Effects of grain size and specimen size on small punch test of type 316L austenitic stainless steel

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Abstract

Miniature specimen test technique has been extensively studied for quantifying the properties of bulk materials. In this paper small punch test (SPT) is used to clarify the effects of specimen thickness (t), grain size (d) and ratio of thickness to grain size (t/d) on mechanical properties of 316L austenitic stainless steel (SS). Five sheet of 316L SS with the same texture but different thicknesses and grain sizes were prepared using rolling and heating treatment technique. Effective SPT yield strength was measured, and then used to correlate with conventional tensile test by empirical equation. The results show that the SPT is sensitive not only to differences in the thickness, but also to changes in the grain size and value of t/d. The present work provides information that enhance the understanding of reliability of SPT in analysis of the mechanical properties of small specimens and bulk materials.

1. Introduction

It is well-known that the strength of polycrystalline metals depends on grain size (GS) (e.g., Hall-Petch relation) [1, 2], though the opposite relationship is found in the nanometer grain scale [3]. Recently, many investigations of miniature, sub-millimeter and sub-micron specimens have been performed to study the relationship between grain size, specimen size, shape and the size and different mechanical properties [4-7]. Despite much effort to extract the characterization of mechanical behavior of materials by using greatly reduced volume of materials, the effect of size on mechanical properties at different scales are still unclear. Miniature specimen test techniques have potential to be used at present in life prediction of in-service components and creep test at elevated temperature. This is especially valid for the nuclear industry where larger specimens are highly radioactive [8]. Small punch test (SPT) is one of the most promising miniature specimen test techniques, which was originally applied in testing irradiated materials [9-12] and then introduced to other fields to evaluate the local mechanical properties that are difficult to measure using the conventional test. For example the properties of the welds in HAZ steel, high Cr ferritic steel, P92, and other engineering materials [13] can be measured this way. At present, there are however some reports that contradict each other and it is possible

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that differences in microstructure of materials are responsible for such differences. For example, Toloczko et al. [14] used miniature shear punch test to measure the yield strength and ultimate strength of SS316L and did not observed any dependence of strength on the thickness and grain size of specimens, based on which they concluded that shear punch test did not have the same sensitivity to specimen thicknesses and grain sizes as the miniature tensile test. Chen et al. [7] reported that micron-sized Ag wire exhibited significant dependence of strength on grain size and specimen size in the micro tensile test. The similar results are also reported by Miyahara [15] and Igata [16] in the tensile test of foil specimen of steels. It is well-known that in addition to the grain size, the specimen size may also have important influence on strength of polycrystalline materials. So far, very few studies have been made on this topic and the correlation between specimen thickness, grain size, specimen size and mechanical properties in SPT is not reported in the literature. This paper will use type 316L austenitic stainless steel (SS 316L) as a model sample to investigate the biaxial deformation behavior for different grain sizes and specimen thicknesses and study their effects on SPT.

2. Experimental procedure

The material used in this study is commercial 316L stainless steel sheet with 1.5 mm thickness supplied by Parker Steel Company. Its chemical composition is given in Table 1. For obtaining the specimens with different grain sizes and thicknesses, the SS316L sheets were rolled until the thickness reached 0.60±0.02 mm, then solution treatment of specimens were carried out at various temperatures between 1223 and 1473 K to obtain different grain sizes. Two different cross sections; one normal to rolling direction and the other perpendicular to rolling direction were polished and chemically etched to measure the grain sizes of the as-received and annealed specimens. Grain sizes were measured using optical microscopic (OM) photos and standard linear interception method (LI) [17].

For preparing small punch test specimens, several disk-type specimens with 10 mm in diameter were cut from the annealing sheets using electric discharge machine (EDM), and then both surfaces of the disk specimens (thickness 0.60±0.02 mm) were ground on silicon carbide papers up to 1200 grit and get different thicknesses of 500, 400, 300, 200 and 100 μm. The bulk texture measurements of samples with different grain-size were carried out by the Bruker D8 DISCOVER with GADDS XRD software. The SPT was conducted on a servo-hydraulic system with central load sets which include a punch, a quenched steel ball and upper & lower dies used to hold the specimen. The detailed SP testing configuration is shown in Fig. 2. The diameter of the ball (d) is 2.5 mm and the diameter of the hole in lower die (a) is 4 mm, the radius of the chamber of lower die (R) is 0.5 mm. In the process of SPT, a load with a constant deflection rate of 0.005 mm/s was applied to the center of the specimens through the quenched steel ball without lubrication. The punch load and central deflection of the specimens were monitored and automatically collected during the test until the final failure. The SPT was repeated at least three times for every same condition specimen. The results obtained are given in Table 2. All the tests were performed at room temperature in the air.

