Effect of Martensitic Transformation and Grain Size to Surface Roughening Behavior in Thin Metal Foils SUS 304 and SUS 316

by Abdul Aziz

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Effect of Martensitic Transformation and Grain Size to Surface Roughening Behavior in Thin Metal Foils SUS 304 and SUS 316

Abdul Aziz (1), Ming Yang (2), Tetsuhide Shimizu (3), Tsuyoshi FURUSHIMA (4)

(1), (2), (3). Advance Materials Engineering Laboratory (AMPEL) Tokyo Metropolitan University, Hinoshi,6-6- Asahigaoka 191-0065. JAPAN

(4). Institute of Industrial Science The University of Tokyo, Meguro Tokyo JAPAN.

1. Introduction

With the large demands for microparts in electronics, automobile components, and biomedical devices, microforming has been received much attention in recent decades. Beside that, micro manufacturing and forming methods have many issues such as the limitation of the material application and requirement of high cost mass production (1-4).

However, in metal forming and miniaturization process, **the surface roughness ratio of the material to the thickness increase with decreasing sheet thickness, this is called as non uniformity** thickness.(5)–(7). **Roughening phenomena** of sheet material will have **strong effect on necking and fracture** behavior of materials. **The inhomogeneous deformation of each grain located near the free surface will cause the roughening phenomena on the free surface of polycrystalline metals**. (8).

Today also the development of industry in the micro scale so rapidly. Namely industries engaged in the field of electronics, biomaterials, micro stamping technology such as micro gasket, micro shim and metal shealing ring, etc (9). To the capability of batch production, **the micro parts used in electronic and biomedical industrial cluster are largely fabricated by micro forming technology**. However, the **material flow** and **failure behavior** of sheet metal with thin thickness are **influenced by size effect** that is mainly caused by the fewer grains in straining zone (10). Furthermore, furushima et all concluded that the free **surface roughening phenomenon increases with the decrease of sheet thickness.** Considering that the ratio of free surface roughness to thickness of thin sheet is comparable to that of conventional sheet, the free surface roughening significantly affect the plastic deformation behavior and ductile fracture in micro forming. Dimple not occur for pure copper thin foils with thickness 0,05mm until 1,0 mm, this means fracture caused by free surface roughening. From their work, **there is an issue** that there is possibility to suggest the new ductile fracture criterion by utilizing the inhomogeneous FE model in the future with use another materials with various thickness.(11). Meng et al, found that the effect of the **free surface roughening on flow behavior and fracture strain is remarkable when the surface non uniformity increases to the same magnitude of sample thickness (12).** Abe et all, constructed a constitutive model to describe the deformation behavior of metal foil based on the free surface roughening phenomenon (13).

The nature of **grain boundary**, **grain orientation**, **grain size** all **play important roles** in **delineating (melukiskan) the intrinsic competition** between **strengthening**, and **weakening. Strengthening, weakening** and **surface roughening** closely **correlated** with **size effect** in miniaturized polycrystalline materials (14).

P. Groche et al , found that **decreasing grain size**, **the flattening of the surface asperities is hindered (terhalang) because of the increase in yield stress due to the hall – petch relationship**. They also found that the statistical influence of single grain vanishes (lenyap), as soon as the plastic zone is assembled of at least 20 grains. **However, lack of study in the influence of polycrystalline workpiece structure on contact based surface evolution. Then, influence of grain size on surface asperity flattening due to contact loading is not well understood. This is a challenge to explore more about correlation between grain size, crystal structure and their orientation based on contact and noncontact on surface roughening or asperities (15)**.

Surface roughness evolution during plastic process depends on the loading path, crystal structure, grain size, texture distribution, initial surface roughness of the product. (16). Stoudt and ricker found that the roughening rate was dependent on Mg-Al alloy grain size, and there is linear correlation between roughening rate and grain size. **Except of grain size , grain shape also affects the surface roughness after plastic deformation. Texture, grain size, have possibility correlation with surface roughness.(17).**

L.,Zhang et al, found that the surface during plastic straining can be divided into heterogeneous deformation surface and homogenous deformation surface according to whether considering the heterogeneity of polycrystalline material or not. **But they only investigate in FCC polycrystalline and uniaxial tension. There is an issue that many possibilities for another crystal structure materials with uniaxial or biaxial tension (18)** .

