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Effect of Microstructural Changes During Creep on The Creep Rate at 823K in Type 304 Heat Resistant Steel

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The effect of stress on the shape of creep rate - time curves at 823K in type 304 heat resistant steel has been investigated. The stress conditions of creep tests at 823K, stress range from 157MPa to 333MPa are higher than the 0.2% proof stress of the type 304 heat resistant steel. At the stresses higher than 265MPa, creep rate decreases monotonously in a transient creep stage and increases monotonously in an acceleration one, such shape of creep rate - time curve is called as normal type. Contrast to this shape in higher stress range of 333 to 265MPa, the shape of the creep rate - time curve in the medium range from 235 to 196MPa turns to the different one with two local minima due to the early inflection, and that shape of creep rate - time curve was designated as W-type. With further decreasing the stress, early inflection in creep rate - time curve turns to obscure one, and almost disappeared at the stresses lower than 177MPa. This shape of creep rate - time curve was designated as V-type. The main cause of changes in the shape of creep rate - time curve is the occurrence of the early infection in creep rate - time curve. Microstructural observation and changes in hardness have indicated that this early inflection has been caused by the dispersion strengthening precipitation of $M_{23}C_6$ on dislocations

1. Introduction

Austenitic heat resistant steels are used for high temperature structural components in the field of nuclear application. Considering the long- term exposure at the elevated temperature, creep deformation should be taken into account in the design of structural components. A lot of researches on creep deformation and microstructural examination in type 304 heat resistant steel have been done [1-13]. However, only a few researches have been done on those at 823K [14-18]. In this study, the creep tests at 823K were done in the wide stress range from 137 to 333MPa and the change in the shape of the creep rate - time curve with stress was discussed in conjunction with changes in microstructure during creep.

2. Material and Experimental method

2.1 Material

Material was sampled from a commercial plate which was produced as follows; an ingot of 16 ton which was produced from 30 ton melt in an electric furnace was hot-rolled to a plate in a thickness of 25mm and solution treated at 1373K for 0.5h and water quench [19]. The chemical composition and the average austenite grain diameter are shown in Table 1. Transmission electron micrograph of the specimen solution treated is shown in Fig. 1. Only a few precipitates of $M_{23}C_6$ were detected on dislocations. Appearance of $M_{23}C_6$ in the solution treated specimen is caused by slow cooling rate in the center of thick plate. The tensile property with the strain rates of 0.003/min for the 0.2% proof stress and 0.075/min for the tensile strength at 823K is given in Table 2.

2.2 Creep testing

The creep testing specimens with 50mm in gauge length and 10mm in diameter were sampled along a rolling direction of the plate. Tensile creep tests were carried out under the constant load condition over a range of stress from 137 to 333MPa at 823K. In order to investigate the microstructural change, creep tests at 823K-235MPa have been interrupted at 0.1h, 10h, 200h, 550h, 1540h and 3200h. Time to rupture in this creep condition is 3470h. Microstructures of the specimens to subjected creep were examined with a scanning electron microscope (SEM) and a transmission electron microscope (TEM). Measurement of Micro-vickers hardness was also done

Table 1 Chemical composition and austenite grain diameter of type 304 heat resistant steel

Material	Chemical composition (mass %)									Diameter
Type	C	Si	Mn	P	S	Ni	Cr	Mo	N	μm
304 steel	0.07	0.59	1.05	0.026	0.005	9.21	18.67	0.1	0.025	85

Table 2 Tensile properties of type 304 heat resistant steel at 823K

Material	0.2% proof stress MPa	Tensile strength MPa	Elongation %	Reduction of area %
Type 304 steel	149	402	44	70

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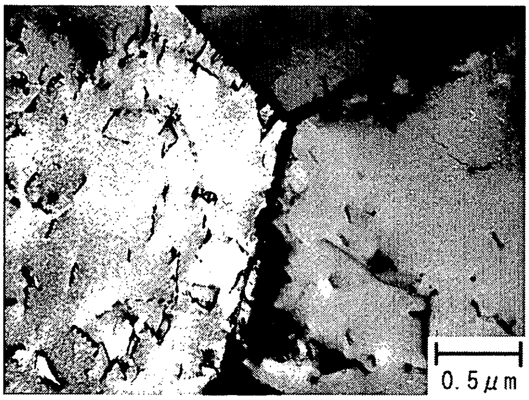


Fig.1 TEM micrograph as solution treatment of type 304 steel.

