

RESEARCH ON FORMABILITY OF 304 STAINLESS STEEL FOIL MICRO-DEEP DRAWING

The 0.05 mm-thick 304 stainless steel foil was annealed within the temperature range from 950°C-1100°C for 10 minutes to obtain different microstructures. And micro-deep drawing experiments of stainless steel foils with different tissue structures were conducted to obtain relevant material forming properties influenced by dimensional effects. On this basis, the influence of the microstructure characteristics on the forming performance of 304 stainless steel foil and the quality of the cup formed by using micro-drawing was studied, and its mechanism was discussed. It can be seen from the results that the stainless steel foil annealed at 950°C exhibits poor forming performance, and the wrinkle phenomenon of the deep-drawn cup is obvious. At the annealing temperature of 1050°C, the quality of the deep drawing cup is significantly improved. When the annealing temperature reaches 1100°C, with the increase of the annealing temperature, the crystal grains size increase sharply, and the coarse-grain effect causes the uneven plastic deformation effect to be obvious. Besides, the drawing quality is obviously deteriorated. The observation of the microstructure of the deep drawing cup shows that the forming effect of the drawing cup is poor due to the rolling defects and the coarse grain effect. The 304 stainless steel drawing cup annealed at 1050°C enjoys the best forming effect.

Keywords: 304 stainless steel; micro deep drawing; microstructure; annealing treatment

1. Introduction

With the rise of micro electro mechanical system (MEMS) and the electronics industry, including microelectronics, micro-mechanics, medical industry, aviation industry and many other applications, micro parts are playing an increasingly important role in the above fields [1-2]. In addition to miniaturization, micro parts are also required to increase their performance in terms of usage function, material properties or structural shape, therefore, the machining accuracy of micro parts is becoming more and more demanding.

Micro deep drawing, as a basic micro-forming process, uses a die to stamp flat blanks into various open hollow parts, or to reduce the diameter and increase the height of open hollow blanks [3]. Though it is widely used in various aspects, due to the size effect in the forming of micro parts [4-6], materials will appear differently in micro forming compared with macro forming. Many scholars have studied the micro parts of different materials [7-9], and found that the fracture behavior would change when the material was at the microscopic scale [10]. Xu Zhutian et al. [11] performed nanoindentation experiments and microhardness tests on thin plates of pure copper, and found

that both the elastic modulus and hardness of the material increased with the decrease of grain size. Suzuki et al. [12] in Japan found that, for pure aluminum foil under micro-stretching with grain size of 48 μm and thickness varying from 23 μm to 50 μm , the yield strength increased with the decrease of thickness, while the strain hardening coefficient increased significantly.

In surface formability studies, Engel and Eckstein [13] conducted extrusion experiments on brass, and found that both the decrease in specimen size and that in grain size can lead to the increase in the surface friction coefficient; hardness experiments on brass showed that the larger the grain size, the more uneven the deformation of the specimen, and the more pronounced the hardening, indicating that the microstructure at the grain level of the material has a significant impact on the forming quality of the part. In 2012, Chan et al. [14] investigated the effect of scale on the evolution of surface roughness during microvolume forming through upsetting experiments of pure copper with different billet sizes and grain sizes, and found that the surface roughness increased with the increase of grain size. This experimental law was also proven by the micro-bending experiments of Shi et al. [15] on thin plates. Manabe et al. [16] fabricated micro-cup shaped parts with a diameter of only 0.45 mm by using two-step forming.

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Chang et al. [17] studied the effect of grain size on the MDD forming of SUS304 stainless steel square cups, and found that the smaller the grain size inside the material, the lower the surface roughness. Gau et al. [18] proposed that the SUS304 microcups annealed at 1050°C will have better performance in quality. Luo et al. [19] found that during the forming process of SUS304 stainless steel foil, the friction effect has a great influence on the surface quality

Though 304 austenitic stainless steel is widely used in our daily life due to its excellent performance, there is little research on the microstructure and micro-drawing quality of 304 austenitic stainless-steel foil. Therefore, it is necessary to establish the relationship between the microstructure characteristics and the quality of the deep-drawn parts to obtain high-quality deep-drawing by using MDD Cup. In this paper, 304 austenitic stainless steel with the thickness of 50 μm is annealed within the range from 950°C to 1100°C, and the forming performance of 304 austenitic stainless-steel foil with different microstructures and the influence of the quality of the drawn cup are studied, thereby proposing a high-quality forming method is proposed. The best annealing temperature for deep drawing cups is also determined.