Romanova et all, did their experiment in HCP pure titanium. Mostly meso/micro scale surface roughening have been investigated in FCC metals. A promising approach appears to be based on the measurement of the meso scale surface roughness with the proviso that a **direct relation between roughness intensity and degree of local plastic strain established.** One of approximation method is flitting curve between roughness intensity versus local strain, **but the other approximation need to be verified. There are an issue correlated to this research** that **first** In the future need to investigate the effect of crystal structure, grain size, grain orientation, inter grain movement during plastic deformation correlated to surface roughening on BCC titanium alloys, that could be implemented in biomedical, electric/electronic application. **The second issue** is need to verify the validity between roughness intensity and local plastic strain. **The third issue** deep understanding of the mesoscale processes would offer a clue for predicting plastic strain localization and fracture of the material far in advance these processes become apparent on the macroscale (19).

Rabee et all, evaluated this relationship by examining the influence of the degree of mis orientation among sets of neighboring points on the surface as a function of plastic deformation. **Their result demonstrated that the heterogeneity of the deformed surface correlated with the changes in crystallographic orientation produced by the plastic strain. The issue is** need to clarify between effect of grain orientation on critical strain localization. The relationship between local microstructure and deformation induced surface roughness need to clarified.(20).

Lei Zhang et all, worked research on quantitative analysis of surface roughness. They found that at present the quantitative description of surface roughness evolution is limited on FCC polycrystalline metals. **There is a chance to work beside FCC structure on investigating surface roughening, necking and fracture behavior prediction of thin foils (21).**

Kengo Yoshida et al (22), **found that the magnitude of surface roughness is mainly governed by the grain size** and is less sensitive with ratio thickness to grain size (Ng) and initial thickness. Hence, the thickness imperfection due to (disebabkan oleh) surface roughness become large relative to thickness as Ng decreases. Roughening of the free surface of the specimen is induced by the grain- scale strain heterogeneity associated with local grain misorientation. **There are some issues** from his worked, **first the influence of grain scale strain inhomogeneity on the limit strains and surface roughness become significant in the forming processes of the miniature products, and deeper understanding on these effect is necessary**. **Second**, the effect of the number of grains across thickness (Ng) is pursued only by maintaining a constant aspect ratio of the specimen. Finite – element analysis based on a crystal plasticity model is performed to incorporate heterogeneity due to the variation of grain orientation and to examine its evolution. **Third need to investigate effect of grain scale heterogeneity on surface roughness and sheet metal necking for material with grain size lower than 10μm.**

Ben D. Beake et al (23), found that a strong size effect was observed, with the stress for incipient (untuk yang baru jadi) plasticity increasing as the indenter radius was decreased. The maximum shear stress approached the theoretical shear strength when W (100) was indented with the tip with smallest radius, whereas the (111) orientation showed pop-ins at lower stress levels, which has been attributed to surface roughness and greater dislocation density on the W (111) sample. **There is an issue** that it is not clear, how much of the reduction in pop ins is due to surface roughening and how much is due to higher pre-existing dislocation in the near surface layer of tungsten. Linfa Peng et al (24), found that with the increase of grain size, the individual grains, especially the surface grains, become less restricted due to the decrease of grain boundary density. Considering that the orientation and structures of individual grains are random distributed, the inhomogeneous and uneven deformations of surface grain become more significant, which lead to the increase of surface roughness with the grain size after tensile test. **There is an issue** that the effect of grain orientation and its evolution on the deformation of micro/meso scale found to be one of the intrinsic reasons of size effect. Further researches are needed to verify the established method under various microforming conditions.

Furushima et al (25), They found that the fracture strain of pure copper and pure titanium dramatically decrease from thickness 0,3 mm to 0,1 mm. **Thickness strongly affect to fracture strain**. Micro metal forming for metal foil with ultra thin thickness has problem on size effect. The ratio of surface roughness to thickness for each material linearty increase with increasing plastic strain.

Fu et all (26), They found that fracture strain decrease with decreasing thickness of specimen in uniaxial tensile test, thus the metal foil in micro scale indicate different fracture behavior from metal sheet in macro scale.

