Mechanical Behaviour of 304 Austenitic Stainless Steel Processed by Room Temperature Rolling

Rahul Singh¹, Sunkulp Goel², Raviraj Verma³, R Jayaganthan⁴ and Abhishek Kumar¹*

¹Department of Applied Mechanics, Motilal Nehru National Institute of technology Allahabad, Allahabad-211004, India
²Herbert Gleiter Institute of Nanoscience, Nanjing University of Science and Technology, Nanjing-210094, China
³Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee, Roorkee-247667, India
⁴Department of Engineering Design, Indian Institute of Technology Madras, Chennai-600036, India

*Corresponding author E-mail: abhishek@mnnit.ac.in

Abstract. To study the effect of room temperature rolling on mechanical properties of 304 Austenitic Stainless Steel, the as received 304 ASS was rolled at room temperature for different percentage of plastic deformation (i.e. 30, 50, 70 and 90 %). Microstructural study, tensile and hardness tests were performed in accordance with ASTM standards to study the effect of rolling. The ultimate tensile strength (UTS) and hardness of a rolled specimen have enhanced with rolling. The UTS has increased from 693 MPa (as received) to 1700 MPa (after 90% deformation). The improvement in UTS of processed samples is due to combined effect of grain refinement and stress induced martensitic phase transformation. The hardness values also increases from 206 VHN (as received) to 499 VHN (after 90% deformation). Magnetic measurements were also conducted to confirm the formation of martensitic phase.

1. Introduction

Austenitic stainless steels (ASS) are widely used in various engineering sectors such as automobile, chemical and petro-chemical, because of their excellent properties (such as good corrosion resistance, excellent weldability, good thermal stability, and superior impact toughness). However, they possess low yield strength due to presence of soft austenite (γ) phase which results in making them less suitable for structural applications [1]. Various strengthening mechanism for metals exist such as grain refinement, transformation strengthening and work hardening. For structural application, material should possess high strength to weight ratio. There are various methods for producing high strength material via grain refinement routes such as equal channel angular pressing (ECAP), high pressure torsion (HTP), multiple compression, hydrostatic extrusion etc., however, the product produced by these processes are of high strength but size limitation (i.e. large size products cannot be produced). Over 70 % of metal products are produced by rolling process in one or the other way, as rolling is an easy method for producing long length sheets [2]. Cold working is a suitable strengthening method as ASS has high strain hardening coefficient [3, 4]. Rolling being a cold working process may be employed to ASS to improve mechanical properties.

The transformation of austenite to martensite is the principle phase transformation and provides the basis for important structural materials. The ASS can be transformed to martensitic either
via heat treatment or plastic deformation. Deformation or strain induced martensite is a unique feature of ASS [1]. Two types of martensite namely $\alpha'$-martensite (BCC) and $\varepsilon$-martensite (HCP) may be formed spontaneously upon plastic deformation of ASS, out of these two $\alpha'$-martensite is ferromagnetic. Therefore, paramagnetic ASS becomes ferromagnetic after deformation. Olson et al. [5] and Sato et al. [6] have proposed two major phase transformation pathways based on stacking fault energy (SFE): (i) $\gamma\rightarrow\varepsilon\rightarrow\alpha'$ (SFE $< 18 \text{ mJ/m}^2$) and (ii) $\gamma\rightarrow$ twinned $\gamma\rightarrow\alpha'$ (SFE $> 18 \text{ mJ/m}^2$). At high extent of deformation, $\alpha'$-martensite (thermodynamically more stable) grows at the expense of previously formed $\varepsilon$-martensite phase. The present work is focussed on the room temperature rolling of 304 ASS and investigation of phase transformation and mechanical behaviour.

2. Experimental
The commercially available of Type 304 austenitic stainless steel was used in the present study and was procured as 300 mm x 50 mm x 3 mm cold-rolled plate stock. The chemical composition of as received material was analyzed and is shown in Table 1. Prior to room temperature rolling, the samples of rectangular size 50 mm x 25 mm x 3mm were cut from the cold-rolled plate stock in the rolling direction to achieve 30, 50, 70 and 90 % reduction in thickness. Reduction of 0.05 mm per pass was given using a standard rolling mill of 110 mm roller diameter and the rolling speed was 8 rpm. The plastic strain rates were assumed to be constant throughout the process. Tensile testing of the rolled stainless steel was carried out on BISS 25KN using ASTM-E8 substandard specimen with gauge length of 16 mm and a crosshead speed of 1 mm/minute. Five samples of each condition were testing to ascertain reproducibility.