3. Results and discussion

The typical SPT load/displacement curves were shown in the Fig. 3. It is seen that for the specimens with the same grain size, the thicker samples have the higher strength; also for the same thickness, the specimens with smaller grain size have higher strength, obeying the common mechanical law applicable in polycrystalline solids. Conventional tensile test could be related to SPT by [18]

$$R_{0.2(SPT)} = 239.44\times10^{-3} P_y + 55.26 \qquad (R^2 = 0.9755) \tag{1}$$

where $R_{0.2\,(\mathrm{SPT})}$ is the yield strength in conventional tensile test (CTT), P_{y} is the yield load in the SPT. The equation (1) was proposed by Huang as a practical and accurate relationship between SPT and CTT based on the data obtained by testing many isotropic ductile materials [18]. Fig. 4 shows that the Hall-Petch relation $\sigma_{0.2\%} = \sigma_0 + kd^{-1/2}$ is basically obeyed in the observed grain-size range. For the grain size of ~50μm, the strength seems to be higher than that expected using Hall-Petch relation. This is reasonable. When the grains in the deformed area decrease, the sources of dislocation will reduce, so that the strength of material will be higher than that of material with more grains.

The textures of specimens with different grain sizes were shown in Fig. 5. It is observed that all the specimens have nearly the same texture. The rolling and annealing conditions that are proposed in this paper are effective to produce the specimens with different grain sizes, but the same texture. The volume fractions of different textures, as illustrated in Fig. 6, further confirmed that the samples with different grain size ranging from 10 μm to 50 μm have nearly the same texture. The effect of texture on changes in the strength observed in SPT test may be neglected, and the thickness, grain size and sample geometry (i.e., ratio of the thickness to the grain size) are main factors affecting the mechanical properties in this study. The SPT load/displacement curves, as given in Fig. 4, show that the SPT is very sensitive to changes in the thickness and the grain size for austenitic stainless steel. The contribution of the change in the strength ($\sigma_{0.2\%}$) comes from three aspects: specimen thickness (σ_t), grain size (σ_{gs}) and the ratio of the thickness to the grain size (σ_R). The $\sigma_{0.2\%}$ may be written as

$$\sigma_{0.2\%} = \sigma_t + \sigma_R + kd^{-1/2} \tag{2}$$

The strength dependence on the grain-size, in the form of $kd^{-1/2}$, results from the Hall-Petch relationship.

Figs. 4 and 7 show that in addition to this dependence, the strength is also related to the thickness and the ratio of thickness to the grain size. For the small grain-size range, the dependence of strength on the grain size follows the Hall-Petch relationship with slope of 1088 MPa (μm)$^{1/2}$. To use Eq. (1) to relate the present results with the data obtained from the conventional test, the change of $\sigma_{0.2\%} - kd^{-1/2}$ with $d^{-1/2}$ is plotted in Fig. 7 (a). To remove the effects of σ_t and σ_{gs}, the change of $\sigma_{0.2\%} - kd^{-1/2} - \sigma_t$ with t/d is plotted in Fig. 7 (b). It is seen that the increase in t/d cause the substantial decrease in $\sigma_{0.2\%} - kd^{-1/2} - \sigma_t$, however, very interestingly, when $t/d > 17$, the value of $\sigma_{0.2\%} - kd^{-1/2} - \sigma_t$ tends to be constant. The specimen thickness change in the range between 100 μm and 500 μm is responsible for about 25 MPa change in the value of σ_t, while the change in the specimen shape (*t/d*) cause a change of about 300 MPa in the same range. This shows that the factor of shape is important in determining by SPT the strength of materials. The reason for this might be the fact that increase of the grain size and decrease of the thickness reduces the number of grains in the through-thickness direction. For example, the sample with 50 μm grain size and 100 μm thickness will have only 2-3 grains in the through-thickness section. This may restrain the plastic deformation. It is known that the strength of polycrystalline materials is significantly affected by the number of operating slip systems [19]. However, for small thickness specimens the number of slip systems is likely to be limited, and therefore more difficult. Therefore, the increase in *t/d* has a significant influence on the measurement obtained by SPT.

