

# Effect of Heat Treatment Temperature on the Microstructure and Mechanical Properties of 30% Cold-Rolled AISI 304 Austenitic Stainless Steel

Indah Uswatun Hasanah<sup>1,\*</sup>, A.Ali Alhamidi<sup>2</sup>, Agus Pramono<sup>1</sup>, and Alfirano<sup>1</sup>

<sup>1</sup>Metallurgical Engineering, Sultan Ageng Tirtayasa University, Banten, Indonesia

<sup>2</sup>Mechanical Engineering, Jendral Soedirman University, Central Java, Indonesia

**Abstract.** Stainless steel is widely utilized across various industrial applications, including as an implant material, due to its favorable biocompatibility, excellent corrosion resistance, and adequate mechanical properties. However, under physiological conditions, the toughness of stainless steel tends to decrease. To enhance its mechanical performance, cold rolling combined with subsequent heat treatment is often employed. In this study, AISI 304 austenitic stainless steel was subjected to a 30% cold rolling reduction, followed by austenitizing at different temperatures (600 °C, 700 °C, 800 °C, 900 °C, and 1000 °C) and then quenched in water to room temperature. The results showed that the highest hardness and tensile strength were achieved in the 30CR-NHT (non-heat-treated) sample, with values of 382.677 HV and 1007 MPa, respectively. The greatest toughness was observed in the 30CR-HT 1000 °C sample, reaching 62.526 J. Meanwhile, the finest average grain size was recorded in the 30CR-HT 800 °C sample, measuring 3.621 µm. These findings indicate that both mechanical strength and microstructural characteristics of stainless steel implants can be significantly influenced by the combination of cold working and controlled heat treatment parameters.

## 1 Introduction

With the rapid advancement of medical technology, the treatment of bone fractures can now be effectively addressed through implantation techniques. Biomaterials commonly used in orthopedic surgery for implants include stainless steel, commercially pure titanium, and titanium alloys [1]. Among these, stainless steel 304 is widely utilized as a bone implant material due to its advantageous properties such as corrosion resistance, non-magnetic behavior, good formability, and biocompatibility [2, 3]. However, stainless steel 304 also presents a notable limitation, particularly in its reduced toughness when exposed to biological environments [4]. To enhance the mechanical strength of austenitic stainless steel, grain refinement techniques can be applied [5]. Materials with submicron or nanostructured grains generally exhibit significantly superior properties compared to conventional materials with coarse grains [6]. Several established methods for grain size modification in austenitic stainless steels include severe plastic deformation, advanced thermo-mechanical treatment, and thermal cycling [7]. Among these, the advanced thermo-mechanical treatment, comprising conventional cold rolling followed by heat treatment, is recognized as an effective approach for producing fine-grained structures. This technique, often referred to as heavy cold rolling and annealing, has demonstrated the ability to improve ductility through phase transformation and grain refinement [8, 9]. Cold

rolling is employed to refine the grain structure and induce strain-induced martensite, thereby enhancing strength, while heat treatment is applied to restore ductility, promote martensite-to-austenite reversion, relieve residual stresses, and stabilize the austenitic microstructure. Therefore, this study aims to investigate the influence of varying annealing temperatures, following cold rolling, on the microstructural evolution, surface hardness, ductility, and toughness of stainless steel 304 plates.

## 2 Method

Stainless steel 304 samples in plate form with dimensions of 100 mm × 100 mm × 6 mm were prepared for cold rolling with a 30% reduction in thickness. Following the cold rolling process, annealing was conducted at various temperatures of 600 °C, 700 °C, 800 °C, 900 °C, and 1000 °C for 30 minutes. Microstructural analysis was performed using metallography with an etching solution composed of 5 ml HNO<sub>3</sub> and 10 ml HCl for 40 minutes. Hardness testing was carried out using the micro-Vickers hardness method with a load of 1 Kgf in accordance with ASTM E92-82. Tensile testing was conducted following the ASTM E8-15a standard, and impact testing was performed according to ASTM E23-16b. The grain boundaries were identified and analyzed in accordance

\* Corresponding author: [indah.uswatun.h@untirta.ac.id](mailto:indah.uswatun.h@untirta.ac.id)

with the ASTM E112 standard using the linear intercept method.