Micro-hardness tests were carried out using UHL VMHT with 100 gf load and 12 second dwell time at room temperature with indentation speed of 35 $\mu\text{m/s}$. Before hardness measurement, samples were surface polished up to 2000 grit size emery paper followed by cloth polishing. Minimum of 10 readings were taken and its average is taken for hardness data. Microstructures were captured by using Leica microscope DMI 5000 prior all specimens were polished by 2000 grit size emery paper followed by cloth polishing using alumina and a mixture of nitric acid and hydrochloric acid (1:3) solution was used as etchant. Room temperature rolled stainless steel samples was characterized by using advanced X-ray diffraction meter RIGIKU SMARTLAB 3KW using Cu $K_\alpha$ radiation with scan rate of 1 ºper minute. Magnetic hysteresis loops were obtained by using vibrating sample magnetometer (VSM) of QUATUM DESIGN VERSA LAB 3 Tesla.

| Element | C  | Cr  | Ni  | Mn  | Si  | P   | S   | Fe  |
|---------|----|-----|-----|-----|-----|-----|-----|-----|
| Content wt% | 0.068 | 18.2 | 8.59 | 1.24 | 0.408 | 0.070 | 0.003 | Bal. |

3. Results and Discussions
X-ray diffraction patterns of the 304 austenitic stainless steel specimens before and after room temperature rolling are shown in Fig. 1. New diffractions peaks starts emerging after rolling as compared with the sample without deformation, indicating that the deformation induced martensite transformation occur in 304 austenitic stainless steel during rolling.
Figure 1. X-ray diffraction patterns of 304 austenitic stainless steel for different percent reduction.

Figure 2. Microstructure as received and deformed 304 Austenitic Stainless Steel.
Fig. 2 shows the optical micrographs of as received and 30, 50, 70 & 90 % deformed 304 austenitic stainless steel. On rolling, grains are elongated in the rolling direction and the grain size reduces of the as-received samples reduces with the deformation. Diffuse nature of grains in specimens with 50, 70 & 90 % reduction is a manifestation of severe distortion.

Stress induced martensitic transformation and twinning due to deformation in austenite (γ) can be influenced by the stacking fault energy of the stainless steel, for stainless steels with SFE = 45 mJ/m$^2$ plastic deformation occurs through dislocation glide while direct transformation of γ→α' is reported in stainless steel with SFE below 45 mJ/m$^2$. Whereas, deformation twinning has occurred in austenitic SS with SFE in the range of 18-45 mJ/m$^2$ [1] The stacking fault energy (SFE) of the austenitic stainless steel is determined by its composition and can be calculated by using the formulae [3].

\[
\text{SFE (mJ/m}^2\text{)} = -53 + 0.7(\%\text{Cr}) - 6.2(\%\text{Ni}) - 3.2(\%\text{Mn}) + 9.3(\%\text{Mo})
\]

By using the above formula, SFE of the 304 austenitic stainless steel used for study was found to be 19 mJ/m$^2$. It is therefore more twin boundaries observed on deformation.

![Figure 3. Effect of room temperature rolling on the tensile properties of 304 austenitic stainless steel.](image)

![Figure 4. Effect of room temperature rolling on hardness of 304 austenitic stainless steel.](image)

Fig.3 (a) demonstrates the effect of rolling on the stress-strain behavior of 304 austenitic stainless steel and Fig.3 (b) provides the summary of stress-strain behavior. The ultimate strength increased from 693 MPa (as received) to 1700 MPa (90% reduction). Percentage elongation (ductility indicator) of the as received sample was 30 % and drops to 1% approximately with 90% deformation. The increase in the ultimate tensile strength is attributed with the transformation of austenite to α'-
martensite [2, 7]. Deformation induced martensite formed in the specimens after rolling resulted in the enhancement of mechanical properties (increased strength & hardness. The effect of rolling on the micro-hardness of rolled samples is shown in Fig.4 (a). Each point is a representation of an average of 10 readings on the rolling surface along the rolling direction. Fig.4 (b) represents the correlation between the deformation induced martensite (computed from VSM data) and micro-hardness with the percentage reduction. The hardness value of the as received specimen was about 206 VHN which increases to 499 VHN approximately for 90% deformed sample.

Ferromagnetic phase is developed in the austenitic stainless steel by room temperature rolling and is quantified by magnetic measurements. Fig. 5 (a) shows the magnetization plot as a function of the magnetic field for as received and deformed samples. Table 2 summarize the magnetic properties, volume percent of α'-martensite, γ-austenite and hardness values of as received and rolled specimens. Table 2 shows that rolled 304 austenitic stainless steel has austenite and martensite phases. These phases have different crystal structures and therefore have different magnetic characteristics. Austenitic phase is paramagnetic at room temperature i.e. saturation magnetization should be zero. Martensitic phase is ferromagnetic in nature, so it can be helpful in determining the amount of ferromagnetic phase in the sample. Deformation of austenite to martensite phase is dependent on percent of reduction, which is seen from the hysteresis plots, as the percent of reduction increases there is a uniform increase in the saturation magnetization. Negligibly small magnetization exists in as received specimen and after rolling the increase in magnetization of the specimens is noticed and the maximum magnetization is obtained for specimen with 90 % room temperature rolling. The formation of martensite primarily depends on the percent of reduction in thickness (Fig. 1). The volume percentage of α'-martensite transformed was calculated using saturation magnetization [7, 8].