4. Conclusions

The specimens with the same texture and different thicknesses, grain sizes and shapes were prepared and their effects on the mechanical properties of type 316L SS were studied using SPT. The results show that in addition to the grain size, specimen size can affect the estimate of strength by SPT. This becomes significant for the large grain size and small specimen thickness. This work allows us to analyze the accuracy of the results of SPT measurement and results obtained can contribute to development of corrections for the specimens with different thickness, grain size and texture.

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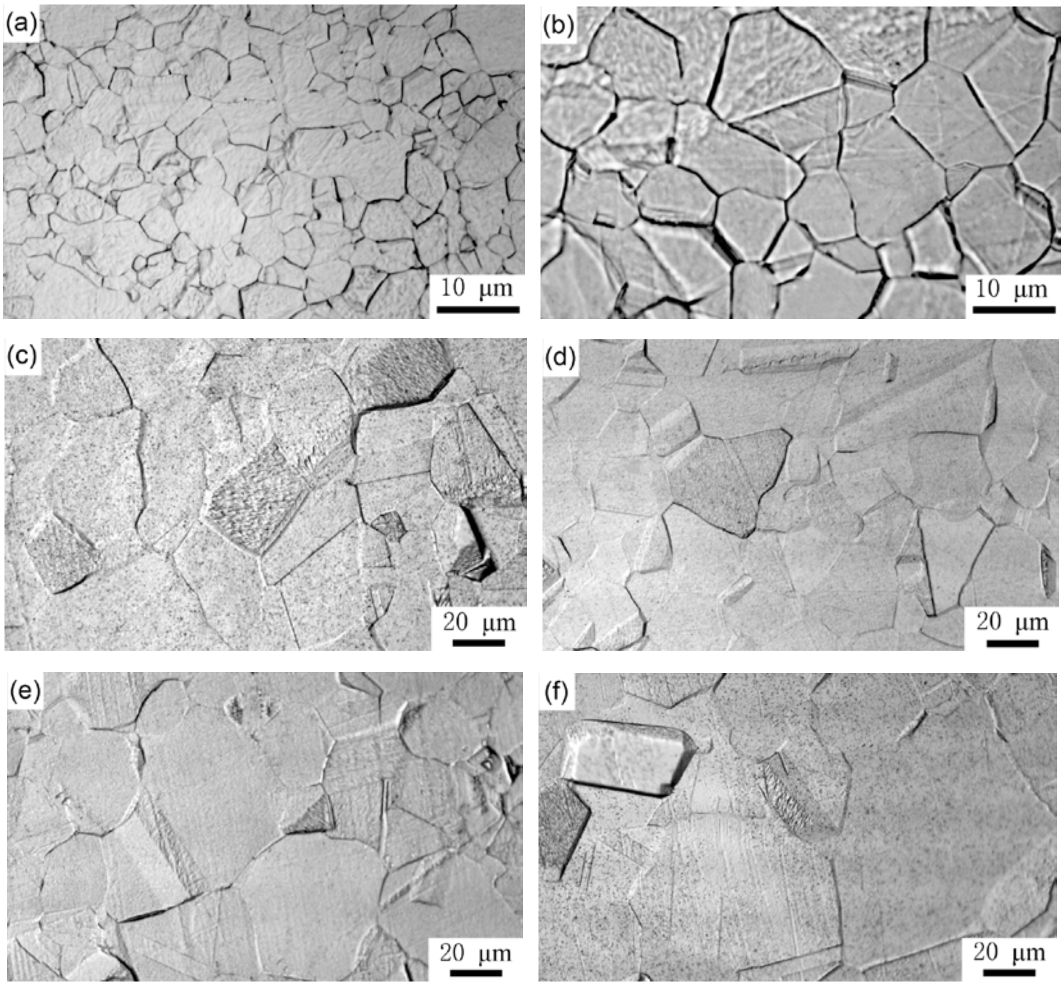


Fig. 1 Metallographs obtained by OM showing the polycrystalline structure in (a) as-received SS316L and SS316L specimens solution-annealed at (b) 950 ℃for 1 hour, (c) 1050 ℃ for 1 hour, (d) 1050 ℃ for 2 hours, (e) 1100 ℃ for 1.5 hours and (f) 1200 ℃for 2 hours.