2. Experiment

2.1. Material preparation

It is shown that the grain organization structure of micro materials during the forming process can seriously affect the formability and fracture behavior of the material [20]. Cold-rolled 304 stainless steel foil was selected, with its main composition shown in TABLE 1 below.

TABLE 1
The main components of 304 stainless steel (wt%)

| C | Si | P | Ni | Cr |
|------|-------|-------|------|-------|
| 0.03 | 0.706 | 0.034 | 8.03 | 18.02 |

Using a TL-1700-1700-1700 vacuum tube furnace, 304 stainless steel foils with the thickness of 50 μm were treated for 10 Min. at 950°C, 1050°C and 1100°C, respectively for annealing to obtain different grain size structures. The whole process was carried out under the protection of inert gas, and the heating rate was kept at 10°C·min⁻¹ to obtain the austenitic 304 stainless steel foils with different tissue structures.

2.2. Tensile experiment

To further investigate the effect of grain size on the mechanical properties and fracture mechanism of the material, micro-tensile experiments were conducted on austenitic stainless-steel foils with different microstructures. The tensile samples were prepared according to ASTM E8/E8M-11, and the dimensions of

the prepared tensile specimens are shown in Fig. 1. The EDM cut tensile specimens were annealed at different temperatures to mitigate the effect of cut hardening. The tensile test was performed by using an INSTRON 5566 universal testing machine at the tensile speed of 0.1 mm·min⁻¹. Four specimens were selected for each condition, and the average value was obtained. SEM was used to observe the fracture morphology of the specimens with different heat treatments to obtain the fracture characteristics of the materials with different organization.

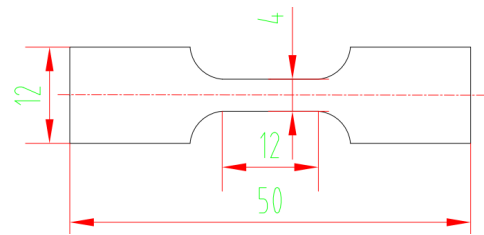


Fig. 1. Micro-forming stretching sample (unit: mm)

2.3. Micro-deep drawing experiment

A high-precision microplastic forming experimental machine is used for micro-deep drawing experimental research, and the equipment and die are shown on the Fig. 2a. The die can be used to realize the compound drawing of punching, cutting and dropping. Firstly, the material is punched and dropped at 0.01 mm·min⁻¹ by using the drawing and dropping die as well as the press plate, and then the drawing punch contacts with the blank for drawing until the drawing is completed to get the cylindrical drawing cup, with the important parameters of the drawing die on the Fig. 2b. The speed of the entire drawing process is controlled at 0.05 mm·s⁻¹, and the pressure sensor with a range of 10 KN is used to collect the changes of the drawing force during the entire process. The experimentally prepared deep-drawing cups of different types were cleaned by alcohol ultrasonic cleaning, and placed under the scanning electron microscope for observation.

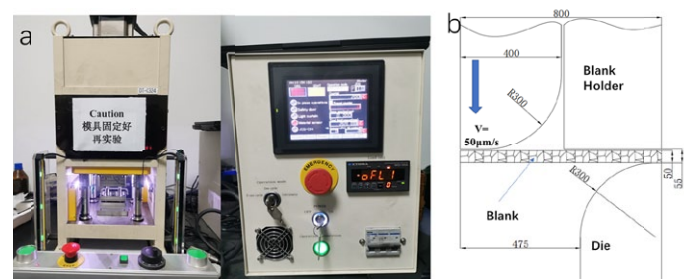


Fig. 2. Experimental equipment and mold diagram: (a) Equipment (b) key parameters of MDD tools (unit: μm)

Measurements were made at the bottom, rounded corner, wall and the mouth of the drawing cup, respectively, as shown in Fig. 3, to further analyze the change trend of deformation at different positions during the drawing process.