![Figure 5](image)

**Figure 5.** (a) Magnetization plotted against magnetic field for as received and after different reduction in thickness of specimen after room temperature rolling. (b) Volume % of γ-austenite and α'-martensite plotted against different percentage reduction.

Figure 5 (b) shows that with progress in rolling, the volume percent of α'-martensite increases at the expense of γ-austenite. This indicates that magnetization in rolled specimen developed because of α'-martensite transformation [7]. The remanence ratio decreases as percent of deformation increases which indicates the magnetic power of austenitic stainless steel are related to the volume percent of α'-martensite. As the percent reduction increases, there is a fusion of grain boundaries and formation of elongated grains, which shows severe distortion in material during rolling process. Deformation induced martensite formed in the specimens after rolling resulted in the enhancement of mechanical properties (increased strength & hardness).
### Table 2. Magnetic properties, volume percent of α’-martensite, γ-austenite and hardness values of as received and rolled specimens.

| S.No | % Reduction | M_S (emu/g) | α’-martensite (%) | γ-austenite (%) | Remanence ratio | Hardness(VHN) |
|------|-------------|-------------|-------------------|-----------------|-----------------|--------------|
| 1    | 0           | 0.729       | 0.47              | 99.53           | 0.049           | 208          |
| 2    | 30% RTR     | 20.421      | 13.26             | 86.74           | 0.121           | 349          |
| 3    | 50% RTR     | 40.813      | 26.58             | 73.42           | 0.047           | 461          |
| 4    | 70% RTR     | 66.044      | 42.88             | 57.12           | 0.029           | 462          |
| 5    | 90% RTR     | 99.28       | 64.47             | 35.53           | 0.026           | 499          |

RTR: room temperature rolling, M_S : saturation magnetization, VHN: Vickers hardness number

### 4. Conclusions
The microstructure, mechanical and magnetic behavior of 304 austenitic stainless steel before and after room temperature rolling were characterized and analyzed systematically, the results are as follows:

1. The amount of α’-martensite (Deformation induced martensite) in 304 austenitic stainless steel increases with percentage deformation.
2. The ultimate strength of the 304 austenitic stainless steel increases with the reduction in thickness by rolling at room temperature, the ultimate strength increased from 693MPa (as received) to 1700 MPa (90% reduction).
3. The micro-hardness increased with the progress in rolling, from 206 VHN to 499 VHN (90% deformation), which is more than twice of as received sample.
4. Saturation magnetization increases with the increase in percentage reduction (the reason of increase is the increase in volume percent of α’-martensite).
5. With increase in percentage of reduction of the sample, there is a significant change in grain size and grain nature (elongated grains in direction of rolling and diffused nature of boundaries of grain boundaries).

### References
[1] Roy B, Kumar R and Das J 2015 Effect of cryorolling on the microstructure and tensile properties of bulk nano austenitic stainless steel Materials Science & Engineering A 631 pp 241–247.
[2] Yu H L, Lu C., Tieu K. and Godbole A 2013 Recent developments in flat rolling technologies F.D.S. Marquis (Eds.), PRICM: 8 Pacific Rim International Congress on Advanced Materials and Processing pp 2139-2146.
[3] Milada M, Zreiba N, Elhalouani F, and Baradai C 2008 The effect of cold work on structure and properties of AISI 304 stainless steel Journal of Materials Processing Technology 203 pp 80–85.
[4] Hedayati A, Najafizadeh A, Kermanpur A and Forouzan F 2010 The effect of cold rolling regime on microstructure and mechanical properties of AISI 304L stainless steel Journal of Materials Processing Technology 210 pp 1017–1022.
[5] Olson G B and Cohen M 1975 Kinetics of Strain-Induced Martensitic Nucleation Metallurgical Transactions 6A pp 791-795
[6] Sato K, Ichinose M, Hirotu Y and Inoue Y 1989 Effects of deformation induced phase transformation and twinning on the mechanical properties Austenitic Fe-Mn-Al Alloys, Iron and Steel Institute of Japan International 29 pp 868–877.
[7] Mumtaz K, Takahashi S, Echigoya J, Kamada Y, and Zhang L F 2004 Magnetic measurements of martensitic transformation in austenitic stainless steel after room temperature rolling Journal of Materials Science 39 pp 85–97.
[8] Mumtaz K, Takahashi S, Echigoya J, Kamada Y, Zhang L F and Sato M 2002 Use of saturation magnetism measurements to detect martensite formation in austenitic stainless steel after compressive deformation at high temperatures *Journal of Materials Science Letters* 21 pp 1199-1201.