Mechanical properties and meso-damage evolution behavior of SUS 304 austenitic stainless steel

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Abstract. Using MTS-Landmark material testing machine, Quanta-650 scanning electronic microscopy and Leica-LM/DM optical properties microscope, the mechanical properties and meso-damage evolution behavior of AISI304 steel at different annealing temperatures were studied. The results showed that the grain of AISI 304 steel grew up with increasing annealing temperature, and annealing twins appeared during the grain growth. The yield strength, tensile strength and micro-hardness of the samples decreased gradually with increasing annealing temperature. Based on the evolution characteristics of austenite grain boundaries, evolution of shape factor of the samples was researched at different plastic strains. Meso-damage evolution equations of the materials were established, and the material constant in the equation were closely related to the annealing temperatures. Normalized shape factor can reflect meso-damage evolution behavior of AISI 304 steel, which simply and effectively revealed the relation between marco deformation and microstructure.

1. Introduction
Damage and fracture of metal materials is closely related to microstructure. For most metals, the fracture undergoes the process of nucleation, growth and coalescence of voids. Many researchers had studied damage evolution at the macro, meso, and micro scales. McClintock [1] put forward a criterion by the growth of voids for the ductile fracture. Uniaxial shear tests of plastic materials under hydrostatic pressure were carried out. The hole is closed under shear, and eccentric under tension. The shear band fracture criterion based on the combination of adjacent voids shows that the fracture strain decreases exponentially with plastic tension. Rice and Tracey [2] studied the enlargement behavior of voids in triaxial stress fields. The cavity in the incompressible plastic material is described by the variational principle. Then an approximate Rayleigh Ritz method is developed and applied to the propagation of isolated spherical cavities in non hardening materials. Kameda [3] proposed a microscopic constitutive equation related to void growth. At the micro and meso scales, microstructure (such as recovery recrystallization, microvoid and grain) accumulation led to the fracture of metallic materials. Bate [4] found that the effect of internal grain size had effect on the deformation behavior of metal materials. During the plastic deformation of metal materials, the grains were stretched, rotated and recrystallized. when the plastic deformation reached a critical value, microvoids preferentially
nucleated around the inclusion particles. Numerous studies had shown that microvoids were accurate and feasible to characterize the damage evolution of materials [5-6]. However, it is not easy to calculate the percentage of the hole area in each transient and instability state.

Grain boundaries are an important part of crystals, which had a significant effect on the damage mechanism of materials. It is significant to study the damage physics of materials by using grain boundaries. Based on grain boundary precipitation kinetics and continuous creep damage mechanics (CDM), Yin [7] simulated the microstructure evolution and creep behavior of metal in power plant. Shi [8] developed a damage variable of grain boundaries length and set up constitutive equations to describe Macro stress-strain relationship for ductile metal materials. In this paper, the plastic strain evolution equation of AISI304 steel ductile metal material under tensile load was established by quantitative metallographic analysis.

2. Experimental

The material data of AISI 304 stainless steel used in this study were listed in Table 1.

| Table 1. Chemical composition of 304 stainless steel. |
|----------------|-------|-------|-------|-------|-------|-------|
| C              | Si    | Mn    | Cr    | Ni    | S     | P     |
| 0.07           | 1     | 2     | 18    | 8     | 0.03  | 0.04  |

The material was cut into tensile samples by wire cutting method, and the sample size was shown in Figure 1. During cutting, water was injected to cool the material to prevent the change of microstructure. Prior to the experiment, four temperatures (450℃, 650℃, 950℃ and 1050℃) were specified to anneal the samples in the annealing furnace (OTF-1200X) and then hold for 60 minutes. The main reason for choosing heat preservation for 60 minutes was that 304 stainless steel was used in the main process of automobile exhaust pipe. The sample was cooled to room temperature in the furnace. Uniaxial tensile tests were carried out on annealed specimens using a testing machine (MTS-Landmark). Nine levels of deformation had been specified to measure the length of grain boundaries. The sampling position of metallographic specimen was not arbitrary, so its representativeness should be considered. For each unloading sample of different numbers (including the original sample without deformation), about 20 mm sample was cut from the middle part (for necking sample, the minimum section of necking was taken as the symmetry plane), and the water injection cutting was carried out for the 20 mm sample along the axial central symmetry plane with the thickness of 1 mm. The metallography obtained from the polished surface was the inner surface of the material. The fracture specimen was sampled near the fracture surface, and attention should be paid not to make the small sample produce additional plastic deformation. The microstructure of 304 stainless steel with different plastic deformation states was observed by Leica DMLM optical microscope. The grain boundary images were obtained by cutting along the axial central plane of the material, but not on the surface of the sample. From the stereological point of view, the grain boundary distribution obtained by splitting the material in any parallel plane was consistent. When the grain boundary was cut in any direction, the evolution of grain boundary was similar from the statistical point of view. The experimental results of internal grain boundary and parallel surface grain boundary were basically consistent. During the stages of yield, strengthening and necking, the unloading specimen intercepted some regions to evaluate the damage evolution and carry out microhardness test. AHVD-1000XY semi-automatic digital microhardness tester was used to test the microhardness of 304 stainless steel sample. The indenter was a regular pyramid diamond indenter. The experimental parameters were as follows: the loading load was 200g and the holding time was 15s. 15 test points were selected for each sample. The fracture morphology was analyzed by SEM (FEI Quanta 650) to obtain the fracture images.
3. Definition of damage variable

