Stress Rupture Properties of Welded Joints of Austenitic Heat-resistant Super304H Steel at 650 °C

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Abstract: With the help of metalloscope, scanning electron microscope and its energy dispersive spectroscopy, this paper investigates the stress rupture properties at high temperature and microstructure changes of the welded joints of austenitic heat-resistant Super304H steel at 650 °C as well as the effects of microstructure changes on stress rupture properties by high temperature creep rupture testing. The results show that at 650 °C, the welded joint of Super304H steel has high breaking strength at elevated temperature, and the fracture position is on the base material away from the fusion line, and the fracture type is mainly a transcrysalline rupture. The microscopic process of creep rupture of Super304H steel welded joints is the nucleation and growth of the void and crack, while the large bulk Nb(C,N) is the main nucleation site of void. As the creep time increases, M23C6 on the grain boundary is also the main nucleation site of the void.

1. Introduction
Super304H steel tube is mainly used in heating surface tubes of ultra supercritical steam generators. It is based on TP304H steel by adding about 3% Cu, 0.45% Nb and a certain amount of N. This allows the steel to pass through the precipitates during its service, including Nb (C, N), Cu-rich phase and NbCrN, endowing the steel with high temperature stress strength [1-6]. Its characteristics are grain refinement and precipitation hardening [7]. However, in welding conditions, these two characteristics in the welded joint will have some changes. Under high temperature, the difference in original tissue state will lead to different changes in tissue and performance [8-10], so it is particularly significant to study the creep rupture strength of welded joints of Super304H steel. In this paper, the microstructure changes of welded joints at 650 °C are studied by high temperature stress rupture testing of Super304H steel. Moreover, this paper explains the reasons for the change of stress rupture properties of Super304H steel welded joints, and provides reference for the technical supervision of Super304H steel parts in ultra supercritical steam generators.

2. Test materials and methods
The Super304H steel pipe for testing is supplied by Japan Sumitomo Corporation and its specification is Φ45 mm×9 mm. The welding wire is a matching argon arc welding wire #T-304H supplied by the Japanese company, and the specification is Φ2.4mm. The chemical composition of steel pipes and wires measured by SPECTROLAB quantitative spectrometer can be seen in Table 1.
Table 1 Chemical composition of Super304H steel pipe and welding wire (wt.%)

| Material       | C  | Si | Mn | P   | S   | Cr  | Ni  | Mo  | Cu  | Nb |
|----------------|----|----|----|-----|-----|-----|-----|-----|-----|----|
| Super304H steel| 0.10| 0.22| 0.85| 0.033| 0.006| 18.4| 8.56| 0.26| 2.41| 0.48|
| # T-304H       | 0.09| 0.22| 3.44| 0.013| 0.002| 18.1| 15.8| 0.76| 3.06| 0.65|

Super304H steel pipe butt joints use V-shaped groove with an angle of 60° and the blunt edge of 1mm, and the root gap is 2mm-2.5mm. It uses a gas tungsten arc welding (GTAW), placed in a 45° fixed position for multi-layer block welding. The welding current is 70A-100A, the interlayer temperature is controlled below 150 °C, and the root is filled with argon protection.

The stress rupture specimens at elevated temperature of the Super304H steel joint are processed according to the standard [11], and the high temperature stress rupture testing is carried out on the RC-1130 stress rupture strength testing machine. The temperature of the test is set to 650 °C, and the stresses are respectively 280 MPa, 260 MPa, and 240 MPa. The metallographic specimen preparation is etched with aqueous solution of ferric chloride hydrochloric acid solution, while the metallographic tissue is observed under an Olympus Model BX51M optical microscope. The creep rupture fracture and the microstructure are observed by a JSM-6380LA scanning electron microscope, and the main alloying elements of the precipitated phase in the tissue are tested by an energy dispersive spectroscopy.

3. Test results and analysis

3.1 Data analysis of stress rupture testing

The welded joint of Super304H steel is subjected to high temperature stress rupture test, and the specimens are all broken on the base metal away from the welded seam. At this point the temperature strength of the welded joint is still high. With the help of the Method of Isotherm, the creep rupture strength of the Super304H steel joint is extrapolated [12]. By creep rupture and fracture equation [13],

\[ \tau = A \sigma^B \] (1)

Where \( \sigma \) is the stress level (MPa) of the specimen loading; \( \tau \) is the fracture time of the specimen (h); A, B are the constants related to the test temperature and material. The logarithm function is used on both sides of equation (1), we get

\[ \log_{10} \tau = \log_{10} A - B \log_{10} \sigma \] (2)

It can be seen that \( \log \sigma \) has a linear relationship with \( \log \tau \), so linear regression can be performed by the least squares method. Test parameters A and B in the equation can be obtained by experiments with different stress levels at the same temperature.

Linear regression is performed on the experimental data and the results are as follows.

\[ \sigma = 426.49 \tau^{0.0714} \] (3)

The correlation coefficient \( R^2 \) is 0.7506 at 650 °C, indicating that the data has a good fitting effect. The stress rupture strength data of Super304H steel welded joints is plotted as Figure 1. It can be seen that the double logarithmic curve of creep rupture time and stress share a positive linear relationship. As a rule of thumb, it is reliable to extrapolate an order of magnitude for stress rupture strength data to derive data. When \( \tau = 10000 \), the result is Super304H steel \( \sigma = 220 \) MPa.
3.2 Creep fracture analysis

The stress specimens of the Super304H steel welded joints are all fractured on the base metal away from the fusion line. A macro photograph of parts of the specimens after fracture is shown in Fig. 2. It can be seen that different degrees of necking occurs at the specimen fracture. Since the test time is relatively short, the specimens are not completely creeped before the fracture, but a certain plastic deformation is generated.

