shows the chemical composition of the steel. The steel was cold-rolled with a reduction of 22%. It is then heated in a tube furnace using gaseous hydrogen at a temperature of 1100 °C and at a heating rate of 5 °C/min with holding times of 900, 1800, 2700, and 3600 s before quenching in the cooling zone.

The microstructure was observed in the steel sample using an optical microscope. The steel is polished by conventional metallographic methods and etched in aqua regia (80% HCl and 20% HNO_3) for 1 min to reveal the boundaries of the austenite grains. Austenite grain size was determined by the mean linear intercept method. A hardness test was then performed on a Vickers micromachine with a load of 0.3 N.

Tabel 1. Chemical composition of 316L ASS (wt.%)

C	Si	Mn	P	S	Ni	Cr	Mo	Fe
0.012	0.3	1.67	0.035	<0.005	9.45	17.33	2.1	Bal.

3. Results And Discussion

Figure 1 shows the microstructure of austenite grain of 316L after 22% reduction of cold rolling. This figure shows that the grain is not elongated after cold rolling, and many precipitates showed a dot in black spread out in the austenite matrix and grain boundary.

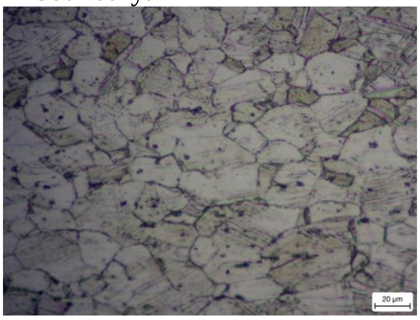


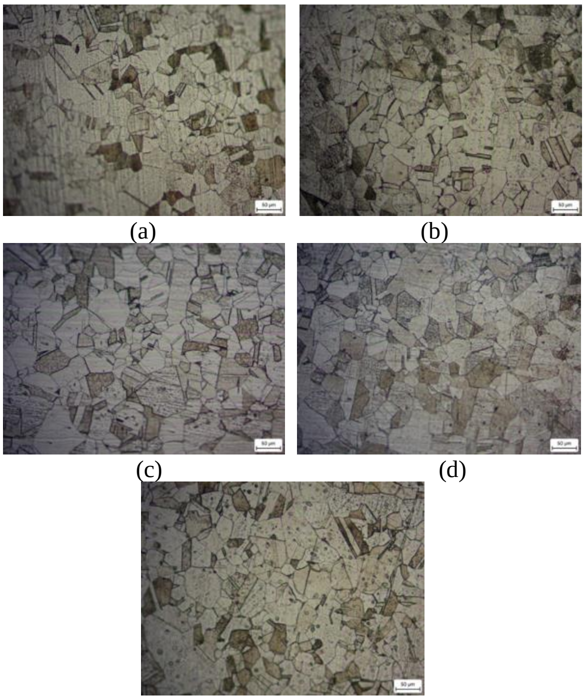
Figure 1. Microstructure of austenite grain boundaries of SS 316L after cold rolling. Aqua regia etchant

Figures 2(a)-2(e) reveal that the annealing twin formed in each condition.

Table 2. Grain size of SS 316L under different holding time at 1100 °C

Holding time (s)	0	900	1800	2700	3600
Grain size (μm)	14.93	23.56	26.18	28.97	29.28

Annealing twin occurs due to the cold-rolling process followed by heat treatment as a result of rearranging the atoms during the grain growth process [11].



(a) (b) (c) (d) (e)

Figure 2. Microstructure of austenite grain boundaries under different heat treatment conditions of SS 316L at 1100 °C with the time (s); (a) 0, (b) 900, (c) 1800, (d) 2700, (e) 3600. Aqua regia etchant

Grain sizes for all annealing conditions are given in Table 2.

The results show that SS 316L grains grow as the holding time increases. Figure 3 depicts the experimental results for changes in grain size under all annealing conditions.