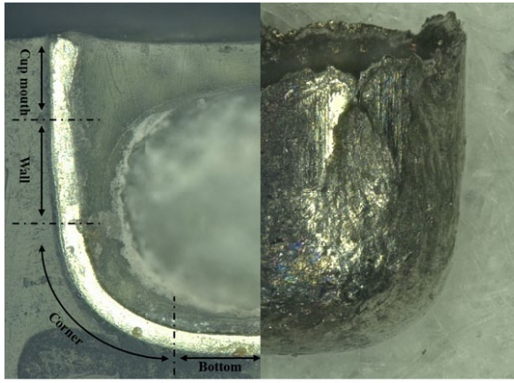


Fig. 3. Diagram of micro-deep drawing cup measurement

2.4. Simulation models

Use abaqus to generate the preliminary finite element model of the MDD, using a dimensionless analytical rigid body, which is easy to calculate, and does not calculate the wear amount of the punch. The schematic diagram of the assembly is shown in Fig. 4. In the process of establishing the interaction, the penalty friction with the friction coefficient of 0.15 is uniformly defined, and the boundary condition of the speed of $50 \mu\text{m} \cdot \text{s}^{-1}$ is applied to the punch part, and the simulation operation is carried out.

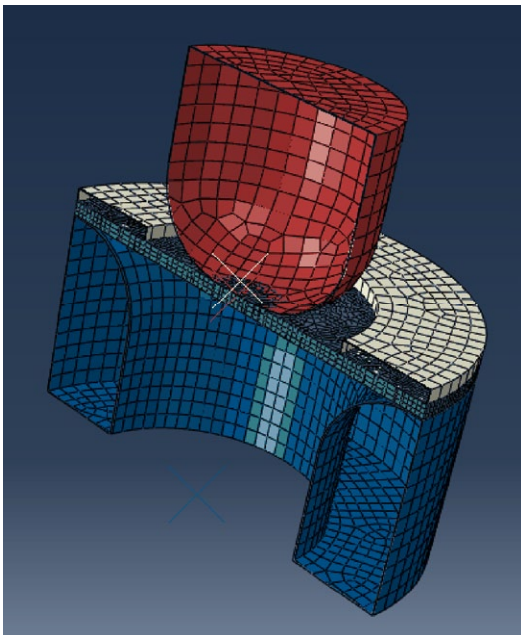


Fig. 4. Consider one-half MDD preliminary finite element model with symmetric boundaries

3. Results and Discussion

3.1. Characterization observation

Different specimens were inlaid with cold inlay material and polished, and a mixture of aqua regia and water was configured for the erosion of metallography and microstructure

observation under a Keenes VHX-2000V super depth-of-field microscope. Image Pro Puls software was used to measure the grain size, specifically, the truncated line method is utilized to obtain a section of the microstructure of the material at three different heat treatment temperatures with length L ; based on the number of grains measured for N ($N > 50$), the formula L/N is used to obtain the average value of the final grain size, the corresponding grain size of different heat treatment specimens is shown in TABLE 2 below, and the microstructure diagram is shown in Fig. 5.

TABLE 2

| Grain size (unit: μm) | | | |
|-----------------------------------|--------|--------|--------|
| | 950°C | 1050°C | 1100°C |
| Holding time | 600 s. | 600 s. | 600 s. |
| Grain size | 10.9 | 20.5 | 35.4 |

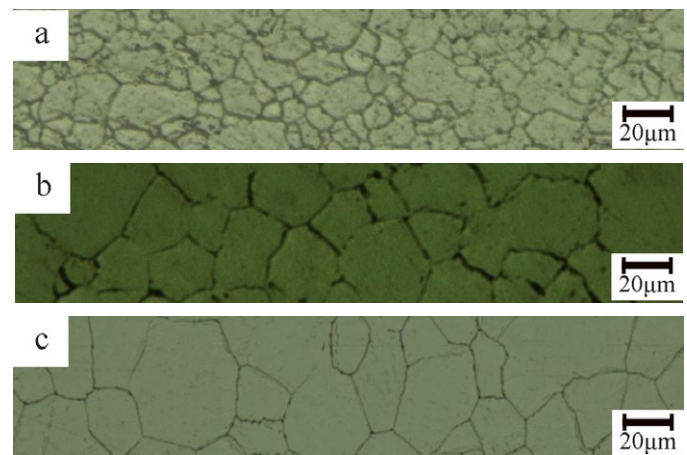


Fig. 5. Microstructure of the annealed specimens: (a) 950°C, (b) 1050°C, (c) 1100°C