Furushima et all (27), They used pure copper C 10220-O with thickness 0,05mm. They showed the free surface roughening behavior of pure copper foil C1020-O under uni-axial tensile state obtained from FE simulation.

It is considered that the weak grain with lower flow stress preferentially deforms with the plastic deformation, which lead to surface roughening. There are several founding, first. The weak grain with lower flow stress preferentially deform with the plastic deformation, which lead to surface roughening. The validity of inhomogeneous FE material modelling can be verified for prediction of surface roughening of uniaxial tensile deformation. **From this also there are some issue**, the **first** is the prediction of free surface roughening and necking behavior based on surface roughening is very important in micro metal forming for metal foils with ultra thin thickness. Material inhomogeneity such as difference in grain orientation and size cause free surface roughening. **The second** issue is the free surface roughening of metal foils may affect not only local problem such as accuracy of products and frictional condition but also global deformation behavior such as necking behavior which is onset on fracture.

Qiu Zheng et al (28), Size effect cause inhomogeneous material characteristic and large scattering of the process parameters, which make the forming process unpredictable at the microscale. The reason in developing heat assisted microforming are low energy consumption, high forming accuracy and limiting heat transferring tools. **Heat assisted micro forming** process was **reduced the size effect** in micro forming and improve spring back effect of pure titanium foils.

M.R. Stoudt et al (29), have done an experiment on investigating relationship between grain orientation, deformation – induced surface roughness and strain localization in an aluminium alloy. Materials used is polycrystalline aluminium AA6022, they did mechanical polished, uniaxial tensile by repeating the previous measurements for different materials with different crystal structures and by combining roughness measurements with information provided by orientation imaging microscopy (OIM). By repeating the previous measurements for different materials with different crystal structures and by combining roughness measurements with information provided by orientation imaging microscopy (OIM), tensile test and SEM EBSD observation. **They found** that the high degree of correlation between the density and location of these large surface displacement and the local plasticity condition indicate that a critical localization event is most likely to initiate in grain boundary regions where unfavorable slip interaction produce the largest plastic strains. This research has obtained several **issues that** need to investigate in the future, **firstly** information about the subsurface grain**. The second** is the activity of individual slip system in each grain and how this affect local inter-grain stresses and strains, and **third** a consideration of how “clusters” of grains affect deformation at the multiple grain length scale.

forming limit diagram, also known as a **forming limit curve**, is used in sheet metal forming for predicting forming behavior of sheet metal (30).

Cheng Cheng et al (31), found for the thin sheet metal whose fracture would be due to free surface roughening rather than nucleation and growth of voids inside the material, the modified M-K model considering the evolution of the actual surface irregularity is more suitable for prediction of fracture limit curve (u-FLC) than the origin M-K model.

Hao Zhang et al (32), **Metal plasticity can be significantly improved with warm/hot working. Elevated temperatures can increase the dislocation mobility, making it possible to impose plastic strain on alloys that are hard to deform at room temperature.** However, furnace heating or induction heating in traditional warm/hot working processes have complex setups and are not easily integrated with the ultra nano surface modification (UNSM) process. In contrast, an electric current can provide thermal energy through Joule heating very efficiently. **They found that electric assited forming (EAF) could reduce the flow stress in deformed samples due to an electro plastic effect.** In addition to the mechanical behavior, the microstructure evolution of the alloy was also studied after the EAF process. Xu et al. (33) **reported that electric current also enhanced the recrystallization rate of cold-worked α-titanium.** A finer recrystallized grain size was obtained due to more efficient recrystallization when an electric current with the appropriate density was used. **The issue,** there is localized heating could potentially facilitate pore closure under ultrasonic striking, because of localized heating, there is possibility to become micro initial crack, because localized heating will onset to become microporous.