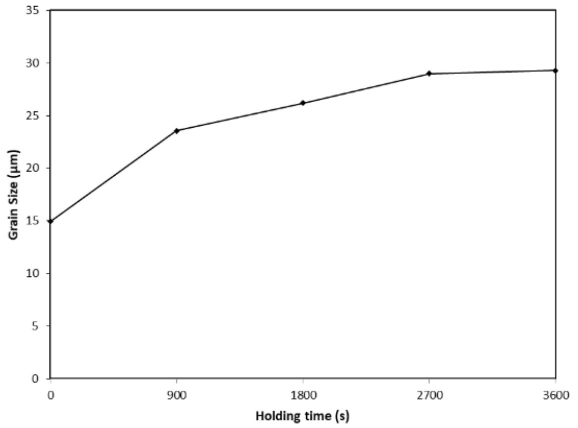


Figure 3. Austenite grain size of SS 316L under different holding times at 1100 °C

Table 3 shows experimental results on grain sizes and hardness of SS 316L annealed under different holding times at 1100 °C.

Table 3. Experimental result on grain size and hardness of SS 316L annealed under different holding time at 1100 °C

Holding time (s)	**0**	**900**	**1800**	**2700**	**3600**
Hardness micro-Vickers (HV)	149,4	148,6	148,1	147,3	146,8

Figure 4 shows that the hardness of the steel increases as the grain refinement degree increases. ($d^{-0.5}$). It is because steels with small grain sizes have more grain boundaries, inhibiting the dislocation movement. Hence, it is difficult for the dislocation to move because it needs more energy to move across the grain boundary due to differences in atomic orientation. The data were obtained by the Hall-Petch relationship, that the hardness of the annealed steel decreases with grain size increases [12].

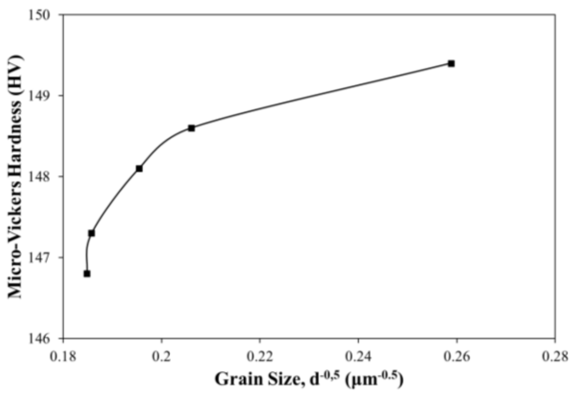


Figure 4. Relationship between grain size, $d^{-0,5}$ and hardness value in SS 316L

Figure 5 compares the hardness value of SS 316L in this study to other steels based on previous research. The hardness of most steels increases as the value of d-0.5 grain size increases. The steel type also influences the hardness value. Stainless steel 316L, for example, has a higher hardness value than FeCoNiMn but a lower hardness value than FeCoNiCrPd steel, 253 MA, FeCo-NiCrMn, and CoNiMn. It means that a high concentration of Cr, W, V, Mo, Ti, Nb, and Mn can increase steel hardness on the exact value of $d^{-0.5}$ grain size [13].

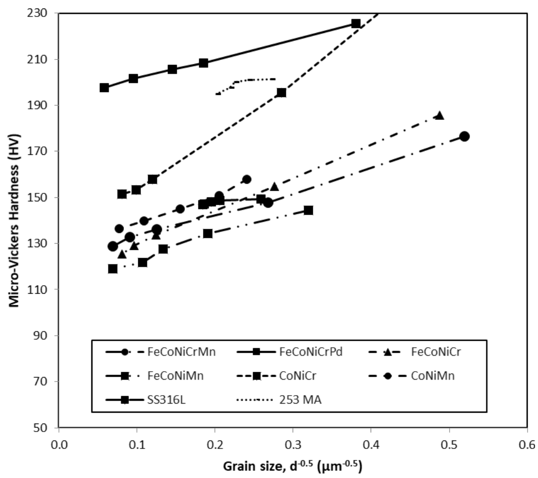


Figure 5. Hardness value of different type of steel under different annealing condition

Different pre-treatment and annealing treatments can result in an other kind of precipitate and affect the hardness of steel [12].

The average grain growth of steel is often expressed by the following empirical Equation 1 [14].

$$D^n = Kt \quad (1)$$

Where D is the average grain size at time t, t is holding time, n is the grain growth exponent, and K is the temperature dependence. The value of n depends on the grain growth mechanism [15]. Based on the basic theory of grain growth, the predicted values of n are usually greater than or equal to 2 [16]. However, previous studies indicated that the value of n for austenitic stainless steel is around 0.128 to 0.443 [17].