3.2. Flow stress and fracture behavior

Fig. 6 shows the average value of true strain-true stress of three repeated tests, and it can be seen that when the annealing temperature is low, the grain size is small and the flow stress is high, which is consistent with the Hall-Petch effect. Smaller grain size implies a relatively large number of grain boundaries, and the dislocation movement of grain boundaries is hindered during plastic deformation, which is prone to dislocation buildup. In the tensile test, the accumulation of dislocations increased the deformation resistance, which was manifested by the fact that the flow stress became higher. Therefore, the highest flow stress was found at 950°C. With the increase of annealing temperature, the grain size gradually increases, while the flow stress gradually decreases. It can be found that after the strain of 0.08, the flow stress remains unchanged at the annealing temperature of 1050°C and 1100°C, which is because that when the annealing temperature reaches 1100°C, the abnormal grain growth occurs, and many small recrystallized grains are generated around the large grains during the entire plastic deformation process.

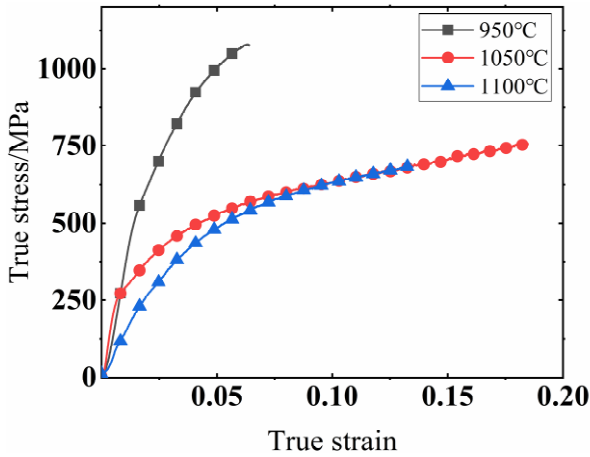


Fig. 6. True stress-true strain curves of 304 stainless steel foil with different grain sizes

By observing the elongation of the material, it can be found that the material exhibits poor plasticity by the annealing at 950°C and 1100°C, but good elongation by the annealing at 1050°C. As shown in Fig. 7, the fracture morphology of the tensile specimens after heat treatment at the three temperatures by using SEM shows that the fracture morphology of the material after heat treatment at 950°C is observed at high magnification as a partial tough nest with a deconfined zone. At the same time, an obvious brittle fracture is observed near the fracture opening, where the rolling defect leads to a significant reduction in

plasticity. As the temperature increased, the number of tough nests increased significantly in the material after the annealing treatment at 1050°C, exhibiting significant ductile fracture. When the temperature was increased to 1100°C, only a very small portion of tough nests existed at the fracture of the material, and an obvious deconstruction zone appeared, showing some brittle fractures and a significant decrease in elongation, which leads to a decrease in material plasticity.

3.3. Deep drawing force

For each kind of specimen, multiply the drawing depth curve by 10, and take the average value to get displacement-stroke force variation curve of 304 austenitic stainless steel, as shown in Fig. 8a. Seen from the figure, for the annealed specimen, the change of the drawing force with stroke can be divided into 3 stages, namely, Stage 1, Stage 2 and Stage 3. Firstly, Stage 1 is dominated by foil bending deformation, and the drawing force shows an obvious increasing trend, besides, the first stage of the specimen after annealing at 1100°C shows a low drawing force due to the grain size. As the drawing continues, the drawing force first gradually decreases and then increases sharply with a secondary peak, which is due to the fact that effect of the cup crease during the drawing process causes a sharp increase in the drawing force, resulting in a secondary increase (Stage 2). Then it enters the Stage 3, where the deep drawing