3. Results and Discussion

3.1 Creep rate - time curve

Creep rate - time curves of a type 304 steel over a range of stress from 137 to 333MPa at 823K are shown in Fig. 2, together with a stress - time to rupture curve. The almost linear relationship between stress and time to rupture in both logarithmic scales is obtained. The time to rupture at the stress of 157MPa was 105,350h. The 100,000 h creep rupture strength at 823K of the type 304 heat resistant steel is higher than the 0.2% proof stress of 149MPa as shown in Table 2.

On the other hand, the shape of creep rate - time curve is markedly changed with decreasing the stress as shown in Fig.2. In the creep rate - time curve at the stresses higher than 265MPa, creep rate decreases monotonously in a transient creep stage and increases monotonously in an acceleration one. This shape of creep rate - time curve was designated as normal-type. With decreasing the stress to a middle range from 235 to 196MPa, the shape of the creep rate - time curve turns to the different one with two local minima in the creep rate due to the early inflection. This shape of creep rate - time curve was designated as W-type. With further decreasing the stress, early inflection in the creep rate - time curve turns to obscure one, and almost disappeared at the stresses lower than 177MPa. This shape of creep rate - time curve was designated as V-type. The main cause of changes in the shape of creep rate - time curve is the occurrence of the early inflection in the creep rate - time curve. Corresponding to disappearance of the early inflection in the creep rate - time curve in the low stress region, decreasing ratio of the creep rate in the transient creep stage becomes large. Therefore, the shape of it changes from W-type to V-type.

The creep temperature of 823K is relatively low, about 0.48 of the melting point of this steel and the *stress range from157MPa to 333MPa is higher than the 0.2% proof stress of the type 304 heat resistant steel.* Therefore, the magnitude of the instantaneous strain at the loading must be evaluated, because the most effective strengthening mechanism is considered as the dispersion strengthening of $M_{23}C_6$ carbide precipitated on the dislocations and dislocation substructure is strongly influenced by the plastic deformation at the loading. From the above viewpoint, the relationship between instantaneous strain and stress of the steel is obtained and shown in Fig.3. Since the 0.2% proof stress is 149MPa, large plastic strain and, therefore, a lot of dislocations are introduced by the loading at the stresses higher than 149MPa. Such dislocations may influence the precipitation behavior of $M_{23}C_6$ during creep, as nucleation sites of that.

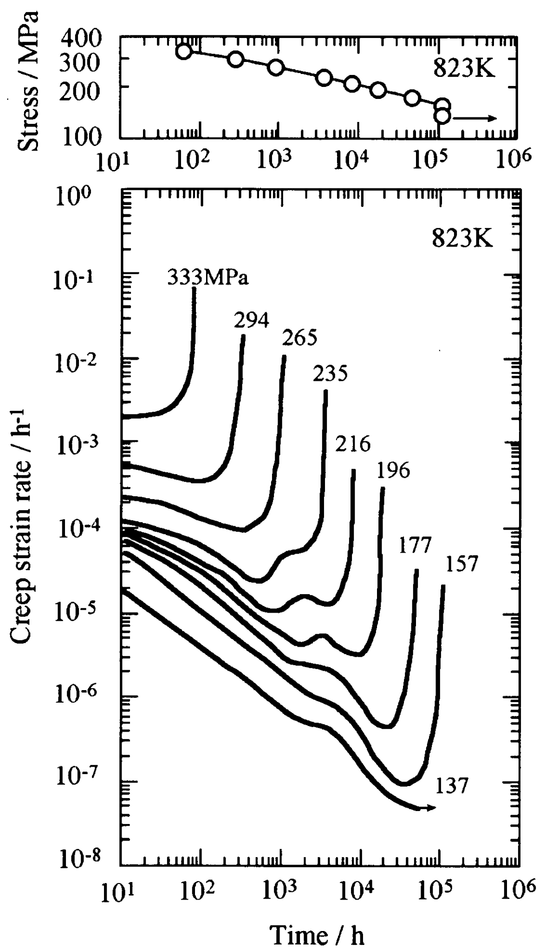


Fig.2 Stress rupture time and creep rate time curves at 823K.

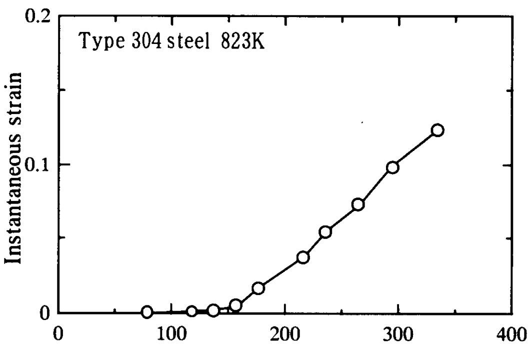


Fig.3 Instantaneous strain of type 304 steel at 823K.