### 3 Results and discussion

#### 3.1 Microstructural analysis

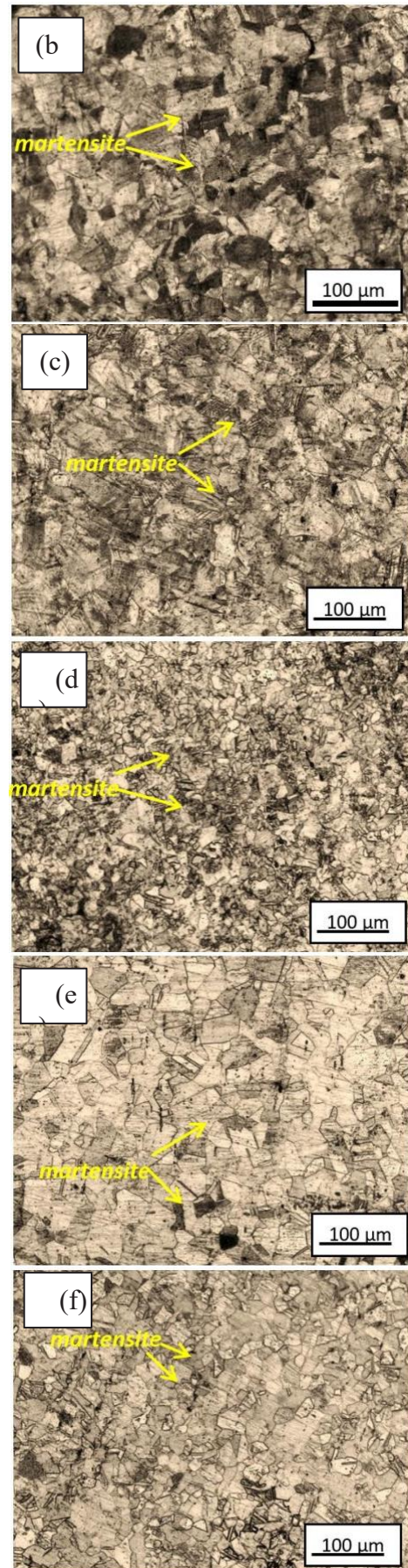
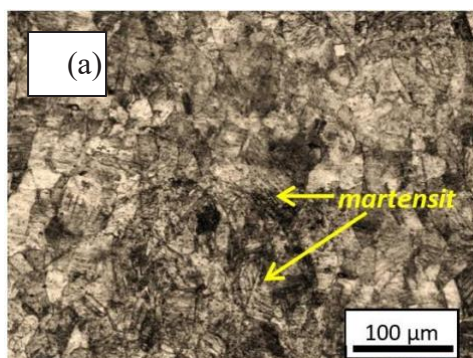
The cold-rolled stainless steel 304 sample without heat treatment Fig. 1 (a) (30CR-NHT) had an average grain size of  $3.544\ \mu\text{m}$  and a martensite phase volume fraction of 41.406%. This indicates that during the cold rolling process, grain refinement occurred due to slip mechanisms and dislocation from twinning [10]. Plastic deformation during cold rolling increases dislocation density, which in this study led to a reduction in austenite grain size, an increase in grain boundaries, and the presence of residual stress.

In Fig. 1 (b), the 30% cold-rolled sample subjected to heat treatment at  $600\ ^\circ\text{C}$  exhibited an average grain size of  $3.941\ \mu\text{m}$  and a martensite phase volume fraction of 36.116%. This suggests that during heating, the diffraction peak intensity of the martensite phase decreased, while the austenite phase began to reform. In Fig. 1 (c), the 30% cold-rolled sample heat-treated at  $700\ ^\circ\text{C}$  had an average grain size of  $4.375\ \mu\text{m}$  and a martensite volume fraction of 31.115%. This indicates a continued decline in martensite formation and the onset of new grain growth, leading to an increase in the formed austenite phase. Twinning deformation tends to increase with higher heat treatment temperatures.