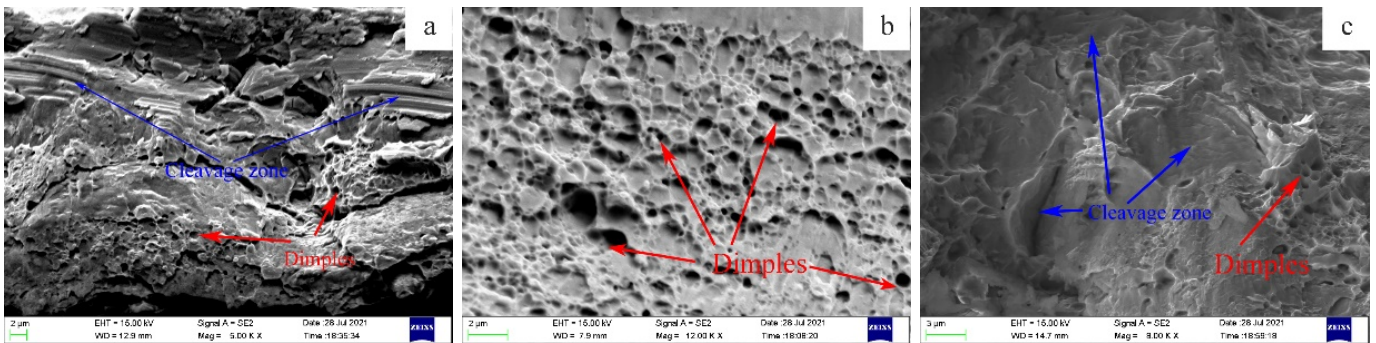


Fig. 7. 304 stainless steel foil fracture morphology: (a) 950°C, (b) 1050°C, (c) 1100°C

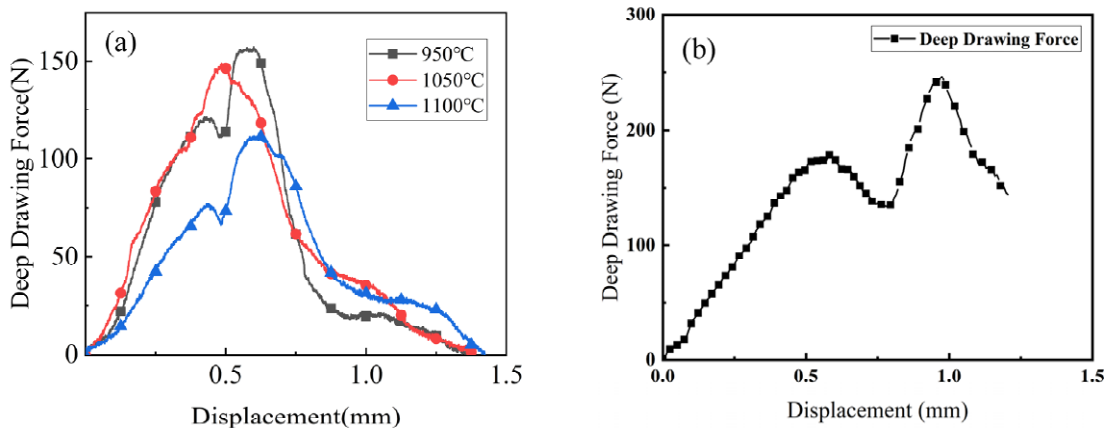


Fig. 8. Travel force variation curve with displacement: (a) experiment; (b) simulation

ends when the stroke reaches 1.2 mm. At this point the pulling force is not zero because of the presence of residual stresses.

Comparing the displacement-forming force curves of the three materials, it can be seen that the materials annealed at 950°C and 1100°C, respectively show obvious secondary growth during the deep drawing process. This is attributed to the presence of the cup crease that makes extra frictional force during the deep drawing process. The materials of the specimens annealed at 1050°C show no obvious secondary growth in the second stage of the deep drawing process, which can prove that under the annealing at this temperature, the material shows better micro-deep drawing properties at this temperature, confirming the results of the previous morphological observations.

