Research on modification of cryogenic treatment on austenitic stainless steel

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Abstract. The effects of cryogenic treatment on the wear resistance, tensile properties and corrosion resistance of 304 stainless steel were investigated in the present work. The results show that one time and two times cryogenic treatment both improved the tensile strength, yield strength and corrosion resistance of 304 stainless steel without reduction of plasticity, and the effect of one time cryogenic treatment is the best. One time cryogenic treatment also improved the wear resistance of 304 stainless steel while no obvious change induced by two times cryogenic treatment. These improvements could be attributed to the release of residual stress and reduction of defect induced by cryogenic treatment.

1. Introduction
Cryogenic treatment is a new technology that places the workpiece to be treated in a specific and controlled low-temperature environment to change the microstructure of the material, thereby improving the performance of the material. At low-temperature, the microstructure of the treated material has changed, which shows the wear resistance of the material, corrosion resistance, dimensional stability, tensile strength, residual stress and other aspects of the material, therefore, domestic and foreign scholars have carried out a lot of related researches [1-2]. With the development of cryogenic technology and the improvement of test methods, people have gradually deepened the research on cryogenic treatment, in addition to steel materials, which have now been extended to powder metallurgy, copper alloys, aluminum alloys and other non-metal materials (such as plastics, nylon) [3-4]. This technology is widely used in many fields, such as aerospace, precision instruments, friction couple parts, tools, measuring tools, textile machinery parts, automobile industry, and military science. The improvement effect of cryogenic treatment on the abrasion resistance of traditional die steel is relatively obvious, the main mechanism is to promote the transformation of residual austenite into martensite in the steel, and at the same time, it can promote the precipitation of ultrafine carbide particles on the martensite matrix. At present, the research on the effect of cryogenic treatment of die steel is relatively common and the mechanism is relatively mature [5-6].

304 stainless steel has superior low-temperature performance and corrosion resistance, which is widely used in the field of low-temperature equipment in a corrosive environment, especially in loading and transport of LNG (-163°C). Low-temperature rotary joint and emergency release system of loading arm which made of 304 stainless steel, the wear performance of the contact surface directly affects the sealing performance of the joint in the process of operation. Thus, the corrosion resistance of the material directly affects the reliability and service life of the device, and improvement of wear resistance, corrosion resistance, and mechanical properties are of great significance to the development
of low-temperature stainless steel equipment. In recent years, some scholars have carried out research on the cryogenic treatment of stainless steel materials, but most of the research focuses on martensitic stainless steel [7-9]. Because austenitic stainless steel has no obvious martensitic transformation and no mature theoretical support for cryogenic treatment, thus the research on cryogenic treatment is less. This article focuses on the deep cooling treatment of AISI304 stainless steel in different processes, focusing on the effect of different processes on the wear resistance, corrosion resistance and mechanical properties of 304 stainless steel.

2. Test materials and cryogenic methods
The test raw material is solid solution 304 forging, and the chemical composition is shown in table 1.

| element | C  | Ni  | Si  | Mn  | P   | S   | Cr  |
|---------|----|-----|-----|-----|-----|-----|-----|
| content, % | 0.012 | 8.32 | 0.43 | 1.14 | 0.04 | 0.0025 | 18.82 |

The raw materials were processed into friction and wear samples, tensile samples and corrosion-resistant samples as shown in figure 1, figure 2 and figure 3, and were divided into three groups (process 0, process 1 and process 2) one by one, each with three samples. At present, it is generally used to put the workpiece directly into liquid nitrogen by the cryogenic treatment method, but the workpiece in the process of cryogenic treatment by thermal shock will produce stress concentration, in serious cases, it may even lead to workpiece cracking. Also, the relevant research shows that the process parameters such as the temperature drop speed of the cryogenic treatment, the holding time under the set cryogenic temperature, and the heating speed have a certain influence on the effect of the cryogenic treatment [10]. Considering the above factors, this test sets the cooling rate and heating rate are both 2 ℃/min, keep the temperature at -80 ℃ for 2 hours and the deep cooling process curve of -196 ℃ for 4 hours, as shown in figure 4. As shown in figure 4, process 0 is the sample without cryogenic treatment in the original state, process 1 curve is cryogenic once, and process 2 curve is cryogenic twice.

