The Relationship Between Surface roughening and Resistance Heating in Austenitic Thin Metal Foils of SUS 304 and SUS 316 During Tensile Test

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Abstract: The effect of resistance heating (RH) on the surface roughening (Ra) of thin metal foils (TMF), SUS 304 and SUS 316, were investigated using tensile testing at an elevated temperature of 500°C with 1.5% as the low strain level and 2.5% as the high strain level for one cycle. A total of five cycles were conducted. After each tensile test cycle, the Ra was measured. The results show that Ra increased proportionally both at the low and high strain levels. The Ra increase was similar in the fine grains of SUS 304 and SUS 316 TMF with the same strain level. The Ra increase was higher in SUS 316 compared to SUS 304 TMF, both at the low and high strain levels. The inhomogeneous grain strength between SUS 304 and SUS 316 TMF was similar in the fine grains but differed in the coarse grains. Using an electron microscope, the electron backscattered diffraction (SEM-EBSD) scanning result was obtained and showed the grain misorientation effect on the inhomogeneous grain strength.

Keywords: Resistance heating (RH), martensitic phase transformation (MPT), grain misorientation (GMO), thin metal foils (TMF), increasing surface roughness (ΔRa).

1. Introduction

The stainless steel SUS 304 and SUS 316 thin metal foil (TMF) have wide applications in the automobile components, electronic parts, biomedical devices, and food industries. The limitation of the material application and expensive mass production are the most significant challenges in the microforming industries [1-3]. Deformation in stainless steel such as SUS 304 and SUS 316 influences the occurrence of martensitic phase transformation (MPT). MPT occurs due to transformation during the austenitic phase after plastic deformation in stainless steel such as SUS 304 and SUS 316 [3,4,11,13]. Zheng et al. [5] concluded that a heat-assisted microforming process reduced the size effect in microforming and improved the springback effect of pure titanium foils. M.R. Stoudt et al. [6] found the strain localized in an aluminum alloy and a relationship between deformation-induced surface roughness and grain orientation. Cheng et al. [7] found that surface roughening is the crucial factor that affects fracturing in thin or sheet metal rather than the nucleation and growth of voids inside the material. Hao Zhang et al. [14] concluded that metal plasticity can be significantly improved when worked under warm or hot conditions. Working with thin or sheet metal at elevated temperatures can increase dislocation mobility, making it possible to impose the plastic strain on alloys that are hard to deform at room temperature. Xu et al.[16] reported that electric currents can also enhance the recrystallization rate of cold-worked α-titanium. Kengo Yoshida et al. [15] concluded that surface roughness is mainly affected by the grain size (Dg) and that there is a need to investigate surface roughness behavior in Dg lower than 10 µm. Aziz et al. [8] concluded that martensitic phase transformation (MPT) is the main factor that affects surface roughness in the coarse grains of stainless steel, but the effect of MPT is low for fine grains.

Conducting tensile tests at elevated temperatures can reduce the effect of MPT and show surface roughening behavior without the effect of MPT, but only with the different deformation mechanisms for various grain sizes. However, the effect of heating on surface roughening is still not clear, and there has been no experiment on the correlation between elevated temperature with MPT, grain misorientation and surface roughening compared with room temperature conditions. The purpose of this research is to clarify the effect of elevated temperatures on MPT, grain misorientation and surface roughness behavior in thin stainless steel metal with various grain sizes (Dg) below 10µm compared to room temperature conditions. To achieve our purpose, thin metal foils of SUS 304 and 316 with various grain sizes below 10µm were subjected to uniaxial tensile testing that used resistance heating; then, the surface roughening was measured. This was repeated five times and then compared with room temperature results. Following this, to investigate MPT and grain misorientation, an SEM-EBSD investigation was applied to the thin metal foils using RH and the results were compared with the room temperature conditions.