For the extraction of the stroke force and displacement curve in the simulated micro-drawing process, as shown in Fig. 8b, the three stages of the micro-drawing process and the secondary increase of the stroke force in the second stage can also be observed from the figure. The phenomenon. However, the simulation results show that the stroke force reaches a maximum at a drawing displacement of 1.0 mm, because under the simulated conditions, the punch contacts the material with a constant coefficient of friction. In the actual processing process, the friction coefficient will wear with the progress of the processing, and the convex and concave die will be worn, resulting in a change in the roughness of the contact surface, resulting in errors between the simulation results and the experimental results.

3.4. Quality evaluation of the drawing cup

Figs. 9 and 10 show the top and side views of the drawdown cup at different annealing temperatures, respectively. It can be found from Fig. 9 that the height of the cup mouth of the specimen shows a gradually uniform characteristic with the increase of the annealing temperature, but when the annealing temperature of the material reaches 1100°C, the cup mouth wrinkle increases and the cup mouth thickness uniformity is poor.

Also, It can be seen that the specimen was annealed at 950°C, and the drawing cup exhibited a serious wrinkling phenomenon (Fig. 10a), indicating that the microplasticity of the 304-stainless steel foil after annealing at 950°C was poor. The reason is that the rolling effect makes the plastic forming properties of the material poorer. When the annealing temperature reaches 1050°C, the wrinkling phenomenon decreases, while the rolling effect is significantly reduced, besides, the plastic deformation of the material becomes uniform, and the plastic deformation capacity is significantly improved. With the increase of annealing temperature, when the annealing temperature is 1100°C, the formability of the drawn cup becomes worse, the cup crease increases, and the defects of the round cup increase. In addition, cracks appear, the symmetry of the drawn cup is weakened, and the phenomenon of ear value becomes obvious. This is because that when the annealing temperature reaches 1100°C, the grains of the material increase significantly, and the

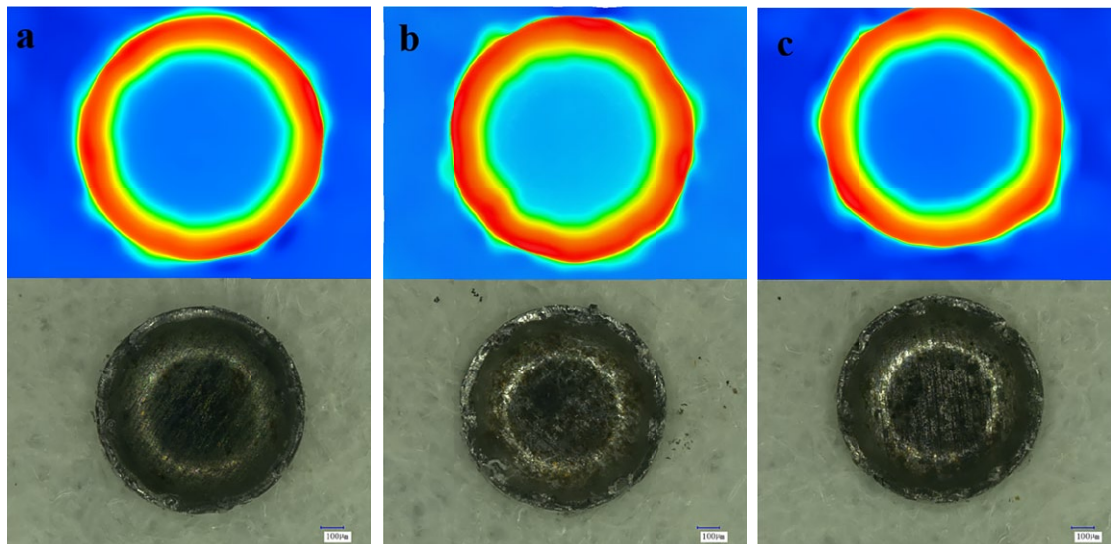


Fig. 9. Top height shape of annealed micro-deep drawing cup specimen: (a) 950°C, (b) 1050°C, (c) 1100°C