The cryogenic treatment is carried out by the cryogenic treatment box with a temperature control function. The temperature of the box is controlled by controlling the amount of liquid nitrogen into the box. Convective heat transfer occurs through the action of the fan and the sample after the liquid nitrogen enters the box, thus achieving the purpose of cooling and heat preservation. The temperature control range of the cryogenic treatment box is -196 ℃ - 250 ℃, the heating/cooling speed is 1 ~ 10 ℃/min, the temperature control accuracy is ± 0.5 ℃ and the temperature difference in all parts of the box is less than ± 2 ℃, which can better meet the requirements of the cryogenic process curve shown in Figure 4.
The friction and wear test was carried out under dry friction and wear conditions by MMU-10G end friction and wear testing machine, the upper sample is 304L stainless steel treated by different processes (0-without cryogenic treatment, 1-one time deep cryogenic treatment, 2-two times deep cryogenic treatment), and the lower sample adopts GCr15 bearing steel after quenching and tempering. The test load is 200N, the test speed is 200R/min, the friction time is 30min, and the friction coefficient value is recorded every 1s during the test. The upper sample was cleaned and weighed respectively before and after the experiment, and the weight change before and after the experiment was taken as the data of wear amount.

Using electronic universal testing machine and by GBT228.1-2010 《Tensile test method for metallic materials at room temperature》 to finish the tensile test and got the tensile strength, yield strength and elongation of the sample. Using the YWX-150 salt spray corrosion test chamber and by ISO 9227:2006 《Corrosion tests in artificial atmospheres-Salt spray tests》 to carry out acetic acid salt spray test (AASs test). The test period was 240 hours, and the influence of different cryogenic processes on the corrosion resistance of the material is qualitatively judged by visual inspection.

3. Test results and discussion

3.1 Effect of cryogenic treatment on friction and wear properties of 304 stainless steel

Figure 5 and Figure 6 shows the variation of wear and friction coefficient of 304 stainless steel after different processing, stainless steel wear has decreased after one time cryogenic treatment (process 1), but stainless steel wear has increased slightly after two times cryogenic treatment (process 2). It can be seen from the change curve of the corresponding friction coefficient that the corresponding friction coefficient of one time cryogenic treatment also decreases. Therefore, the wear resistance of stainless steel treated by process 1 is improved.
To reveal the effect of cryogenic treatment on the wear mechanism, the wear surfaces of the samples treated by process 0 and process 1 were tested by metallographic microscope, and the results are shown in Figure 7. From the aspect of wear mechanism, when two interacting surfaces contact, the real contact is only on a few encouraged micro convex body tips, and high local stress is generated on these contact areas, which exceeds the yield strength of the contact point and produces plastic deformation, leading to the increase of friction surface temperature, resulting in the direct contact of the exposed metal surface Sticky. Due to the continuous relative movement of the friction surface, the newly formed stick point is destroyed by shear, and new stick point is formed in some places. Therefore, the wear process of the stick point is the process that the stick point is formed and cut continuously [11]. It can be seen from the figure that the wear surface of the samples without cryogenic treatment has a wide furrow and a certain depth, and a large number of adherent oxides are formed near the furrow, while the wear surface of the samples after cryogenic treatment has a relatively uniform furrow, and the oxides in the wear process are evenly distributed on the wear surface, which greatly reduces the local stress concentration in the wear process and makes the wear process uniform and stable, so that the friction coefficient and wear amount are reduced, and the wear resistance of the material is improved.
3.2 Effect of cryogenic treatment on tensile properties of 304 stainless steel

Table 2 shows the different tensile strength of 304 stainless steel after different treatments. From the table, it can be seen that the strength of extension and yield strength of the stainless steel are all improved after one cryogenic treatment, in which yield strength is increased by 21Mpa. After two cryogenic treatments, the strength of extension and yield strength improve, too, and the yield strength increases by 19Mpa. Besides, the two treatments have no effect on the ductility, which means that cryogenic treatment, especially cryogenic treatment in a circular process, can greatly improve the strength of the 304 stainless steel with no decline in plasticity.