Yang et all (34), developed **ultrasonic vibration** in phosphor bronze C5191 thin foils showed **that surface roughness (Ra) was reduced from 102nm to 21nm**. However several issues in temperature distribution and properties conductivity of workpieces need to be solved in real condition of manufacturing process. **It is found that reduction of roughness increases with increase of the amplitude for both frequencies.** The SEM images of typical deformed specimens in different original height after conventional static and **ultrasonic assisted compression** were **composed of strain induced martensitic phase and austenitic phase.**The average grain size decrease to 1,5 μm, which brings in improvement of the deformation uniformity and the production quality in the metal forming processes. Moreover, in order to achieve a diffusion bonding under the low stress, **surface roughness** was also improved. **The integrated approach can not only improve formability of the work material, but also accuracy of product.**

From the previous research there are lack research in surface roughening behavior in various thin metal foils with body central cubic (BCC) and Face Centered Cubic (FCC) structure with different grain size below 10μm and their phase transformation.

The purpose of these research are first is to investigate how the martensitic transformation affect to the surface roughening behavior. The second purpose is to investigate the effect of very small grain with 0,5 μm in size to the surface roughening behavior compared to the large grain with 9μm or more in size.

2. Materials and Research Methode

2.1. Materials

Thin metal foils of commercial SUS 304 and 316 with width 4mm, thickness 1mm and gauge length 20 mm according to DIN 50125. The sample made in dog bone type according to the fig.1.

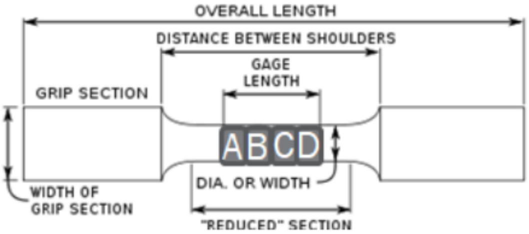


Fig.1. Specimen of Tensile Test

2.2.Methode

Before sample tensiled, sample cleaned using ethanol combined with ultrasonic vibration to increase the cleaning of surface. Sample tensiled until five steps with constant strain. After sample tensiled, surface roughness measured using confocal laser microscope. Tensile test using Shimadzu tensile machine with capacity 50 KN.

2.2.1. Uniaxial Tensile Test

Surface roughening behavior deformation of stainless steel 304 and 316 with various grain size investigated using uniaxial tensile test. Uniaxial tensile test was conducted step by step using constant strain over the yield point of thin metal foils. The gauge length of thin metal foil is 20 mm. width of thin foil is 4mm and 0,1 mm thickness. The used of fillet radius is 20 mm. In order for consistency, The uniaxial tensile test machine using a commercial tensile test machine of Autograph AG-IS 50 KN (Shimadzu Corporation). The strain rate of uniaxial tensile test is

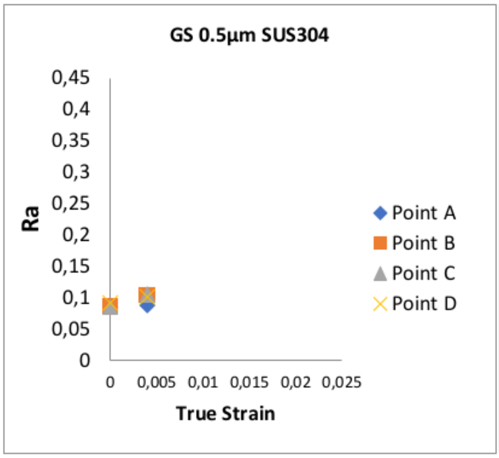
1,6 x 10^{-3} s^{-1}. The surface roughness behavior , stress strain curve were measured and observed for different grain size, different materials and constant thickness. The elongation was measured optically with video noncontact extensiometer (DVE-201, Shimadzu Corp), because of contact extensiometer could not be pasted onto the metal foil. Surface roughness during deformation was measured using uniaxial tensile testing machine that halted for every step. In universal tensile testing machine the sample tensiled For each step, the tensile test was halted then the specimen was taken out from the chuck for measuring surface roughness behavior using Keyence Confocal Laser Microscope (VE 8800, Keyence Co). The area of surface roughness measurement in the rolling direction at each step using contact surface roughness measurement. The surface roughness measured until five step in the same position with constant strain. beside surface roughness, the Rz value was evaluated. The area of surface roughness measurement in the length of 0,7 mm.