In order to quantitatively describe the deformation evolution of the grain boundary in the microstructure and metal structure of 304 stainless steel [9-10], the shape factor of grain boundary was defined to describe the change characteristics of the interface microstructure. 10 metallographic photos were taken in randomly selected regions, and 10 fields of view at different positions were randomly selected for each metallographic photo to measure grain boundary length, and the length of austenitic grain boundaries in 10 different fields of view was averaged. The definition of grain boundary shape factor was given as follows:

$$\varphi = \frac{\sum L}{\sum A}$$  \hspace{1cm} (1)

Where L (mm) was the total length of austenite grain boundary on the plane section of any deformation state (including cracks and voids), and A (mm$^2$) was the area of any microscopic field of view. The grain boundary produced by martensitic transformation was ignored here.

The microscopic field area in any plastic deformation state is equal to that in the initial undeformed state in Figure 2. Therefore, when the area of the crystal shape factor was $A$, the ratio of the perimeter of the grain boundary and the circumference of the undeformed state in any deformation state was expressed, that was, the change of the unit grain boundary length. The larger the shape factor of grain boundary was, the greater the degree of plastic deformation was. When the number of field of view measured had enough statistical significance, the shape factor can reflect the change state of the internal microstructure of the material, that is, it was reasonable and feasible to use the grain shape factor to quantitatively describe the statistical evolution law and change characteristics of the internal interface shape. The damage variable had the characteristics of clear concept, easy to measure and sensitive to reflect.

The shape factor of grain boundary increased with the increase of plastic deformation. Therefore, relative shape factor $\psi$ can be used to further describe the relative change of shape factor of AISI 304 steel under certain damage state. The relative shape factor can change the shape factor more obviously at different annealing temperature. The relative shape factor $\psi$ can be expressed by the following equation:
\[ \psi = \frac{\varphi - \varphi_0}{\varphi_0} \]  

(2)

Where \( \varphi \) were shape factors at a certain moment under external loading, \( \varphi_0 \) were shape factors for as-received materials.

In order to realize the change of damage variable from zero to one and characterize the damage evolution of materials, the normalized shape factor formula was established as follows:

\[ D = 1 - \frac{\varphi_f - \varphi}{\varphi_f} \]  

(3)

Where the normalized shape factor \( D \) can reflect the damage variable of grain boundary damage evolution from the beginning of plastic deformation to fracture, and \( \varphi_f \) was shape factors at the moment when the materials fractured.

(1) If \( D = 0 \), the material is in a non-destructive state.
(2) If \( D = 1 \), the material failure and fracture.
(3) If \( 0 < D < 1 \), the material is just located in a plastic deformation state.

4. Results

4.1. Mechanical properties

The stress-strain response of the material had been achieved as shown in Figure 3. With the increase of annealing temperature, the plastic properties were improved. When the annealing temperature was 950 °C, the plasticity decreased due to the increase of martensite in the microstructure. With the increase of martensite, the toughness of 304 steel decreased, but the brittleness was improved.

![Figure 3](image-url)  

**Figure 3.** The stress-strain response of the materials.

![Figure 4](image-url)  

**Figure 4.** Mechanical properties at different annealing temperatures.

Mechanical properties at different annealing temperatures were shown in Figure 4. With the increase of annealing temperature, the tensile strength and yield strength of the samples decreased gradually, while the elongation increased gradually. However, after annealing at 1050°C, the elongation of the material decreased, which was related to the grain size of the materials [11].

The micro-hardness with plastic deformation at different annealing temperatures was shown in Figure 5. The micro-hardness was similar with plastic deformation at different annealing temperatures. At the initial stage of plastic deformation, the micro-hardness increased significantly with plastic deformation, which was due to the hardening of the material and the strengthening of the slip interaction between grains. At the later stage of plastic deformation, the micro-hardness of the material increased slowly, which was because the grain was elongated, and the slip band became more dense with the increase of deformation[12-13].
Figure 5. The micro-hardness with plastic deformation.