Figure 3 shows the characteristics of the creep fracture of the welded joint of Super304H steel. Under high stress, the fracture of the specimen is mainly plastic deformation. As the stress is different, and the degree of plastic deformation will also be different. Under the creep condition of 650 °C / 240MPa (Figure 3(a)), the creep fracture is mainly in the form of transcrystalline rupture. There is a circular cleavage plane in the grains, and there are a large number of tear ridges between the grains. This indicates that the material is a ductile transcrystalline rupture, but a small number of short secondary cracks are observed. Under the creep condition of 650 °C / 280 MPa (Figure 3 (c)), the fracture morphology is also mainly the transcrystalline rupture. In the meanwhile, the tear ridges are significantly increased, and a small amount of secondary crack also occurs.

![Figure 3](image-url)

(a) 240MPa; (b) 260MPa; (c) 280MPa

Figure 2 Photo of stress specimens of Super304H steel joint

3.3 Tissue analysis of creep specimens

At 650 °C, the morphology and microstructure of the Super304H steel joint creep fracture are shown in Figure 4. The grain of the fracture specimen undergoes significant plastic deformation, and it is...
severely elongated (Figure 4(a) and (b)). It is identified as a transcrystalline, and precipitates along the grain can be observed at the boundary. More importantly, however, cracks will preferentially occur on large bulk precipitates. As described in the literature \cite{5}\cite{10}, the presence of the bulk Nb(C,N) in the base metal will adversely affect the creep rupture strength of the material. Moreover, the more continuous and larger the precipitated phase on the grain boundary, the more unfavorable the creep rupture strength. The void in the photo reflects the decrease in the binding force of the precipitated phase to the matrix during the elongation of the grain boundary. During the specimen preparation, the precipitated phase is peeled off, and the deformation becomes larger. Lower bonding force is further developed to connect the voids to form cracks.

(a) and (b) 650°C/240MPa/2253.4h; (c) and (d) 650°C/260MPa/1138.7h;
Figure 4 Scanned photo of the creep fracture specimens at 650 ° C

The precipitated phase in the creep specimen mainly has large-sized bulks and fine particles. The bulk precipitates are mainly distributed near the grain boundaries, and the results of the spectroscopy analysis (Figure 5 and Table 2) indicate that the main alloying elements are Nb. The number of fine particle precipitates is significantly increased compared to that in the supply state. Especially at the grain boundary, the longer the creep time, the more the number of precipitates on the grain boundary. The precipitation phase on the grain boundary is almost continuously distributed after creeping at 650 ° C/240 MPa for 2253.4 h. Spectroscopy analysis shows that the precipitates on the grain boundaries are mainly Cr and Fe compounds, and some of the grain boundary precipitates contain a certain amount of Nb.
4. Analysis and discussion

Under high stress, the material will generate a large amount of dislocation during the deformation. At high temperatures, the dislocations are affected by the interaction of stress and thermal activation. The creep behavior of Super304H steel belongs to the second type of creep behavior. The creep rate is controlled by the climb, and the self-diffusion energy of the matrix is similar to the creep activation energy\(^{[14][15]}\). Therefore, as the temperature goes up, the self-diffusion coefficient increases, causing the creep rate to increase significantly. Under high stress, the grain boundary sliding accounts for a small proportion in the creep deformation. The grain boundary is elongated along with the grain and exhibits the feature of transcryalline upon fracture. As the stress decreases, the proportion of grain boundary sliding deformation increases in creep deformation. To a certain extent, a void is formed at the grain boundary to develop a crack, which gradually transforms into an intercrystalline feature. As is known, the slidable interface of the precipitated phase and the matrix transmits only normal stress and cannot transmit tangential stress, and the plasticity of the precipitated phase is relatively poor. When there is a precipitated phase large in size in the grain boundary and the grain, cracking occurs to then become a crack. Therefore, it can be observed in the tissue that the larger-sized precipitate phase first produces crack.

Under the test circumstance, the creep fracture of the welded joint of Super304H steel is located on the base metal away from the fusion line, and the fracture morphology is mainly transcryalline rupture. Besides, a certain degree of necking of the specimen occurs at the fracture site. Analysis of the microstructure of the specimen reveals that after high temperature creep, the matrix structure is still austenite and precipitate. The results of spectroscopy show that the bulk precipitates are mainly Nb compounds. The precipitates on the grain boundary are transformed from particles or strips to net, chain and isolated particles. In addition, the precipitates on the grain boundary are mainly Cr and Fe compounds, and some precipitates are analyzed to contain element Nb. The second phase precipitate plays an important role in the void nucleation. Observing the characteristics of the creep fracture, it can be found that the creep damage of Super304H steel mainly appears as the formation of voids at the second phase precipitate, especially at the large Nb (C, N). In addition, the coarse precipitates on the grain boundary are also the main position of the void nucleation.

5. Conclusion

(1) Under high stress, the creep fracture of the welded joint of Super304H steel is located on the base...
metal away from the fusion line, and the fracture morphology is mainly transcrystalline rupture.

(2) The microscopic process of creep rupture of Super304H steel welded joints is the nucleation and growth of the void and crack, while the large bulk Nb(C,N) is the main nucleation site of void. As the creep time increases, M$_2$C$_6$ on the grain boundary is also the main nucleation site of the void.

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