Fig.2. laser Confocal Microscope

Fig.3. Shimadzu Tensile Machine With Capacity 50 KN

3. Result and Discussion

3.1.Surface Roughening Behavior in Grain Size (GS) 0,5µm and 1µm



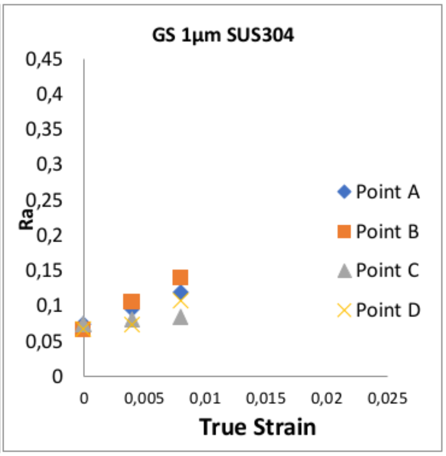


Fig.4. Ra-True Strain GS 0,5µm

Fig.5.Ra-True Strain GS 1µm

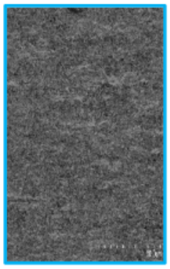
For GS 0,5 µm and GS 1µm tensiled with 0,4% strain. for GS 0,5µm only with one step tensile test, the sample fracture. For GS 1µm the sample fracture after second step of tensile test. The increasing roughness is very small, because of martensitic transformation occurred in the grain. The martensitic transformation occur as shown in fig 6. From the SEM EBSD result. Material with high volume of martensitic phase become more difficult to deform. The increasing martensitic phase cause surface roughening more difficult to occur.

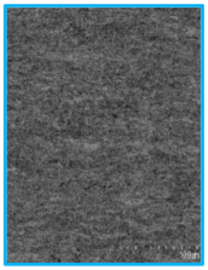
Localized deformation at fracture region occur when grain number decreased. When the grains are constrained and hampered among the grains will cause cross slip easier to form and also shear deformation and coordination much more difficult because of the different grain orientation in the cross section. With the increasing of foil thickness, the grain number and grain boundaries increase. Dislocation movement will be obstacle by the grain boundary. Thus the increase of grain boundary number will increase the obstacle of dislocation movement. The shear stress concentrates at the grain boundary regions and causes the parabolic dimple formation during micro tensile process. During tensile test, parabolic dimple formation occur because of shear stress concentrates at the grain boundary. The increasing number of grain or increasing foil thickness cause increasing dimples in microstructure of thin metal foils.(53).

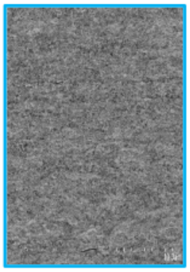
3.2. SEM Investigation in Various Grain Size

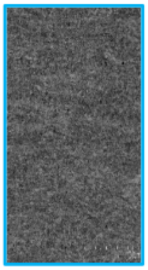
3.2.1. Grain Size (GS) 0,5µm.


SEM Before Tensile GS 0,5


SEM at Point A After

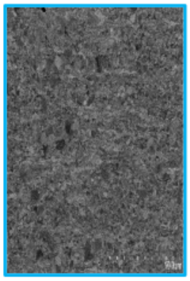

SEM at Point B After

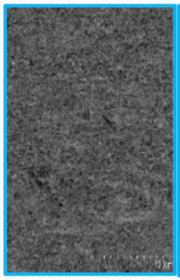

SEM at Point C after

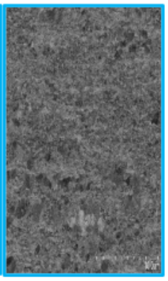

SEM at Point D after

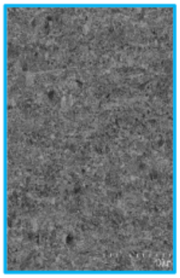
After tensile test, there a lot of dimple pattern in SUS 304 GS 0,5. The fracture deformation mechanism caused dominating by dimple pattern more than slip separation that caused by slip band movement. Dimple pattern in GS 0,5 is smoother than GS 1,0µm.