The relationships between creep rate and strain of the steel are shown in Fig.4. The strain includes instantaneous strain. Increase in decreasing ratio of the creep rate in the transient creep stage is clearly observed with decrease in stress as mentioned above. The strain when the creep rate shows minimum value decreases from 0.2 to 0.02 with decrease in stress.

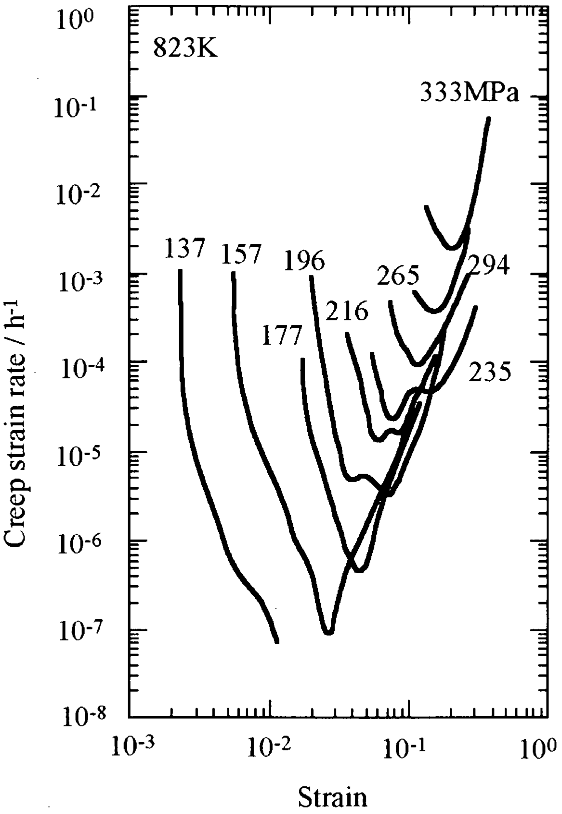


Fig.4 Creep rate –strain curves at 823K of type 304 steel.

3.2 Hardness within grain

The change in the shape of creep rate – time curve with decreasing the stress is caused mainly by the sudden decrease in creep rate during transient stage. And this sudden decrease in creep rate is restricted in the medium stress range from 196 to 235MPa. To elucidate the origin of the occurrence of this sudden decrease in creep rate, changes in hardness and microstructure during creep are investigated under focusing on the creep deformation at 235MPa that is the highest stress which shows such sudden decrease in creep rate.

Creep rate - time curve at 823K-235MPa is shown in Fig.5. Creep tests at 235MPa were interrupted at the times of 0.1h,10h, 200h, 550h, 1540h and 3200h. The change in Micro-Vickers hardness of creep interrupted specimen with increasing the creep testing time is shown in Fig.6. Increase in hardness of 30 was detected in a time just after loading. Further 20 increase in hardness was attained after crept for 200h. After creep for more

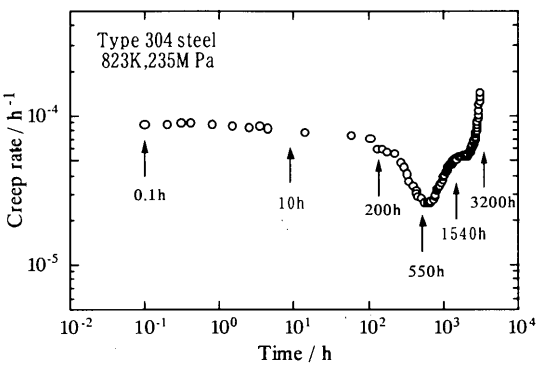


Fig.5 Interrupted crept condition at 823K and 235MPa of type 304 steel.

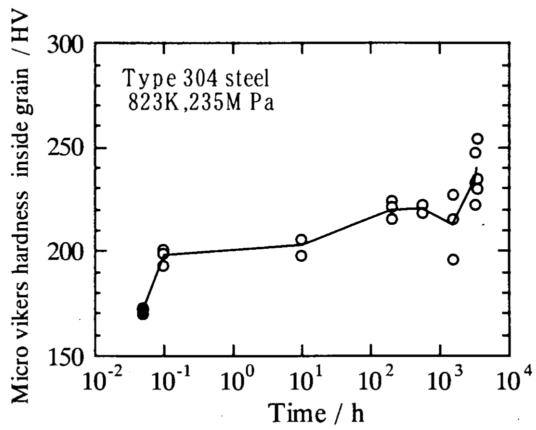


Fig.6 Relationship between Micro-vickers hardness and crept time.