2. Materials and Research Method

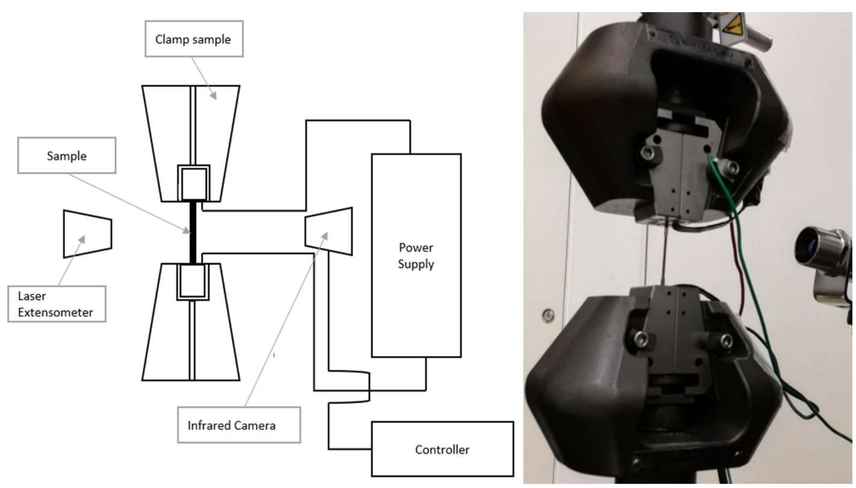
2.1. Materials

This experiment using the same as receive condition with the previous study , such as material compositions, sample dimension, microstructure and how to calculate the grain size (8). The grain size (Dg) for whole materials used in this experiment are 1.5 µm; 3.0 µm and 9.0 µm. The material samples used in this experiment are SUS 304 and SUS 316 thin metal foils. the thickness of the sample was 0.1 mm.

2.2. Method

Ethanol solution and electrical vibrations were performed for 30 minutes to clean the material samples of SUS 304 and SUS 316 TMF. SUS 304 and SUS 316 were subjected to uniaxial tensile testing for five cycles with a constant strain level. Surface roughness was measured after one cycle of tensile testing. The AGX-50KNVD machine, with a 50 KN capacity, produced by Shimadzu, Co, Japan, was used for the tensile testing. The heating system shown in Fig. 1a consisted of a laser extensiometer, infrared camera, controller, power supply, and a jig that used a ceramic insulator in the tensile testing. The extensiometer was used for measuring the gauge length displacement during the tensile test. The infrared camera was used for measuring and controlling the temperature during the tensile test. The function of the controller was to control the temperature input and control the stability of heating by finding the proportional (P), integral (I), and derivative (D). The controller transmitted orders to the power supply. The function of the power supply was to heat the workpiece to the appropriate temperature. The temperature in the sample was controlled by a thermal sensor. The thermal sensor gave information to the controller about the temperature of the workpiece that had been heated with electricity from the power supply. The controller always ordered the power supply to heat the workpiece with the appropriate temperature that had been set by the author. A photo of the clamp used to hold the sample is shown in Fig. 1b, consisting of the jig, workpiece, and thermal sensor.

The resistance heating during the tensile test was used for three minutes for every sample. To heat the samples above the sensitization temperature and below the embrittlement temperature, the resistance heating temperature was 500^{o}C. To keep the temperature constant during heating, and to counteract the electrical current striking the tensile machine, the sample was insulated using ceramics and the temperature was insulated in the inner ceramic area. Equipment used for resistance heating consisted of a thermosensor using Optris Xi-400, made in Japan, and the power supply. The thermosensor gave the order to the power supply to increase the temperature of the workpiece. The emissivity of the samples was 0.94. To obtain a stable temperature, the proportional PID was set up. The PID was set before the resistance heating was started. The PID value in this experiment was P = 113.6 ; I = 1.5 ; D = 0.2. Surface roughness was measured using a confocal laser microscope with the specification OLS-5000, produced by Olympus Co., Japan.



(a). Schematic of resistance heating system (b). Photo of clamp used to hold sample

Figure 1. Configuration of tensile test equipment with resistance heating (RH).

The sample dimensions were as follows: thickness, 0.1 mm; gauge length, 20 mm; fillet radius, 3.6 mm. The samples were subjected to uniaxial tensile testing step-by-step for five cycles with the provision that the materials, SUS 304 and SUS 316 TMF, were clean. During tensile testing, the strain rate was 1.6 x 10^{-3} S^{-1}. The elongation during tensile testing was measured using the camera that was installed in the tensile testing machine. The camera used in this experiment was the DVE-201 camera, produced by Shimadzu Corp., Japan. After the tensile test finished, the sample was taken out and the surface roughness was measured. The surface roughness measurement was carried out in four areas A, B, C, and D (8). The roughness measurement was 0,7 mm as the radius of the surface. When the five steps of uniaxial tensile testing of SUS 304 and SUS 316 TMF were completed, the samples were investigated using a scanning electron microscope, employing electron backscattered diffraction (SEM-EBSD). The results from the SEM-EBSD investigations were grain misorientation (GMO) analysis and phase mapping. SEM SU-70, produced by Hitachi High Technology, Corp., Japan, in normal mode was used as the microstructural analysis specification in this experiment. The acceleration voltage was 5kV, the emission current was 16 µA, and the observation distance was 10 mm. An EBSD Digi View (EDAX) was used to investigate the phase transformation in field-free mode, with an emission current of 16 µA, an acceleration voltage of 15kV, and an observation distance of 20 mm. The pixel binning was 8 x 8, the step resolution of the EBSD machine was 0,1µm, and the area of surface roughness observation was 30 µm x 50 µm.