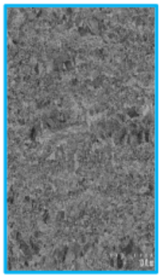
3.2.2. Grain Size (GS) 1,0µm


Before Tensile


After A


After B

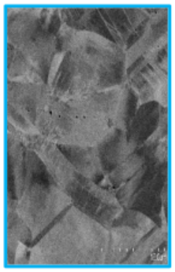

After C

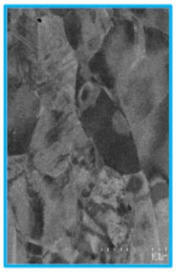

After D

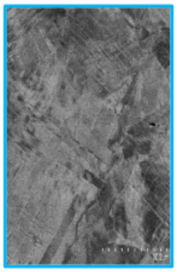
The dimple pattern structure in GS 1 is smoother than GS 0,5. Grain surface boundary in GS 0,5 is higher in quantity than GS 1, thus Dislocation obstacle in GS 0,5 is higher than GS 1. This condition caused thin foils with GS 0,5 is stronger than GS 1.

3.2.3. Grain Size (GS) 9µm


Before Tensile


After A

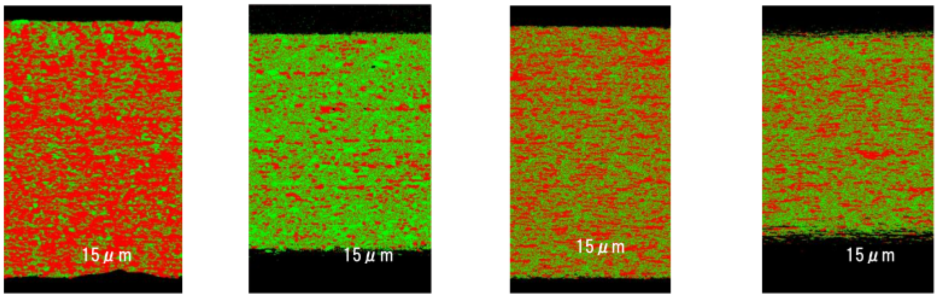

After B


After C

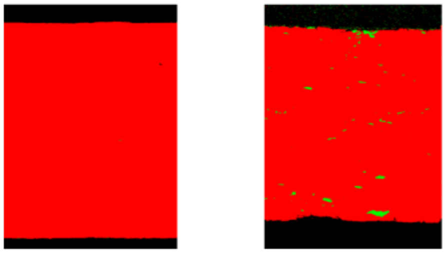

After D

No dimple pattern occurred in sample with GS 9 on SEM investigation before and after tensile test. With the same thickness, the quantity of grain is decreased in the same area with same constant deformation. The share of surface grain is increased, which result in the decreasing flow stress (51). Slip band easier to move and dislocation density increased and the roughness easier to form than thin foils SUS 304 with GS lower than 9µm.

3.3. Phase Transformation Behavior



GS 1µm Before Tensile Test GS 1µm After Tensile Test GS 0,5 µm Before Tensile Test GS 0,5µm After Tensile Test



GS 9 µm Before Tensile Test GS 9 µm After Tensile Test

Fig.6. Martensitic Transformation in GS 0,5µm, 1µm and 9µm.

The red color is gamma (γ) iron. The green color is alpha (α) iron. From the SEM –EBSD investigation the volume of gamma iron decreased dramatically in GS 1µm because of tensile until fractured at second step. The martensitic phase transformed dramatically because of plastic deformation bigger than GS 0,5 and GS 9 with same strain at 0,4%. Grain deformation at larger grain size is bigger than smaller grain size. From the SEM-EBSD showed that at GS 9 the grain deformation occur at all area. The martensitic transformation occur only in one grain. At GS 0,5 and GS 1 the plastic deformation occur at many grain since RZ > GS or RZ = GS. The plastic deformation caused grain deformation and phase transformation. The phase transformation occur optimum at GS 1 after second step until fracture. The phase transformation not occurred optimum at GS 0,5 because at GS 0,5 fractured after one time of tensile test state. The martensitic phase not yet transformed optimum until fracture.