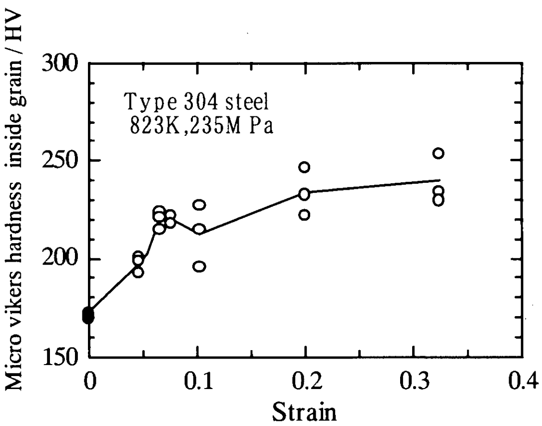


Fig.7 Relationship between Micro-vickers hardness and creep strain.

than 200h, hardness remains constant value. A little increase in hardness in ruptured specimen should be attained during accelerating stage. Fig.7 shows the relationship between hardness and creep strain. Continuous increase in hardness with increase in the strain was detected.

3.3 Microstructural evolution

As mentioned above, strong stress dependence of the shape of creep rate - time curve is mainly caused by the stress dependence of the decreasing ratio of the creep rate in the transient creep stage. So the stress of 235MPa is chosen as the stress to discuss the origin of sudden decrease in creep rate.

Scanning electron micrographs at the grain boundary of the specimens creep interrupted at 0.1h, 550h, 1540h, and 3200h at 823K-235MPa are shown in Fig.8. Even in the specimen crept for 0.1h (Fig.8-a), precipitates were found at the grain boundary. In the specimen crept for 550h (Fig.8-b), a series of granular phases continuously precipitated on the grain boundary is observed. A slightly coarsened precipitates are detected at the triple point of the grain boundary in the specimen crept for 1540h (Fig.8-c). In the specimen crept for 3200h, further coarsened precipitates are observed at the grain boundary and a ratio of the grain boundary area covered with precipitates to the total area of that decreases (Fig.8-d).

The transmission electron micrographs of the specimens creep interrupted at 0.1h, 550h, 1540h and 3200h at 823K-235MPa are shown in Fig.9. In the specimen crept for 0.1h (Fig.9-a), a lot of fine $M_{23}C_6$ are precipitated on dislocations. By comparison of the dislocation densities of the specimens crept for 0.1h (Fig.9-a) with that in the specimen crept for 550h (Fig.9-b), higher density of dislocation can be detected in the specimen crept for 550h. A lot of tangled dislocations are observed on the precipitates in the specimen creep interrupted at 1540h (Fig.9-c). There are large number of fine particles precipitated on the dislocations in the specimen creep interrupted at 3200h (Fig.9-d).

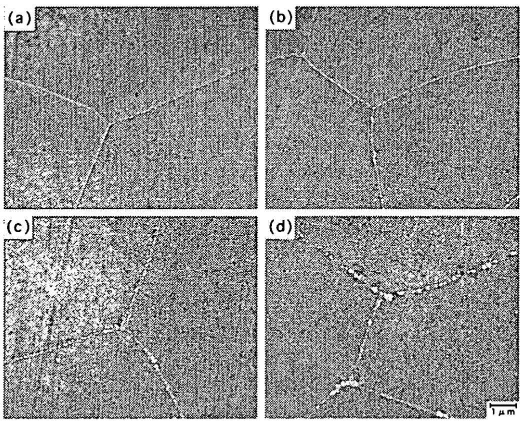
From the above observations, microstructural changes during creep deformation at 823K-235MPa are summarized as follows;

1) Dislocation density increases with creep deformation in the transient creep stage, and a substructure of high dislocation density is formed when the creep rate shows minimum value.

2) High precipitation density of fine particles of $M_{23}C_6$ occurs on dislocations after creep deformation for 2000h and more.