In Fig. 1 (d), the sample cold-rolled by 30% and heat-treated at  $800\ ^\circ\text{C}$  showed an average grain size of  $3.621\ \mu\text{m}$  and a martensite phase volume fraction of 23.933%. The image reveals partial recrystallization of austenite, with the martensite phase and residual stress beginning to diminish.

Fig. 1 (e) and 1 (f) show samples cold-rolled by 30% and heat-treated at  $900\ ^\circ\text{C}$  and  $1000\ ^\circ\text{C}$ , respectively, with average grain sizes of  $4.162\ \mu\text{m}$  and  $4.479\ \mu\text{m}$ . The martensite phase volume fractions at these temperatures were 20.898% and 15.018%, respectively.

Based on the observations, cold rolling followed by heat treatment induces changes in microstructure. Therefore, it is feasible to achieve fine-grained structures through post-cold rolling heat treatment, as compared to the condition before cold rolling.



**Fig. 1.** Microstructure (a) 30CR-NHT, (b) 30CR HT  $600\ ^\circ\text{C}$ , (c) 30CR-HT  $700\ ^\circ\text{C}$ , (d) 30CR-HT  $800\ ^\circ\text{C}$ , (e) 30CR-HT  $900\ ^\circ\text{C}$  dan (f) 30CR-HT  $1000\ ^\circ\text{C}$

### 3.2 Hardness Test Analysis

Based on Fig. 2, the hardness value increased after the cold rolling process. This finding is in line with the statement [11], which states that as the heat treatment temperature increases, the hardness of stainless steel tends to decrease. The initial hardness value of stainless steel 304 before processing was 180 HVN. After undergoing 30% cold rolling without any heat treatment, the hardness increased significantly to 382.6767 HVN. When a metal is plastically deformed, dislocations are mobilized, resulting in an increase in dislocation density. In this study, plastic deformation led to a reduction in austenite grain size, an increase in the number of grain boundaries, and the formation of strain-induced martensite due to the 30% deformation, as well as the presence of residual stress within parts of the crystalline structure. However, after heat treatment followed by rapid cooling in water, the hardness values progressively decreased with increasing heat treatment temperatures. This reduction in hardness can be attributed to the relief of residual stress caused by the cold rolling process as the temperature increases, as well as the reduced formation of martensite due to the higher heat treatment temperatures, which promotes the reversion to the austenite phase. Moreover, as the heat treatment temperature increases, the resulting grain size becomes larger, which can lead to a reduction in grain boundary density, subsequently decreasing both the hardness and strength of the material.

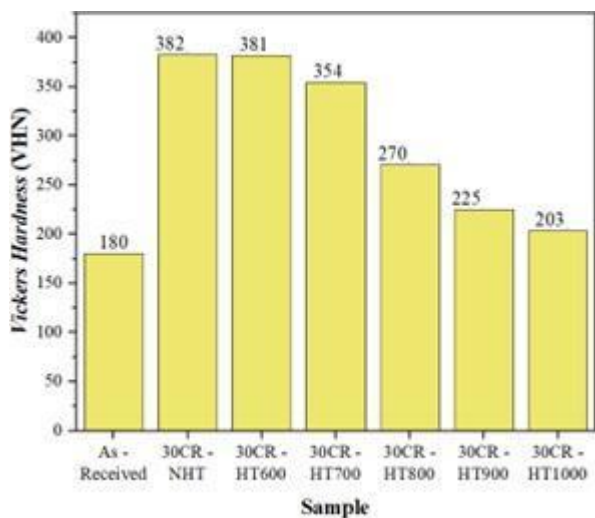


Fig. 2. Hardness Vickers

### 3.3 Tensile Test Analysis

The tensile strength as Fig. 3 increased after the cold rolling process without heat treatment, which is consistent with the literature stating that a higher cold rolling reduction percentage leads to an increase in tensile strength [12]. The as-received sample exhibited relatively low tensile strength, measured at 678 MPa, whereas the 30% cold-rolled sample without heat treatment showed a significantly higher tensile strength of 1007 MPa. This increase is attributed to the formation of martensite phase and the presence of coarse grains

resulting from the cold rolling process, which collectively enhance the tensile strength.