3. Results of the tensile test and EBSD analysis

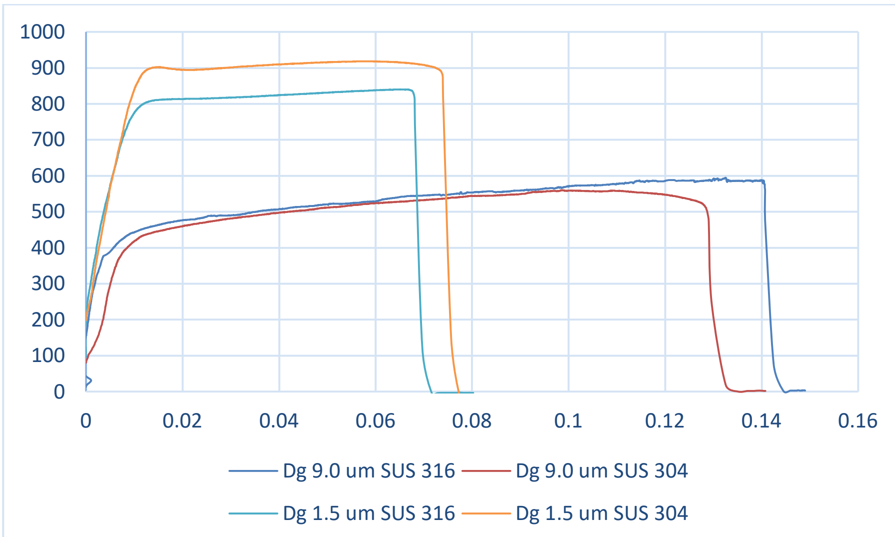
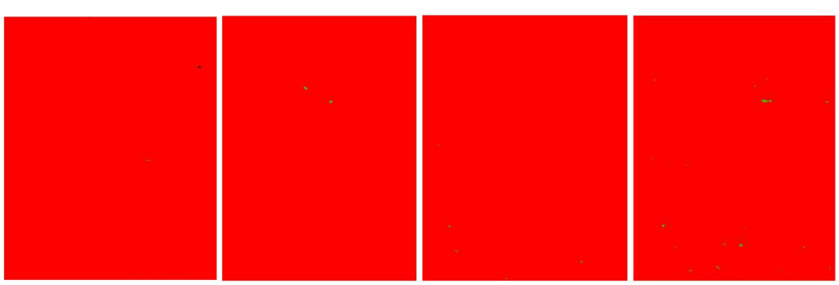


Figure 2. Stress–strain curve in SUS 304 and SUS 316 TMF during resistance heating (RH).

The SUS 316 TMF had higher strength and ductility in the fine grains and higher ductility in the coarse grains during tensile testing using RH when compared to SUS 304 TMF, as shown in Fig. 2. The strength and ductility of the thin metal foils, shown in the stress–strain curve in Fig. 2, correlate with the surface roughening behavior in SUS 304 and SUS 316 TMF. The microstructure had an effect on surface roughness behavior in thin metal foils after resistance heating treatment, which will be explained in the next section.

Fig. 3a–d shows the phase map of SUS 304 and SUS 316 TMF. The phase map consists of the phase transformation from the austenite phase (indicated by the red color) and the martensite phase (indicated by the green color) as received until the high strain level (12.5%). In the SUS 304 coarse grains, the slip band intersection decreased significantly after the plastic deformation, thus, the probability of the slip band intersection equal to zero that was a result of no MPT in the coarse grains of SUS 304. In SUS 316 TMF, austenite does not transform to become MPT because of the existence of an austenite stabilizer, such as nickel.