The surface roughness increased linearly with increased grain size and increased exponential on the texture distribution. (50). Flow stress decreases with the increasing of grain size, which can be explained with the surface grain weakening model. Flow stress increases with the decreasing of grain size, which can be explained with the surface grain weakening model (51-52). According to the surface layer theory, with the increase of grain size while keeping the foil thickness constant, the share of surface grain increases, which result in the decreasing of flow stress.

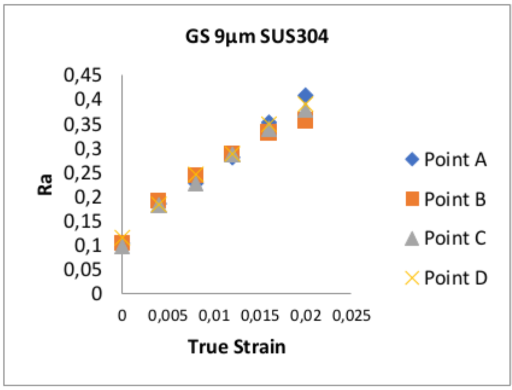


Fig.6. Ra-True Strain GS 9µm

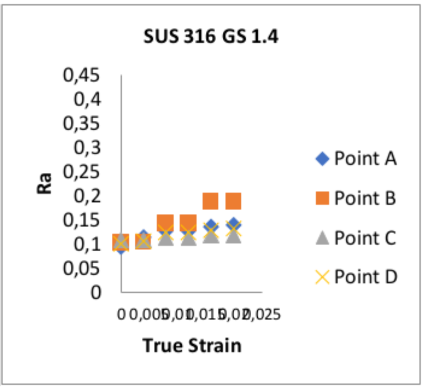


Fig.7.Ra-True Strain GS 1,4µm

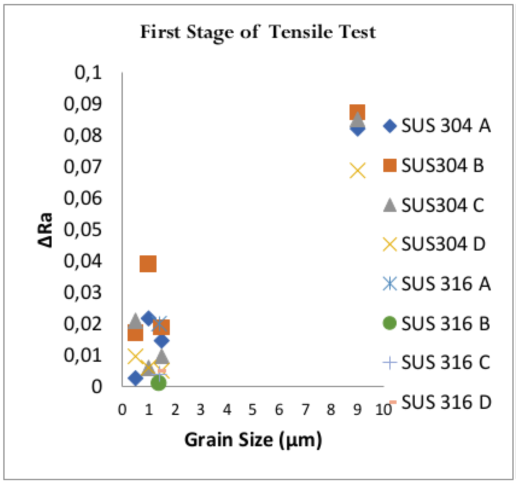


Fig.8.Δ Ra-Grain Size (µm)