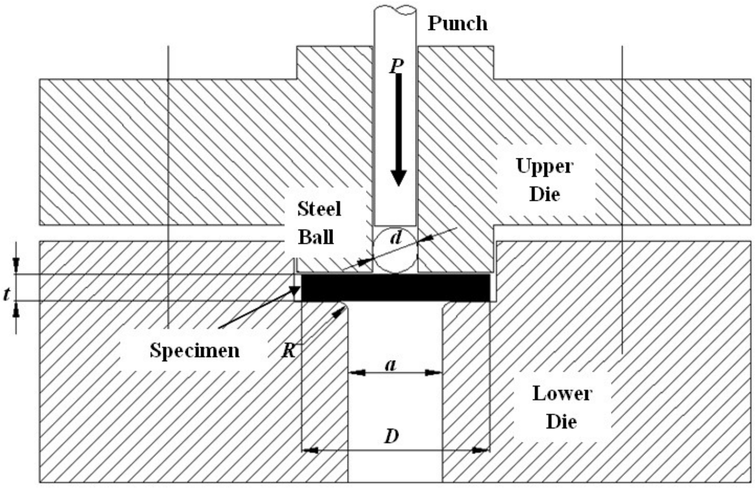
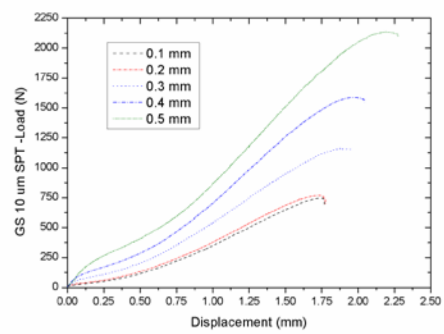


Fig. 2. Schematic representation of SP test configuration

(a)



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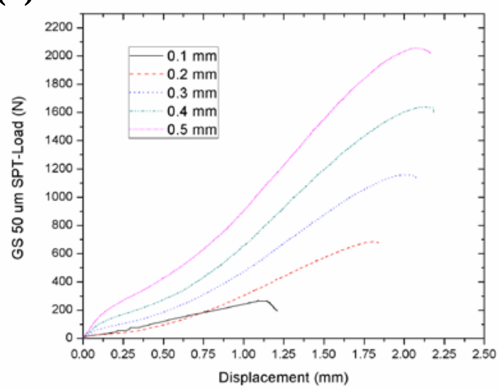

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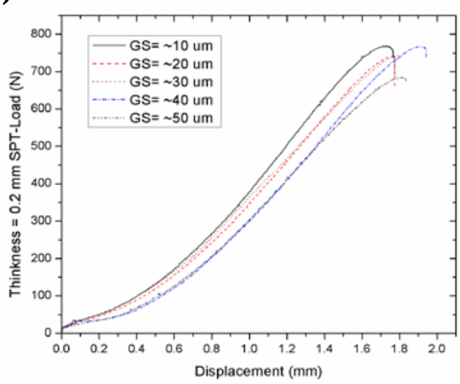


Fig. 3. Typical SPT load-displacement curve on (a) sample thicknesses differ from 0.1 mm to 0.5 mm with 10 µm GS. (b) sample thicknesses differ from 0.1 mm to 0.5 mm with 50µm GS and (c) samples with same thickness but varying GS respectively.