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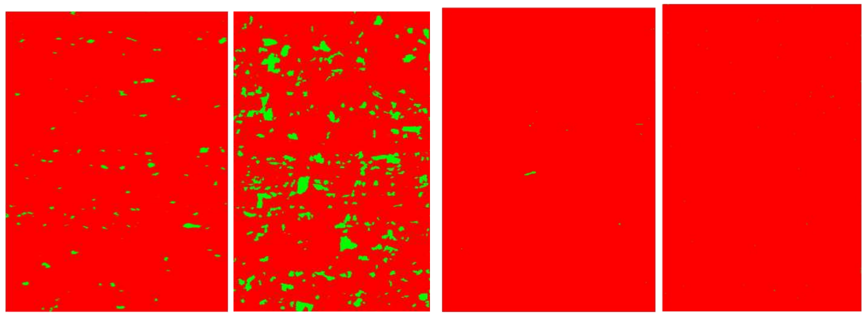
(a). Phase map EBSD Dg 9.0 µm of SUS 304 (b). Phase map EBSD Dg 9.0 µm of SUS 316



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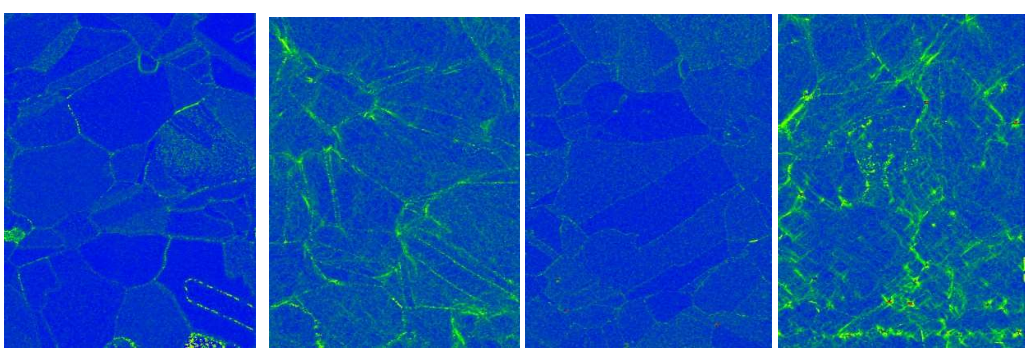
(c). Phase map EBSD Dg 1.5 µm of SUS 304 (d). Phase map EBSD Dg 1.5 µm of SUS 316

• = MPT
• = Austenite

Figure 3. SEM-EBSD in coarse grains and fine grains of SUS 304 and SUS 316 TMF.

According to the phase map, the MPT did not occur in the coarse grains of SUS 304 and SUS 316 TMF because there was no slip band intersection in the coarse grains during tensile testing that used resistance heating (RH). The MPT decreased significantly in the fine grains of SUS 304 TMF compared to at room temperature, because the slip band intersections decreased during tensile testing that used RH. The MPT did not occur in the fine grains of SUS 316 TMF because there were no austenitic phase transformations to MPT after uniaxial tensile testing, which used RH during the testing. The evidence that there was no MPT in the coarse grains of SUS 304 and SUS 316 TMF is shown in Fig. 3a,b. The evidence that there was no MPT in the fine grains of SUS 304 TMF is shown in Fig. 3c. The evidence that there was no MPT in the fine grains of SUS 316 TMF is shown in Fig. 3d.

The grain deformation of coarse grains is inhomogeneous grain deformation because the grain strength was not uniform. The inhomogeneous grain strength in the coarse grains was affected by grain size (Dg) and grain misorientation (GMO), as shown in Fig. 4a,b. At room temperature, the inhomogeneous grain strength in SUS 304 TMF was dependent on the grain size, MPT, and GMO [8,9]. The homogeneous grain strength in the fine grains of SUS 316 TMF was dependent on Dg and GMO, as shown in Fig. 4c,d. The homogenous grain strength in the fine grains of SUS 304 TMF and SUS 316 TMF were similar.



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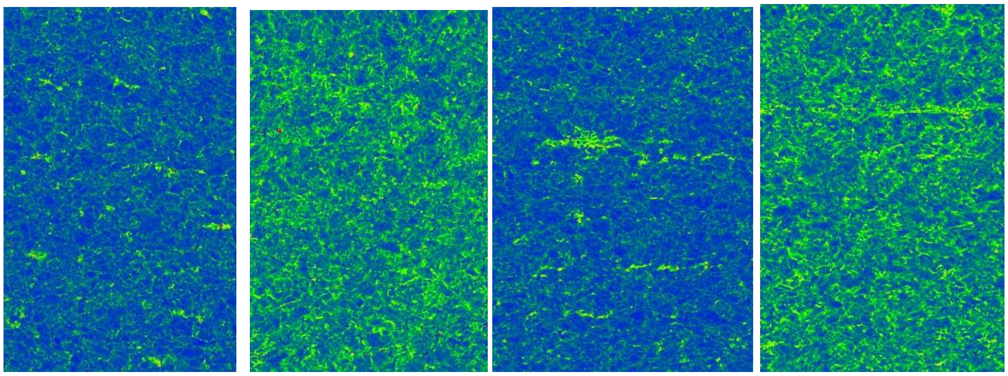
(a). KAM map in SUS 304 Dg 9,0 µm (b). KAM map in SUS 316 Dg 9,0 µm



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(c). KAM map in SUS 304 Dg 1,5 μm (d). KAM Map in SUS 316 Dg 1,5 μm.