4.2. Evolution characteristics of microstructures

Microstructure at different temperatures were compared in Figure 6. After annealing at 450℃, some grains of 304 stainless steel became larger and rounded, indicating that recrystallization at this stage was incomplete and uneven. After annealing at 650℃, the grain size grew significantly and tended to be more round, and the materials were completely recrystallized. After annealing at 950℃, the grain size of the material continued to increase, and annealing twins were formed during the grain growth process.

![Figure 6](image)

(a) None-treated, (b) 450℃, (c) 650℃, (d) 950℃

Figure 6. Microstructure images at different temperatures.

Microstructure images for 1050℃ annealed the studied materials are shown in Figure 7. As can be seen from Figures 7, the grains were gradually elongated along the tension direction with increasing strain. The elongation of grains became more significant when it reached at the local deformation stage. For 1050℃ annealed samples, plastic deformation induced martensitic transformation, and a large number of dislocations continued to promote martensitic transformation[14-15]. Due to the low stacking fault energy of 304 austenitic stainless steel, a large number of dislocations and stacking faults were produced in austenitic stainless steel with low stacking fault energy during cold...
deformation. A large number of stacking faults and the proliferation of dislocations increased the free energy of the material and promote the nucleation in the process of transformation, which lead to the transformation of Y austenite phase into transitional martensite phase.

Figure 7. Microstructure of 1050°C annealed the studied materials at different strains.

Figure 8. The evolution of shape factor $\varphi$ with plastic strain.
The evolution of shape factor $\varphi$ with plastic strain was quantified from Figure 8. Similar variation trend of shape factor for AISI 304 steel with plastic strain at different temperatures was obtained. The grain boundary shape factor increased slowly with the increase of annealing temperature. This was due to the increase of annealing temperature, the grain size of the material grew, while the increment of grain boundary length in the fixed micro-field decreased.

The evolution of relative shape factor $\psi$ with plastic strain was calculated from Figure 9. It can be seen from the figure that the change trend of relative shape factor of AISI 304 steel at different temperatures was very similar. With the increase of plastic deformation, the relative shape factor increased gradually. When the strain reached the critical value, the relative shape factor increased obviously.

![Figure 9. The evolution of relative shape factor $\psi$ with plastic strain.](image)

In Figure 10, the evolution of the normalized shape factor $D$ was displayed. It can be found from the curves that the samples at lower annealing temperatures entered the plastic deformation stage earlier than the samples at higher annealing temperatures. When the plastic deformation went beyond a certain threshold, the samples at lower annealing temperatures would deform and damage faster than the samples at higher annealing temperatures. However, when the annealing temperature was 1050°C, the material was damaged quickly before the annealing temperature was 950°C. Due to high temperature annealing, the coarse grain of the material was not easy to plastic deformation, and the increase of martensite transformation made the toughness of the material worse.

![Figure 10. The evolution of the normalized shape factor $D$.](image)

The fitting equation can be expressed in general form as follows:

$$D = a + be^{c/\epsilon}$$  \hspace{1cm} (4)
Where \( a, b \) and \( c \) are material constant and can be determined experimental results by annealing temperatures. The material constant for AISI 304 steel were listed in Table 2.

### Table 2. Material constant list of fitting equation for AISI 304 steel.

| Constant | Initial | 450°C | 650°C | 950°C | 1050°C |
|----------|---------|-------|-------|-------|--------|
| \( a \)  | 0.0132  | 0.0576| 0.074 | 0.147 | 0.07   |
| \( b \)  | 0.033   | 0.009 | 0.001 | 0.134 | 0.06   |
| \( c \)  | 0.06    | 0.06  | 0.05  | 0.32  | 0.22   |

The damage evolution equation of 304 steel based on the shape factor of grain boundary can more accurately reflect the meso damage evolution law of 304 steel at different annealing temperatures, thus revealing the relationship between microstructure evolution and macro plastic deformation of 304 steel.

### 5. Conclusions

Tensile damage evolution behaviour of AISI 304 steel was being investigated at different temperatures. The following conclusions were drawn from this investigation:

1. With the increase of annealing temperature, strength and micro-hardness of the samples decreased gradually, while the elongation increased gradually. However, after annealing at 1050°C, the elongation of the material decreased, which showed this behavior due to formation of deformation induced martensite.

2. The shape factor of AISI 304 steel at different temperatures showed similar variation trend with the increase in different strains. The samples had earlier plastic deformation stage with increasing strain at lower annealing temperatures.

3. The evolution rule of the normalized shape factor with plastic strain can be expressed by equation of \( D = a + be^{\varepsilon_c} \) for AISI 304 steel at different temperatures.

4. The grain boundary shape factor can be used to describe the meso damage evolution of 304 stainless steel. It is a parallel method to describe material damage with microvoids.

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