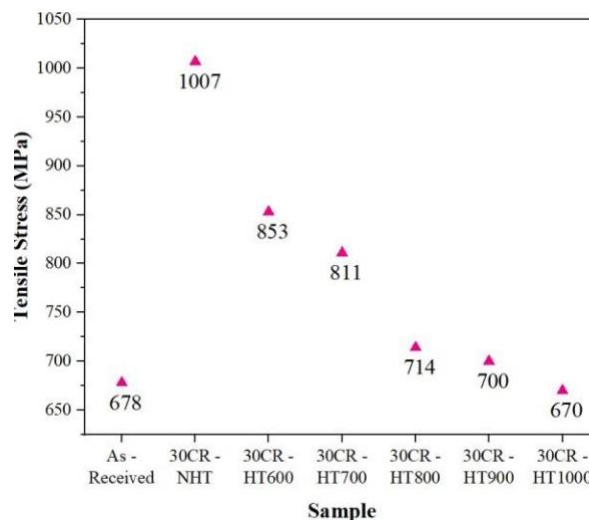


Fig. 3. Tensile Strength

However, in the 30% cold-rolled samples that underwent heat treatment followed by rapid water quenching, a decrease in tensile strength was observed with increasing heat treatment temperature. This decrease can be explained by the elimination of residual stress introduced during cold rolling, which occurs progressively with higher heat treatment temperatures. Additionally, the heat treatment process tends to lower the yield strength and tensile strength, but significantly improves the ductility compared to the samples subjected only to cold rolling [13].



Fig. 4. Fracture surface image of Tensile Test

### 3.4 Impact test analysis

The toughness value of stainless steel 304 before the experimental procedures was recorded at 50.92 J. However, after undergoing 30% cold rolling without heat treatment, the toughness decreased significantly to 21.276 J. This reduction is attributed to the deformation experienced by the metal during the cold rolling process. When the metal is plastically deformed, dislocations become mobile, leading to an increase in dislocation density. In this study, such plastic deformation resulted

in a reduction in austenite grain size, an increase in the number of grain boundaries, and the formation of strain-induced martensite, along with the presence of residual stress within parts of the crystal structure. Fig. 4 shows the fracture surface resulting from the tensile test. Following heat treatment and rapid cooling using water, the toughness of the material was observed to increase with rising annealing temperatures. This trend suggests that the martensitic phase gradually transforms back into austenite during the heat treatment process. At higher temperatures, this reverse transformation accelerates, leading to a significant reduction in the martensite phase and an improvement in impact resistance. The enhancement in toughness is primarily due to the release of residual stresses induced during the cold rolling process, which are progressively eliminated as the temperature increases. Additionally, elevated heat treatment temperatures result in grain growth, which reduces grain boundary density and consequently leads to lower hardness and tensile strength.