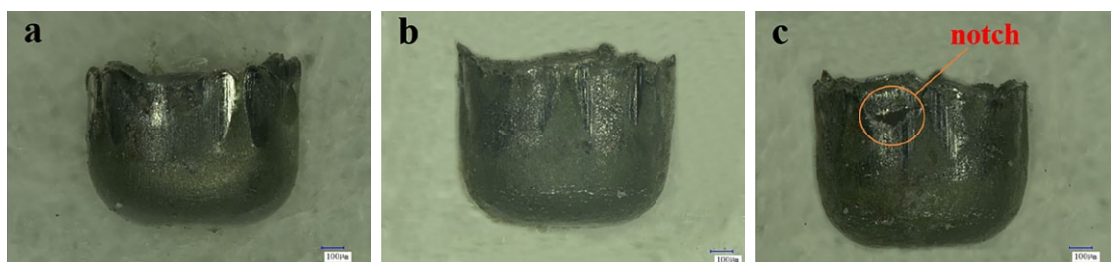


Fig. 10. Side shape of annealed micro-deep drawing cup specimen: (a) 950°C, (b) 1050°C, (c) 1100°C

anisotropy of the grains leads to the poor plastic deformation ability, resulting in the phenomenon of pulling and cracking in the sidewall part of the miniature cup. In addition, the coarse grain effect leads to the poor quality of the formed cup. It was found that the micro-drawing deep forming effect was better when the annealing temperature was 1050°C.

3.5. Thickness strain distribution

The thickness of deep-drawing cups with different heat treatments was measured, and some points were taken at equal intervals, as shown in Fig. 11. Three deep-drawing cups were selected from each group for respective measurement, and the average of the measured values was calculated to obtain the thickness distribution diagrams of different parts of the three deep-drawing cups, as shown in Fig. 12. It can be found that the thickness of the cup mouth increases significantly, while the thickness of the cup wall does not change much during the drawing process, and the thickness at the bottom of the cup and near the fillet decreases significantly. The thickness of the cup wall remains almost unchanged during the drawing process, which is because that during the entire drawing process, and the cups are subjected to radial tensile stress and axial compressive stress at the same time. In addition, the compressive stress leads to the thickening effect at the cups. During the drawing process, the rounded corners suffer the greatest drawing force, resulting in a higher degree of rounded corner thinning. The thickness change of the bottom of the deep-drawing cup is mainly due to the compressive effect of the radial force, which leads to the reduction of thickness. In addition, the thickness strain variation of the deep-drawing cup treated at 1100°C is the largest, which is mainly due to the presence of large-size grain anisotropy during the deep-drawing process, resulting in uneven material deformation and exhibiting a large thickness strain variation.

The results of microstructure observation of various parts of the drawing cup after different heat treatments are shown in Fig. 13(a-c), respectively. Based on the microstructure at the cup mouth of the drawing cup at 950°C-1100°C, it can be found that the grains near the cup mouth produce obvious accumulation. In particular, Fig. 13(b) shows that when the annealing

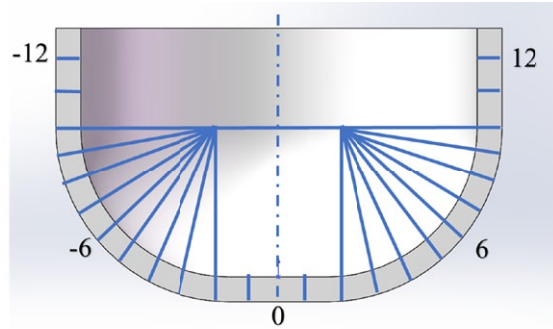


Fig. 11. Schematic diagram of micro part section taking points

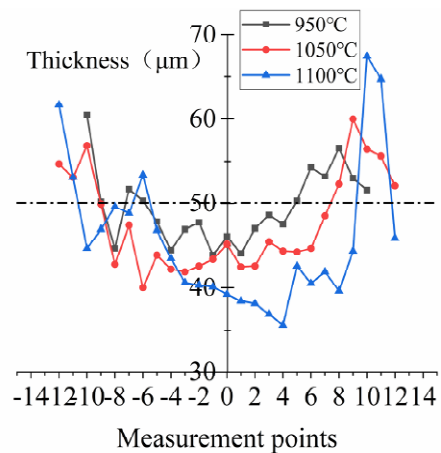


Fig. 12. Side wall dimensions of molded parts

temperature reaches 1050°C, the orientation of the grains at the cup mouth is more obvious in the form of thin and long stretching. And the larger grains lead to a more obvious thickening of the cup mouth.