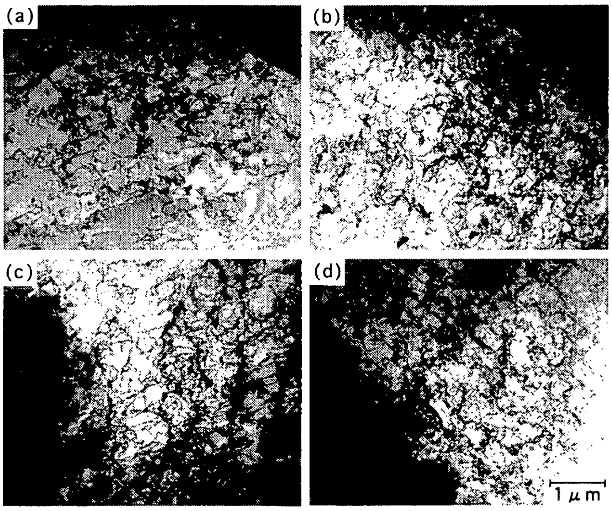
3) Ratio of the grain boundary area covered with precipitates to the total area of that increases with creep deformation in the transient creep stage, and it decreases in the acceleration one corresponding to increase in creep rate after showing the largest value.

The first phenomenon is strain hardening, the second one is precipitation strengthening by the secondary precipitation of $M_{23}C_6$ on the dislocation within grain and the final one is the grain boundary precipitation strengthening due to the coverage of grain boundary by the second phase. Interaction between such distribution of the precipitates and dislocation substructure is very important factor that affects creep strength and, consequently, a shape of creep rate - time curve. In the high stress region higher than 265MPa, effect of fine particles of $M_{23}C_6$ precipitates on creep strength is not so large, because significantly high density of dislocation is generated and the amount of fine precipitates is too small to act as a barrier of the moving of dislocation. In the medium stress range from 196 to 235MPa, fine $M_{23}C_6$ particles precipitated on the dislocations play a significant role effectively as a barrier of the moving of dislocation, because of good balance of the amounts of fine precipitates and dislocation density. Therefore, early inflection of the creep rate - time curve is produced and it represents W-type shape. Since dislocation density decreases and coarsening of the precipitates occurs with decrease in stress, early inflection of creep rate - time curve turns to smaller one and the shape of it changes from W-type to V-type. Creep strength of the type 304 heat resistant steels at 823K is



a) Crept for 0.1h
b) Crept for 550h
c) Crept for 1540h
d) Crept for 3200h

Fig.8 SEM micrograph of interrupted crept specimens at 823K and 235MPa.



a) Crept for 0.1h
b) Crept for 550h
c) Crept for 1540h
d) Crept for 3200h

Fig.9 TEM micrograph of interrupted crept specimens at 823K and 235MPa.

strongly influenced by precipitation and coarsening of $M_{23}C_6$ particles. It has been concluded that change in the shape of creep rate - time curve is caused by changes in creep strength corresponding to changes in microstructure during creep.

4. Conclusions

The effect of stress on the shape of creep rate - time curves at 823K in type 304 heat resistant steel has been investigated. The stress conditions of creep tests at 823K, stress range from 157MPa to 333MPa are higher than the 0.2% proof stress of the type 304 steel. From the results of this work the following conclusion have been drawn:

1) The shape of creep rate - time curve in which creep rate decreases monotonously in a transient creep stage and increases monotonously in an acceleration one, has been observed at the stresses higher than 265MPa. This shape of creep rate - time curve was designated as normal-type. With decreasing the stress to a middle range from 235 to 196MPa, the shape of the creep rate - time curve turns to the different one with the early inflection, and that shape of the creep rate - time curve was designated as W-type. With further decreasing the stress, early inflection in creep rate - time curve turns to obscure one, and almost disappeared at the stress lower than 177MPa. This shape of the creep rate - time curve was designated as V-type. The change in the shape of the creep rate – time curve is caused mainly by sudden appearance of decrease in creep rate during transient creep stage.

2) Increase in micro-Vickers hardness of about 30 immediately after loading, and gradual increase in it of about 20 with creep deformation for 200h were detected. Hardness in grain increases about 60 after creep rupture.

3) Precipitation and coarsening of $M_{23}C_6$ particles on dislocations and at grain boundaries were observed during creep deformation. Increase in dislocation density is also detected with progress in creep deformation in the transient creep stage. It has been concluded that the change in shape of the creep rate - time curves has been caused by the change in creep strength that has been strongly influenced by the balance of the amounts of fine $M_{23}C_6$ particles precipitated on dislocations and dislocation density.

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