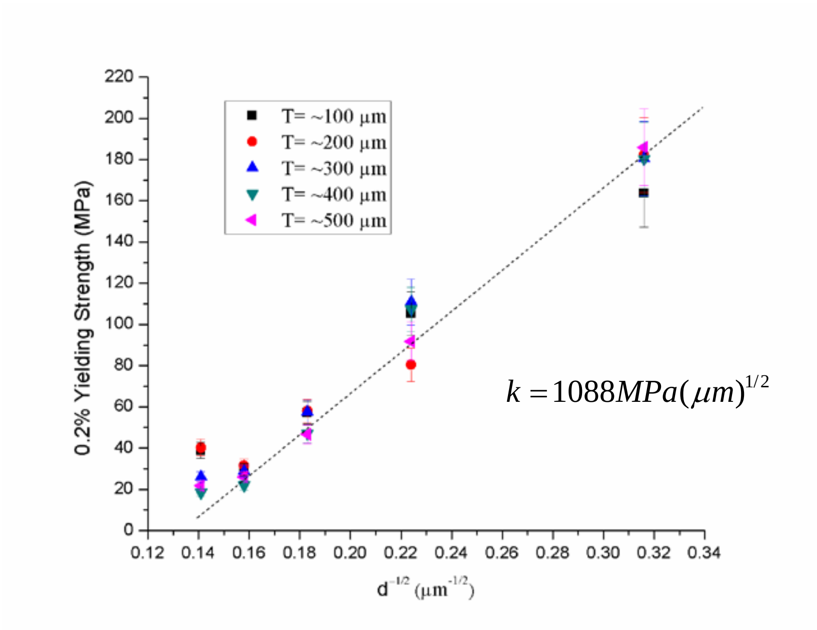
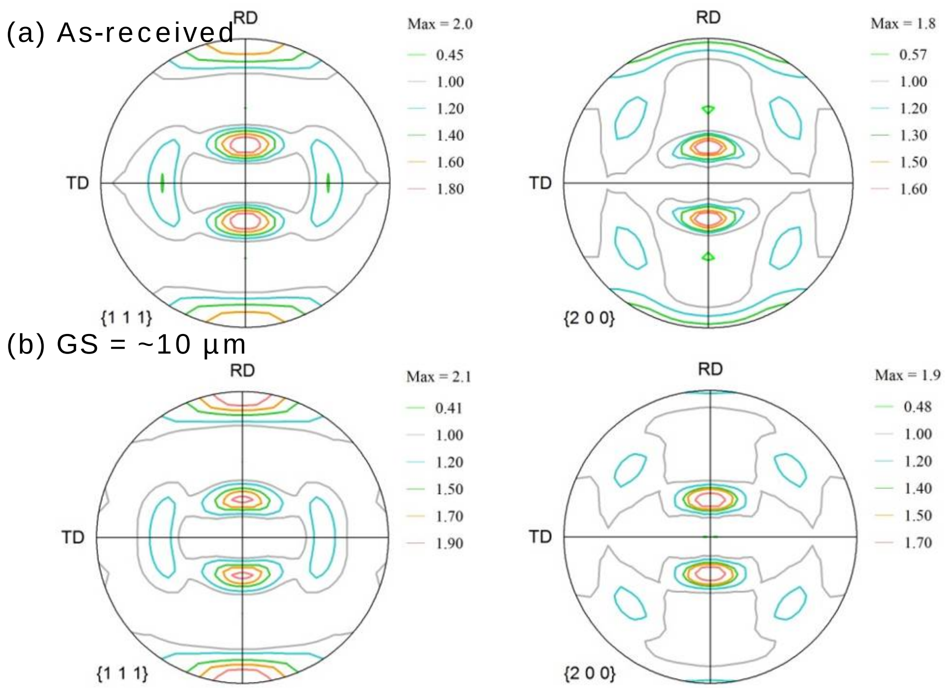


Fig. 4. Hall-Petch relationship applied to the 0.2% yield strength



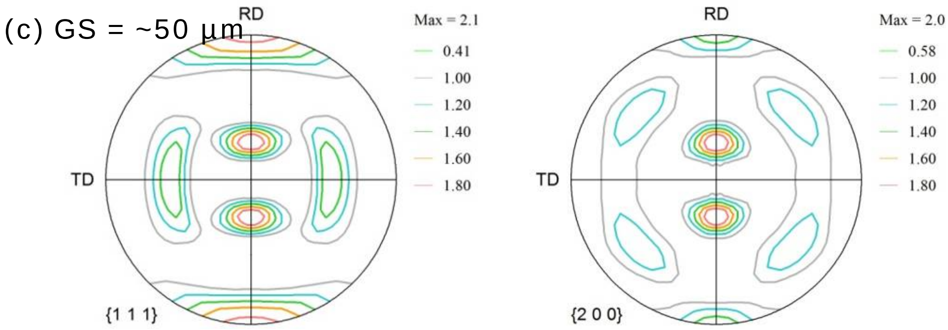


Fig. 5. Pole figures of the (a) as-received, (b) grain size is around 10 µm and (c) 50 µm 316L austenitic stainless steel samples