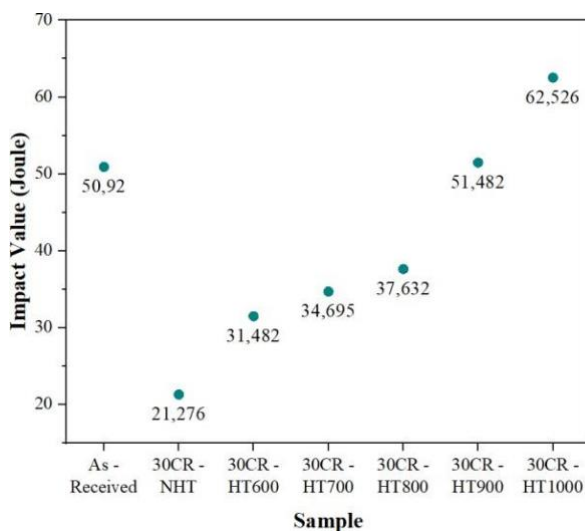


Fig. 5. Impact Value

The impact strength value tends to increase with temperature, meaning that the toughness of the material is inversely related to its tensile strength and hardness [14]. This trend is consistent with the experimental results obtained, where a decrease in tensile strength and hardness corresponded with an increase in toughness. Therefore, austenitic stainless steel shows significant sensitivity in mechanical properties, such as tensile strength, hardness, and toughness, depending on the cold rolling reduction percentage and heat treatment temperature. These effects are due to phase transformations and microstructural changes in stainless steel, and its mechanical properties can be optimized through appropriate cold rolling and heat treatment processes.

The fracture observed in Fig. 6 above is characterized as a ductile fracture, marked by significant plastic deformation prior to failure. Its primary features include the presence of dimples and a cup-and-cone morphology on the fracture surface. In ductile fracture, necking typically occurs due to the coalescence of dimples, which serve as stress concentration sites ultimately leading to fracture. Ductile fracture surfaces

may not be clearly visible at lower heat treatment temperatures; however, as the temperature increases, the size of the dimples formed also increases. With increasing deformation, the number of fine grains increases. Since dimples nucleate at grain boundaries, the formation of fine dimples is associated with the presence of fine grains. The rounded dimples represent micro-voids, which initiate the ductile fracture and gradually grow over time [15].

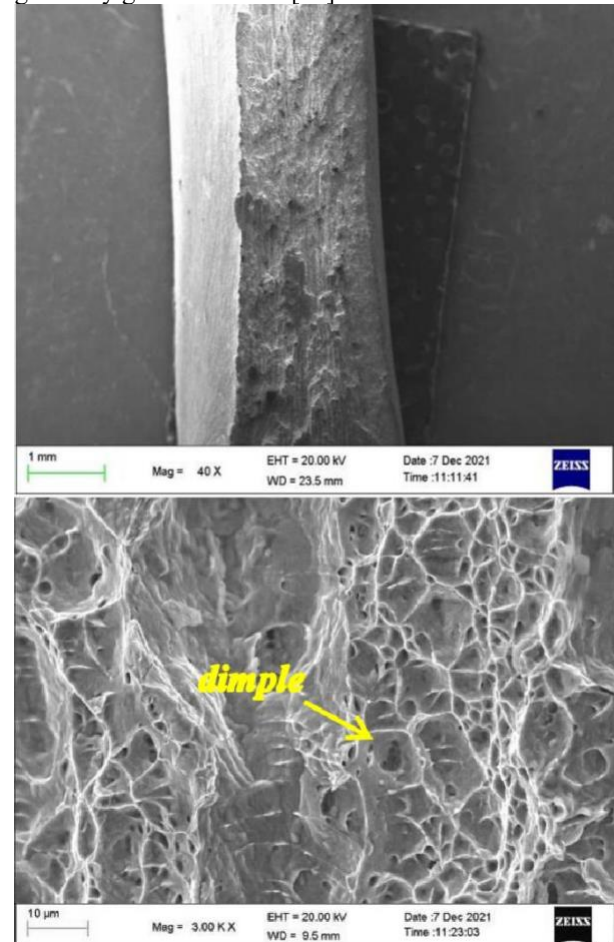


Fig. 6. Fracture surface micrograph of impact tested

## 4 Conclusion

The cold rolling process can enhance mechanical properties such as hardness and tensile strength; however, it results in a reduction of ductility and toughness. This occurs due to deformation-induced strain hardening, which is caused by grain refinement and an increase in dislocation density. When cold rolling is followed by heat treatment, mechanical properties such as hardness and tensile strength tend to decrease, while ductility and toughness improve with increasing temperature. This improvement is attributed to the release of residual stress from deformation and the growth of grain size. The highest toughness value was obtained at a temperature of 1000 °C, reaching 65.26 J.