• = 2º Green Misorientation
• = 0º Green Misorientation

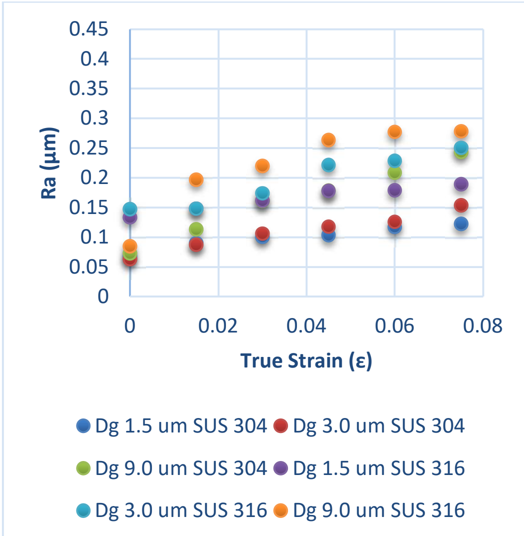
Figure 4. KAM map in fine grains (1.5 μm) of SUS 304.

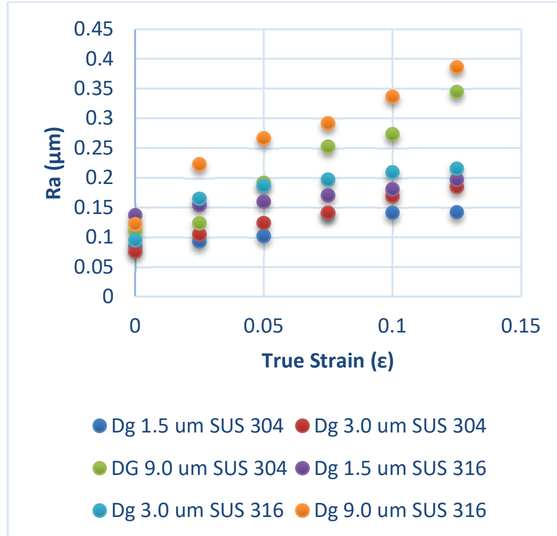
According to Fig. 4, green represents the 2º green misorientation, and blue the 0º green misorientation. KAM (Kernel Average Misorientation) was the average of the grain misorientation (GMO) in the center, and surrounding area, after plastic deformation in the metal.

The change in GMO in the coarse grains in SUS 316 TMF was higher than in SUS 304 TMF, as shown in Fig. 4a,b, which increased the inhomogeneous grain strength in SUS 316 TMF in comparison to SUS 304 TMF. Consequently, the surface roughening in the coarse grains of SUS 316 TMF was higher than in SUS 304 TMF. The difference of increasing surface roughness in SUS 316 TMF compared to SUS 304 TMF at elevated temperatures was lower than the increasing surface roughness of SUS 304 compared to SUS 316 TMF at room temperature [8,13]. Slip band movement increased at higher temperatures compared to at room temperature and the probability of the slip band intersection being the place of martensitic nucleation lessened [8,11-13]. Therefore, the MPT disappeared in the coarse grains of SUS 304 and decreased significantly in the fine grains of SUS 304 TMF at elevated temperatures.

The GMO in fine grains between SUS 304 TMF and SUS 316 TMF were similar; there was a similarity in the GMO effect and a similarity in the surface roughening behavior in the fine grains. Because of resistance heating (RH) during tensile testing, the increase in the surface roughening value in the fine grains was higher compared to the increase in the surface roughening in the fine grains at room temperature, as shown in Fig. 5. This means that the inhomogeneous grain strength in the fine grains using RH was higher than the inhomogeneous grain strength at room temperature (without RH).

Based on the KAM map, as shown in Fig. 4, we concluded that the grain misorientation (GMO) in the fine grains of SUS 304 and SUS 316 TMF were similar, thus, the roughness became similar because the homogeneous grain strength was equal. However, in the coarse grains, based on the KAM map results, the inhomogeneous grain strength in SUS 316 TMF was higher than in SUS 304 TMF, which indicated that the inhomogeneous GMO in SUS 316 TMF was higher than in SUS 304 TMF.