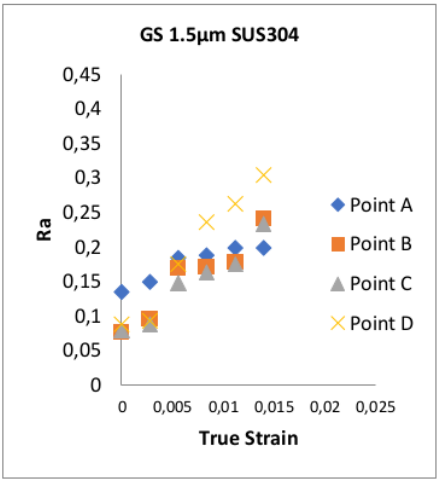


Fig.9. Ra-True Strain GS 1,5µm

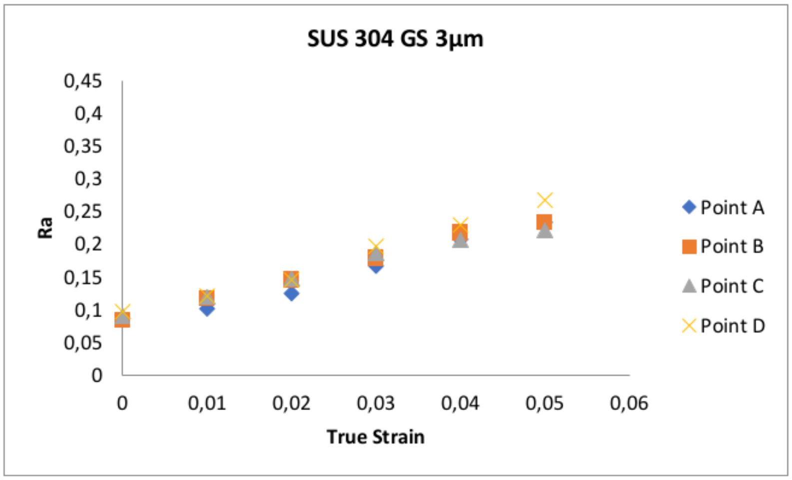


Fig.10. Ra-True Strain

Grain deformation in larger grain size (GS 9μm) is bigger than in smaller grain size (GS 3;1,5;1,0 & 0,5μm), thus surface roughness in larger grain size is higher than in smaller grain size with same strain. Ra behavior for SUS 316 GS 1,4 is lower than GS 1 SUS 304 with same strain, also lower than SUS 304 GS 1,5 even in lower strain, because of complicated phase in SUS 304. The increasing Ra in GS 3 in every point is higher than GS below 3µm. The scaterring data at every point of investigation is smaller than GS below 3μm. This means the grain deformation is bigger than GS below 3μm, thus Ra obtained nearly the same at all area of investigation during tensile test and the slope is higher than GS lower of 3μm.

Grain misorientation and crystal misorientation in GS 9 is higher then the other samples. Beside that, the complicated phases have an important role in showing highest Ra. There are possibilities in martensitic transformation in all SUS 304 samples.

The increasing Ra of SUS 316 is lowest than all at first stage but the scaterring is highest at first stage, because of highest volume of austenitic phase than all.

Scaterring Value at first stage : GS 0,5 = 7,87;GS 1=6,5; GS 1,4 =20; GS 1,5=5,11334; GS 9 = 1,269346. At first stage GS 1,4 has highest scaterring than all, but GS 9 has highest increasing Ra. As shown in fig.8.

For initial roughness Rz0 < Dg (Dg=9μm) The roughness (Ra) increase with increase of plastic strain (εp), as shown in Fig.6. The grain deformation occurred uniform in thin metal foils with GS 9 μm. The martensitic phase transformation occurred in small area. Thus, grain easier to deform in larger grain size. Small martensitic transformation and uniform grain deformation caused surface roughness increased obviously. The martensitic transformation shown in figure 23 and uniform grain deformation shown in fig 23,24,25. For Rz0 = GS (GS = 1μm) or Rz0 > GS, The Ra not increase proportionally with plastic strain (εp). When Rz0 = Grain Size (GS) or when Rz0 > GS, grain deformation not occurred uniformly as shown in fig 13,19 and also there are a lot of martensitic transformation obviously in the grain as shown in fig 12,18. Ra in GS 1,5 SUS 304 with 0,28% strain is higher because of complicated phase consisted of martensitic, austenitic and ferritic phase than GS 1,4 at 0,4% strain because of Rich austenitic phase as dominating phase. Because of plastic deformation, austenitic phase will transform to become martensitic phase. The increased volume of martensitic phase in the grain will cause grain more rigid and strength thus grain more difficult to deform. The more difficulties on grain deformation will cause more difficulties on increasing surface roughness because of high plastic deformation.

4. Conclusion

For large grain size (GS = 9μm), Ra increased obviously. For smaller grain size (GS=1 and GS=0,5) Ra not increased obviously.

For initial roughness Rz0 < Dg (Dg=9µm) The roughness (Ra) increase with increase of plastic strain (εp). For Rz0 = Dg (Dg = 1µm) or Rz>Dg (Dg = 0,5), The Ra not increase proportionally with plastic strain (εp).

In GS 9µm grain deformation occurred uniform in all point of grain. Thus the surface roughness (Ra) increased significantly.

In GS 1 the martensitic phase transformation occurred significantly . this condition caused the surface roughness is significantly lower than GS 9.

In GS 3, the slope is higher than GS below 3 and also the Ra increased higher than GS below 3. The scaterring is lowest than metal thin foils below than 3.

In SUS 316 showed the Ra value below than SUS 304 even in the same grain size because of rich austenitic phase. The toughness of austenitic is higher than martensitic and ferrite thus surface roughness more difficult to occur.

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