| NO. | Tensile strength Rm(MPa) | Average | Yield strength Rp0.2(MPa) | Average | Reduction of area Zt(%) | Average | Elongation rate A(%) | Average |
|-----|--------------------------|---------|--------------------------|---------|------------------------|---------|---------------------|---------|
| 0-1 | 615.6                    | 617     | 296.6                    | 299.9   | 78                     | 77      | 76.40               | 72.19   |
| 0-2 | 616.7                    | 617     | 293.8                    | 299.9   | 77                     | 77      | 74.32               |         |
| 0-3 | 616                      |         | 294.8                    | 299.9   | 77                     | 77      | 72.92               |         |
| 0-4 | 620.6                    |         | 314.5                    | 321.1   | 75                     | 75      | 65.12               |         |
| 1-1 | 635.9                    |         | 360.9                    | 321.1   | 81                     | 79      | 70.76               |         |
| 1-2 | 621.5                    | 626     | 286.3                    | 321.1   | 79                     | 79      | 72.56               | 71.20   |
| 1-3 | 623.2                    | 626     | 317.7                    | 321.1   | 83                     | 79      | 67.44               |         |
| 1-4 | 624.6                    |         | 319.3                    | 321.1   | 79                     | 79      | 74.04               |         |
| 2-1 | 630.6                    | 626     | 332.2                    | 318.9   | 81                     | 81      | 70.52               | 74.13   |
| 2-2 | 635.5                    | 626     | 358.9                    | 318.9   | 81                     | 81      | 72.80               |         |
| 2-3 | 616                      |         | 288.3                    | 318.9   | 81                     | 81      | 77.88               |         |
| 2-4 | 622.3                    |         | 296.3                    | 318.9   | 84                     | 84      | 75.32               |         |

It can be seen from the results of friction wear and tensile test, one-time cryogenic treatment can improve the wear resistance and tensile properties of 304 stainless steel. The micro internal stress of stainless steel increases due to lattice shrinkage at low temperature in the process of cryogenic treatment, the interaction between the internal stress and the original residual stress can promote the microplastic deformation of the material, thus release internal stress and improve the crack resistance of stainless steel. In addition, the dislocation movement caused by lattice shrinkage can promote the plastic flow of the micro defects of the material, thus reducing the internal defects of the material and making the structure tend to be uniform and stable.

3.3 Effect of cryogenic treatment on friction and wear properties of 304 stainless steel

The corrosion situation of 304 stainless steel samples treated by different processes after 240 hours of acetic acid salt spray test (AASS test) in figure 8. Figure 8 shows the sample without cryogenic treatment, treated according to process 1 and treated according to process 2 from left to right. Through visual inspection, it can be concluded that the corrosion degree of the sample after cryogenic treatment is significantly lower than that of the sample without cryogenic treatment (There is no obvious difference between the corrosion degree of the sample treated by process 1 and process 2, this article does not make a quantitative comparison.) Thus, cryogenic treatment can effectively improve the corrosion resistance of 304 stainless steel.
Figure 8. The change of friction coefficient of stainless steel 304 after different processing

The stainless and corrosion resistance of austenitic stainless steel is mainly due to the passivation of steel promoted by chromium and the stable passivation of steel. Chromium can quickly produce a very dense and stable passivation film on the surface of the stainless steel in the oxidation medium, the passivation film can be quickly repaired after being destroyed. Thus, the corrosion resistance of the material is effectively improved. In the process of cryogenic treatment, ultra-low temperature makes the lattice of the material shrink and the Gibbs free energy goes down between the crystal lattice, thereby corrosion resistance has been improved. In addition, the dispersed carbide particles precipitated during the cryogenic treatment can also increase the corrosion resistance of stainless steel. At the same time, the relevant research shows that the chromium carbide formed in the process of cryogenic treatment has a certain contribution to the improvement of the corrosion resistance of austenitic stainless steel [12].

4. Conclusion

Cryogenic treatment can improve the wear resistance of 304 stainless steel material, and the improvement effect is closely related to the treatment process, one-time cryogenic treatment can significantly improve the wear resistance, while two times cryogenic treatment is adverse to the wear resistance.

The cryogenic treatment can improve the strength of 304 stainless steel without reducing the plasticity, the results show that the strength is improved by one-time cryogenic treatment and two times cryogenic treatments, the yield strength is increased by 21 MPa after one time cryogenic treatment that is better than two times cryogenic treatment.

Cryogenic treatment can also significantly improve the corrosion resistance of 304 stainless steel materials.

In the process of cryogenic treatment, the release of internal stress and the reduction of microstructure defects are the main factors to improve the wear resistance and strength of materials.

Acknowledgments

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