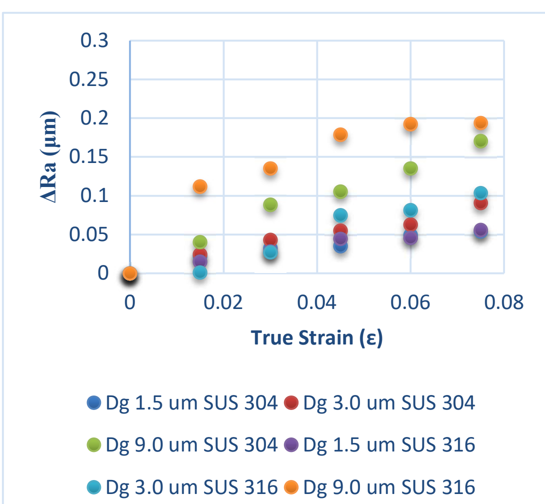


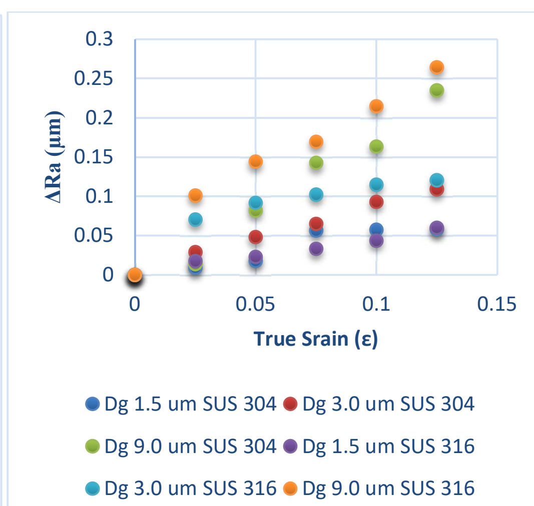
Low strain level High strain level

Figure 5. Surface roughness behavior in SUS 304 and SUS 316 TMF.

Surface roughening increases proportionally both at the low strain and high strain levels, as shown in Fig. 5. The surface roughening increased more at the high strain level compared to the low strain level. The surface roughening increased more for the coarse grains than for the fine grains at both the low and high strain levels, as shown in Fig. 5.

Based on Fig. 5, SUS 316 TMF and SUS 304 TMF have the same tendency for surface roughness (Ra). This result differs at room temperature in investigations on the effect of MPT on surface roughness. Aziz et al. [8] concluded that MPT has a significant effect on the surface roughness of SUS 304 thin metal foils at room temperature. Additionally, because of RH, the ultra-thin layer in SUS 304 and SUS 316 TMF, called the passive layer, consisting of Cr_2O_3, removed that effect in the same slip band movement in SUS 304 and SUS 316 TMF after plastic deformation with the same strain level [5]. Surface roughness in SUS 316 was slightly higher than in SUS 304 TMF because SUS 316 TMF is stronger and more ductile than SUS 304 TMF, as shown in Fig. 5. Additionally, the slip band may move more easily in SUS 316 TMF than in SUS 304 TMF after tensile testing with the same strain level. This means that MPT has no effect on Ra behavior in SUS 304 after tensile testing using the resistance heating (RH) technique.