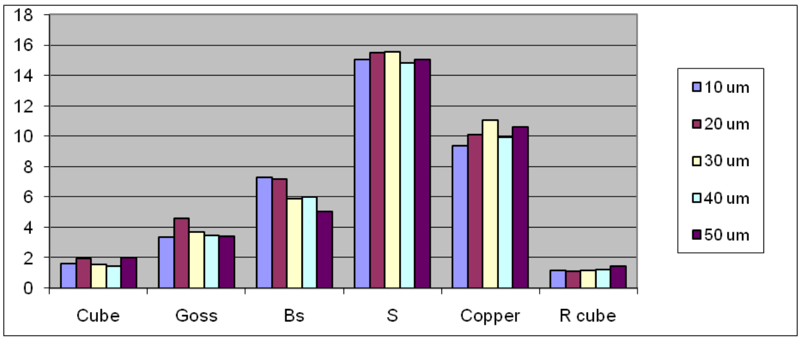
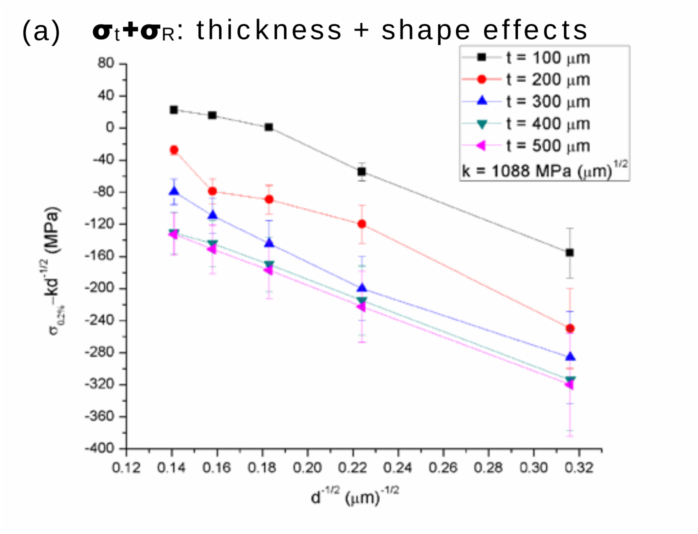


Fig. 6. Different sorts of texture volume fractions comparison between samples with different GS



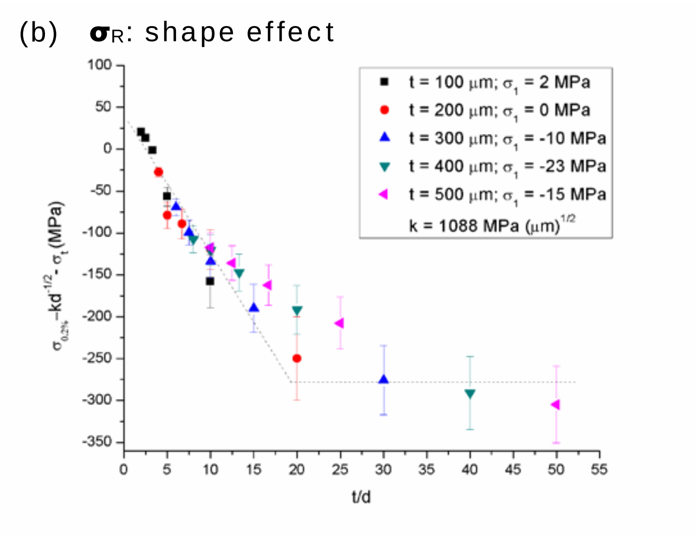


Fig. 7. Data analysis of strength according to equation (2). (a) curves of $\sigma_{0.2\%} - kd^{-1/2}$ vs. $d^{-1/2}$. (b) curves of $\sigma_{0.2\%} - kd^{-1/2} - \sigma_t$ vs. t/d, where the σ_t values are chose to make all the data collapse into a narrow band as far as possible.

Table 1
Chemical composition of type 316L stainless steel (wt %)

C	Mn	P	S	Si	Ni	Cr	N	Mo
.03 max	2.0 max	.045 max	.03 max	1.0 max	10.0-14.0	16.0-18.0	.10 max	2.0-3.0

Table 2
Thickness, grain size of SS316L specimens and number of tests for each condition

Solution treatment temperature (K)	As-received	1223	1323	1323	1373	1473
Holding time in furnace (hr)	Nil	1.0	1.0	2.0	1.5	2.0
Grain size (μm)	5.2±1.5	10.5±2.0	24±4.0	32.5±3.0	42±3.0	65±6.0
Specimen thickness	Number of valid tests					
100±20 μm	3	3	3	3	3	3
200±20 μm	3	3	3	3	3	3
300±20 μm	3	3	3	3	3	3
400±20 μm	3	3	3	3	3	3
500±20 μm	3	3	3	3	3	3

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