## References

1. T.N. Wibowo, P.T. Iswanto, B.H. Priyambodo, N. Amin, Pengaruh Variasi Waktu Shot Peening

- terhadap Struktur Mikro dan Kekerasan Permukaan Pada Material Implan Aisi 304. ROTOR. 70 – 73, (2016)
2. R. Rahajeng, Analisis Kekerasan Baja Tahan Karat Aisi 304 Ternitridasi Padat Sebagai Alternatif Material Sendi Tulang Panggul Tiruan. (2020)
  3. D. Pathote, D. Jaiswal, V. Singh, C.K. Behera, Optimization of electrochemical corrosion behavior of 316L stainless steel as an effective biomaterial for orthopedic applications. *Materials today: proceedings*, **57**: p. 265 – 269, (2022)
  4. N.I.W. Lestari, Efek Variasi Energi Implantasi Ion Nitrogen Terhadap Karakteristik Stainless Steel Untuk Material Implan, Universitas Airlangga. 2017
  5. S. Mohammadzahi, S. and H. Mirzadeh, Grain refinement of austenitic stainless steels by cross rolling and annealing treatment: A review. *Journal of Ultrafine Grained and Nanostructured Materials*. **57**(2): p. 112 – 119. (2024)
  6. D.M. Xu, G.Q. Li, X.L. Wan, R.D.K. Misra, J.X. Yu, G. Xu, On the deformation mechanism of austenitic stainless steel at elevated temperatures: A critical analysis of fine-grained versus coarse-grained structure. *Materials Science and Engineering: A*. **773**: p. 138722. (2020)
  7. A. Järvenpää, M. Jaskari, A. Kisko, P. Karjalaine, Processing and properties of reversion-treated austenitic stainless steels. *Metals*. **10**(2): p. 281. (2020)
  8. M. Salehi, M. Yeganeh, R.B. Heidari, M. Eskadari, Comparison of the microstructure, corrosion resistance, and hardness of 321 and 310s austenitic stainless steels after thermo-mechanical processing. *Materials Today Communications*. **31**: p. 103638. (2022)
  9. M. Park, K. Kim, J. Jang, H.C. Kim, H.S. Moon, J.B. Jeon, D.G. am, K.Y. Sohn, B.J. Kim, Influence of heat treatment on mechanical properties for cold worked 304 austenitic stainless steel. *Korean Journal of Metals and Materials*. **56**(7): p. 490 – 498. (2018)
  10. S. Mironov, S. Zherebtsov, and S. Semiatin, The grain-refinement mechanism during heavy cold-rolling of commercial-purity titanium. *Journal of Alloys and Compounds*. **895**: p. 162689. (2022)
  11. S. Kheiri, H. Mirzadeh, and M. Naghizadeh, Tailoring the microstructure and mechanical properties of AISI 316L austenitic stainless steel via cold rolling and reversion annealing. *Materials Science and Engineering: A*. **759**: p. 90 – 96. (2019)
  12. M.T. Kim, T.M. aPark, K.H. Baik, W.S. Choi, J. Han, Effects of cold rolling reduction ratio on microstructures and tensile properties of intercritically annealed medium-Mn steels. *Materials Science and Engineering: A*. **752**: p. 43 – 54. (2019)
  13. C. Wang, P. Zhu, Y.H. Lu, T. Shoji, Effect of heat treatment temperature on microstructure and tensile properties of austenitic stainless 316L using wire and arc additive manufacturing. *Materials Science and Engineering: A*. **832**: p. 142446. (2022)
  14. Liang, X., et al., Coupled effect of temperature and impact loading on tensile strength of ultra-high performance fibre reinforced concrete. *Composite Structures*. **229**: p. 111432. (2019)
  15. Long, S.-L., et al., Study on the formation of microvoids during ductile fracture. *Materials Research Express*. **5**(1): p. 016515. (2018)