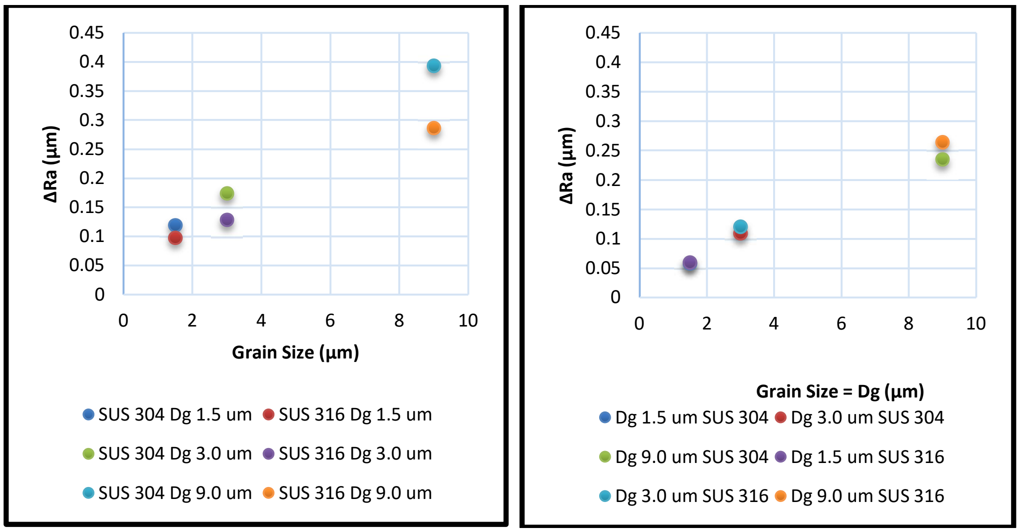
Low Strain High Strain

Figure 6. The increase in surface roughness in various Dg of SUS 304 and SUS 316 TMF.

The increase in surface roughening (ΔRa) in the coarse grains was higher than in the fine grains in both the low and the high strain levels, as shown in Fig. 6. The ΔRa increased more at the high strain level compared to the low strain level in both the fine grains and the coarse grains, as shown in Fig. 6.

Based on Fig. 6, the increase in surface roughness (ΔRa) in the fine grains was similar in both the SUS 304 and SUS 316 TMF at the low and high strain levels. In the coarse grains, the ΔRa was higher in SUS 316 TMF compared to SUS 304 TMF. The ΔRa was higher with the higher strain level after uniaxial tensile testing, but it was not so high in the fine grains and the tendency of ΔRa was similar between SUS 316 TMF and SUS 304 TMF. This also means that the slip band and dislocation movement in SUS 316 TMF and SUS 304 TMF were similar, which resulted in a similar ΔRa.

4. Discussion on Different in Deformation Between Resistance Heating (RH) and Room Temperature

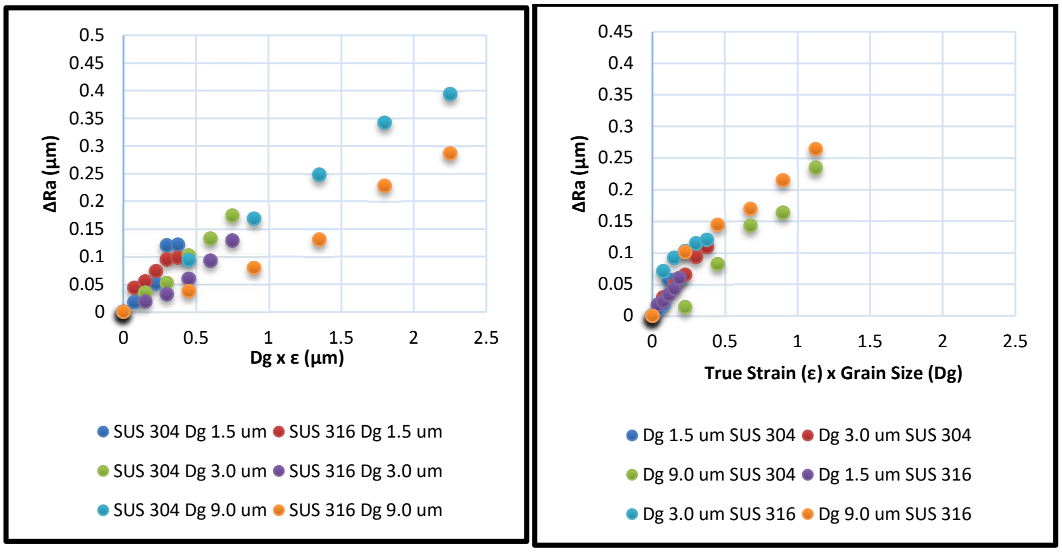


(a). Room temperature (b). Elevated temperature

Figure 7. The increase in surface roughness (ΔRa) behavior in SUS 304 and SUS 316 TFM

For revealing the effect of elevated temperature on surface roughening, the roughening behavior for various grain size was compared to that in room temperature. The increase of surface roughening in SUS 304 was higher than SUS 316 in room temperature as shown in Fig.7a, because SUS 304 has MPT that spread nonuniform in coarse grain and no MPT in SUS 316 coarse grain. The increase of surface roughening at elevated temperature was similar between SUS 304 and SUS 316 as shown in Fig.7b, because no MPT both in SUS 304 and SUS 316 coarse grain. at elevated temperature, the passive layer removed, thus the slip band movement become similar between SUS 304 and SUS 316 thin foils (5).