In addition, for the deep-drawing cup rounded parts observed in Fig. 14(a-c), it can be found that after the heat treatment at 1100°C, the rounded corners of the deep-drawing cups are significantly thinned, and the grains in the rounded parts are refined, in addition, the uneven deformation of the grains leads to the uneven thickness of the deep-drawing cups and even fracture. Through the comparison, it is found that the uniform deformation of the grains at each position of the drawing cup

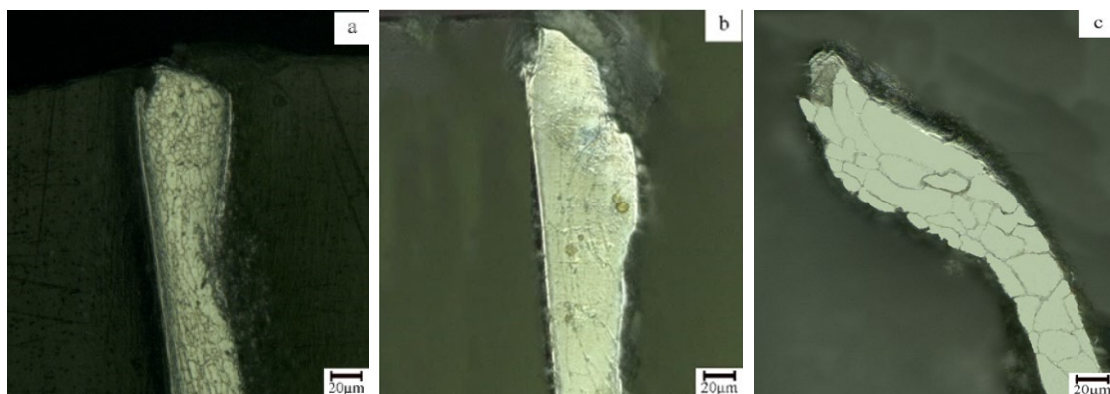


Fig. 13. Microstructure at the mouth of the micro-deep drawing cup: (a) 950°C, (b) 1050°C, (c) 1100°C



Fig. 14. Microstructure at the rounded corner of the drawing cup: (a) 950°C, (b) 1050°C, (c) 1100°C

occurs after the heat treatment at 1050°C, and the forming effect is the best among those under the three temperatures.

When this compressive stress exceeds the critical stress of the material, the cup will be wrinkled, resulting in a concentration of stress at the mouth of the cup and a thickening phenomenon. The bottom part of the cup, due to the combined effect of the radial tensile stress and the circumferential tensile stress, leads to a thinning zone in this part of the material. As the grain size in the material increases, this stress concentration becomes more pronounced.

4. Conclusion

The effect of different microstructures on the forming properties of stainless-steel sheet and the quality of the drawn cups was studied, and the optimal annealing temperature for forming high-quality drawn cups was proposed. The main conclusions drawn are as follows:

1. With the increase of annealing temperature, recrystallization and subsequent grain growth occur in austenitic 304 stainless-steel foil, the grain size grows significantly, and the average grain size of annealed specimens at 950°C, 1050°C and 1100°C is 10.9 µm, 20.5 µm and 35.4 µm, respectively.
2. Comparing the tensile properties and the fracture mechanism of different heat-treated materials, it can be found that the tensile specimens annealed at 1050°C show better plasticity and obvious ductile fracture.
3. Based on the comparison of the drawing force curves of different materials and the quality analysis of forming cups, it can be found that the forming quality of drawing cups at 950°C and 1100°C is poor, and there is obvious uneven plastic deformation. The forming cups at 1050°C have a high quality, and the phenomenon of secondary growth does not appear in the drawing curve.

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