Equation 1 describes the nature of grain growth when D is greater than the initial grain size (D_0). However, the neglect of D_0 can affect the value of the grain growth exponent. So, the influence of D_0 on the grain growth behavior should be considered, and the equation was developed in Equation 2.

$$D^n - {D_0}^n = Kt \quad (2)$$

The relationship between the constant K and temperature follows the Arrhenius equation in Equation 3.

$$K = K_0 exp(-\frac{Q_{gg}}{RT}) \quad (3)$$

Where K_0 is the experimental parameter, Q_{gg} is the activation energy of grain growth, R is the molar gas constant of 8.314 J/molK, and T is the heating temperature.

Equations 2 and 3 can be combined to form a new equation as expressed in Equation 4.

$$D^n - D_0{}^n = K_0 exp(-\frac{Q_{gg}}{RT})t \quad (4)$$

The grain growth activation energy (Q_{gg}) of SS316L steel used in this study was data from the Barbosa study with a Qgg value of 320,000 [18].

Equation 5 was obtained by the logarithmic transformation of Equation 4.

$$\ln(D^n - D_0{}^n) = lnK_0 - \frac{Q_{gg}}{RT} + lnt \quad (5)$$

The constant values of n and K_0 are calculated with Microsoft Excel's Solver. As a result, the optimal values for n and K0 are 5.78 and 1.35×10^{17}. The value of n in this study is similar to the basic grain growth theory. The difference between the simulated value and the study result is determined by calculating the SSE (sum of squares error). The resulting SSE value is 0.7. This indicates that the simulation error value is 0.7, and the simulation is close to the study results. Then, the empirical model of steel grain growth can be expressed as Equation 6.

$$D^{5.78} - D_0{}^{5.78} = 1{,}35 \times 10^{17} exp(-\frac{320.000}{RT})t \quad (6)$$

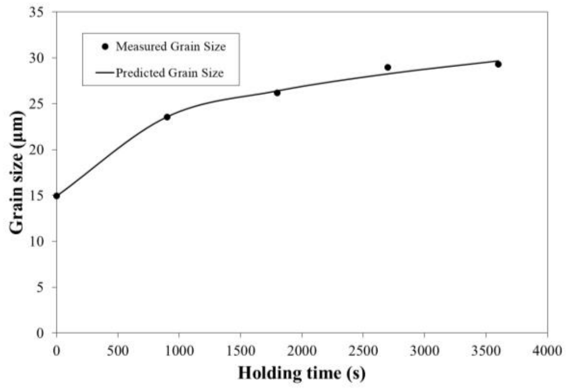


Figure 6. Comparison grain size resulting from experiment and prediction in SS 316L

Figure 6 is a comparison of the measured grain size and the predicted grain size. This figure shows that the measured grain size strongly conforms with the modeling results. The research model produces a sloping graph, and it is because the research model has a significant value of n. An important value of n indicates the resistance due to sediments that impede grain growth. As the value of n increases, the simulation graph is skewed, indicating that the grain growth rate is not constant and the grain growth rate is decreasing. The constant value of K0 depends on the composition and process of the steel [19]. Therefore, the obtained n and K0 constant values can predict grain growth of 316L austenitic stainless steel after cold rolling with a 22% reduction.

4. Conclusion

From the test results, it can be concluded that the effect of annealing holding time on the grain growth behavior and hardness of SS316L after low reduction cold rolling can be concluded: Growth of austenite grains usually occurs with longer holding times. When the annealing holding time was lengthened, the crystal grain size became slightly coarser, and the hardness decreased somewhat. The slight difference in crystal grain size and hardness at each annealing holding time is due to the slight reduction in cold rolling of 316L austenitic stainless steel. An empirical formula for predicting austenite grain growth at different retention times is obtained, and the prediction results end the experiment with an error of 0.7.

Acknowledgment

The author would like to thank the Universitas Indonesia and the National Research and Innovation Agency for their financial support. The experiment was carried out at Research Center for Metallurgy National Research and Innovation Agency.

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