The increase in surface roughening was higher for larger grain sizes (Dg) than fine grains with the same strain level for both room temperature and elevated temperature, as shown in Fig. 7. The increase in surface roughening was higher in SUS 304 than in SUS 316 at room temperature, as shown in Fig. 7a (8). The increase in surface roughening was higher in SUS 316 than in SUS 304 TMF at an elevated temperature. The increase in surface roughening at room temperature was higher than at an elevated temperature, as shown in Fig. 7 because at room temperature, SUS 304 has a higher inhomogeneous grain strength than SUS 316 TMF (8). The increase in surface roughening was higher in SUS 316 TMF than in SUS 304 TMF at an elevated temperature, as shown in Fig. 7b, because SUS 316 TMF has a higher inhomogeneous grain strength than SUS 304 TMF, as shown in Fig. 4a,b. From the previous result [8-9], a higher inhomogeneous grain strength showed higher surface roughness than a lower inhomogeneous grain strength at the same strain level. The various increases in surface roughness (ΔRa) are shown in Fig. 7. The ΔRa after RH in fine grains in both SUS 304 and SUS 316 TMF were similar because of the similarity of the homogeneous grain strength. The ΔRa in SUS 304 compared to SUS 316 TMF in fine grains after RH was lower than without RH because the fine grains of SUS 304 and SUS 316 after RH have a higher homogeneous grain strength in comparison with room temperature. ΔRa in the coarse grains after RH in SUS 304 compared to SUS 316 TMF was low and lower than at room temperature because the inhomogeneous grain strength in SUS 304 and SUS 316 TMF after RH was lower than at room temperature (RT).



(a). Room temperature (b). Elevated temperature

Figure 8. Relationship between increase in surface roughness (ΔRa) production of grain size (Dg) and true strain (ε).

The relationship between ΔRa production, Dg, and ε showed a higher increase at a higher strain level. The relationship between ΔRa production, Dg, and ε showed a higher increase at a higher Dg at both the low and high strain levels, as shown in Fig. 8. Previous research [10] shows that $R_a = C \cdot D_g \cdot \varepsilon + R_o$……… (1).
The relationship between ΔRa production Dg and ε was $\Delta R_a = R_a - R_0$. We could write this equation as: $\Delta R_a = C \cdot D_g \cdot \varepsilon$ ……… (2), where D_g is grain size, ε is true strain, ΔR_a is the increase in surface roughness (μm), R_0 is the initial surface roughness (μm), and C is the material constant or gradient.
The gradient at room temperature between fine grains and coarse grains differed. At room temperature, as shown in Fig. 8a, the gradient of increasing surface roughness in fine grains was higher than in coarse grains, because fine grains are more homogeneous and their grain strength is higher compared to coarse grains. The slip band movements in fine grains were lower than in coarse grains with the same strain level (17). Slip band movement was easier and faster under RH conditions because of the passive layer removed during heating treatment (5). In a previous study [8], the materials constant, *C*, was affected not only by the grain size and plastic strain level, but also by MPT. In our results, there was no MPT in the coarse grains to affect the material constant (*C*). Since there is no MPT in SUS 304 and SUS 316 TMF coarse grains, this affected the similarity of ΔR_a and the gradient in the fine grains and the coarse grains at both the low strain and high strain levels of SUS 304 and SUS 316 TMF. Because there are no effects due to MPT in coarse grains, the material constant (*C*) in our results was affected by the grain size and grain misorientation. Based on Fig. 8, the gradient tendency of the graph is similar in fine grains and coarse grains because there is no MPT effect.

Conclusion

1. Surface roughening properties were investigated in comparison to room temperature. The findings are shown below. The MPT did not occur in the coarse grain of either the SUS 304 TMF or the SUS 316 TMF. The MPT occurred in the fine grains of the SUS 304 TMF, but the volume fraction was much smaller than that under room temperature conditions.
2. After uniaxial tensile testing using RH, the coarse grains showed lower inhomogeneous grain strength compared to room temperature, which had the effect of lowering the increasing Ra in coarse grains after RH compared to room temperature.
3. After tensile testing using RH, the increase in surface roughness in SUS 316 was higher than in the SUS 304 TMF both at the low and high strain levels, showing the differing behavior in comparison with room temperature.
4. The homogeneous grain strength in fine grains after RH was lower than in room temperature which increased surface roughness in comparison to room temperature.

5. The gradient tendency for the relationship between increasing Ra and the product grain size (Dg) and strain level (ε), both in the fine grains and coarse grains of SUS 304 and SUS 316 TMF after RH, was more similar than that at room temperature.

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