Materials Science Research International, Vol.3, No,1 pp.10-15 (1997)

General paper

LONG-TERM STRESS RELAXATION PROPERTIES AND MICROSTRUCTURAL CHANGES OF NCF 800H

Toshio OHBA*, Osamu KANEMARU*, Koichi YAGI* and Chiaki TANAKA**

* *Environmental Performance Division*
National Research Institute for Metals
Tsukuba 305, Japan
** *Space Center*
National Space Development Agency of Japan
Tsukuba 305, Japan

Abstract: Long-term stress relaxation behavior was investigated on NCF 800H alloy. Complicated stress relaxation behaviors such as rapid decrease of residual stress and increase of residual stress were observed. Relationship between stress relaxation behavior and microstructural changes was examined. The decrease of residual stress was caused by the reduction of creep deformation resistance due to coarsening of carbides. Minus relaxation behavior which is equivalent to the increase of residual stress was related to shrinking of testing material due to precipitation of carbides. Therefore, it is difficult to extrapolate the long-term stress relaxation curve from the result of short-term tests, and the long-term residual stress value should be predicted using aged material.

Key words: *NCF 800H alloy, Stress relaxation, Microstructural changes, Minus relaxation behavior*

1. INTRODUCTION

Anomalies in creep curves of heat resistant steels and alloys under long-term loading are realized[1-3]. Microstructure of metallic materials is instable at high temperature, and changes with exposed time[4,5]. Therefore, it is necessary to investigate the anomalies of creep deformation behavior from the viewpoint of microstructural changes. However, the effect of microstructural changes on creep deformation behavior has not yet been understood, because it is difficult to obtain the systematic data set of long-term creep curve. Recently, it began to be recognized from analyses of long-term NRIM creep data that the creep deformation is strongly dependent on the microstructural changes, especially at low stress conditions[6,7].

The understanding of stress relaxation behavior is important for designing structural components in high temperature plants[8]. Long-term stress relaxation behavior would reflect the creep deformation behavior at low stress conditions, and it might be affected by the microstructural changes. Therefore, it is necessary to study the long-term stress relaxation behavior with microstructural changes. However, there are not any reports on long-term stress relaxation behavior of high temperature structural materials with microstructural changes[9,10]. The test data obtained are not enough to analyze the effect of microstructural changes on stress relaxation behavior. Therefore, the long-term stress relaxation behavior of some heat-resistant alloys should be investigated from the viewpoint of microstructural changes[4,11].

In the present work, the effect of temperature and total strain on long-term stress relaxation behavior of NCF 800H alloy which shows the microstructural instability[12] was studied, and the relationship between stress relaxation behavior and microstructural changes was examined.

2. TESTING MATERIAL AND EXPERIMENTAL PROCEDURE

2.1. Testing Material

The material tested is NCF 800H alloy bar of 25mm in diameter. The chemical composition is listed in Table 1. The solid solution heat treatment condition is 1150°Cx5h, and the austenitic grain size number is 3.3. The tensile properties at room temperature are listed in Table 2.

Table 2. Tensile properties at room temperature.

0.2 proof stress MPa	Tensile strength MPa	Elongation %	Reduction of area %
240	564	48	74

2.2. Stress Relaxation Test

The stress relaxation tests were conducted using automatically controlled tensile-type testing machines with load capacity of 100kN[13]. The specimen is 10mm in diameter and 100mm in gauge length.

The stress relaxation test were carried out according to JIS standard, and the specimens were soaked for 20h at testing

Table 1. Chemical composition of NCF 800H alloy (mass%).

C	Si	Mn	P	S	Ni	Cr	Mo	Cu	Co	Ti	Al	N
0.08	0.23	0.92	0.009	0.001	31.46	20.31	0.031	0.06	0.072	0.35	0.34	0.0180

Received August 30, 1996

temperature prior to testing.

2.3. Stress Relaxation Testing Condition

The stress relaxation testing condition is listed in Table 3. The stress relaxation tests were carried out under the total strain condition of 0.05 to 0.25% at 600 to 750°C. The longest testing period was 12000h.

Table 3. Stress relaxation testing condition.

Temperature ℃	Total strain %
600	0.05
	0.20
650	0.15
	0.20
	0.25
700	0.20
750	0.15
	0.20
	0.25

3. RESULTS AND DISCUSSION

3.1. Result of Stress Relaxation Tests

The stress relaxation curves for the total strain of 0.20% are shown in Fig.1. For 600°C, the residual stress rapidly decreases at 300h, gradually increases from 1000h and then decreases again at 7000h. For 650°C, the residual stress rapidly decreases at 10h, increases at 250h and then decreases again at 2000h. The stress relaxation behavior at 700 and 750°C is also similar to that at 600 and 650°C, and the residual stress has already decreased at 0.1h and then a small change in residual stress is found. The stress relaxation behavior of this material is complicated, and the relationship between testing temperature and residual stress is not simple.

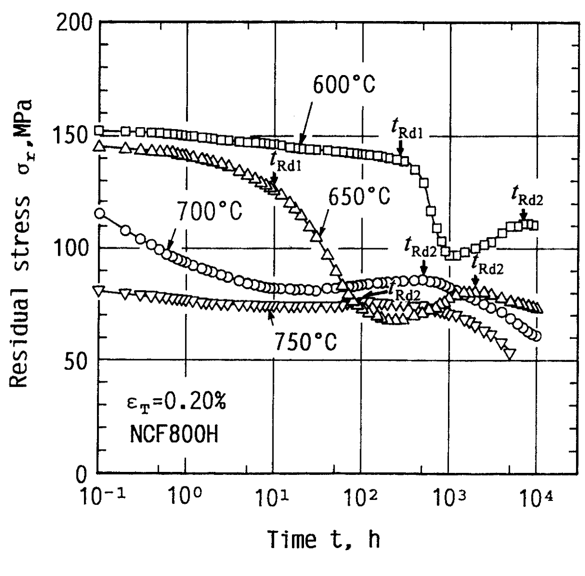

Fig.1. Stress relaxation curves of NCF 800H alloy.

3.2. Effect of Total Strain on Stress Relaxation Behavior

The effect of total strain on stress relaxation behavior at 600, 650 and 750°C for total strain from 0.05 to 0.25% was examined. The results are shown in Figs. 2, 3 and 4. For 600°C, the value of residual stress is different between 0.05 and 0.20% in total strain conditions, but the rapid decreasing time and the increasing time are almost the same in both conditions.

For 650 and 750°C, little effect of total strain on residual stress is recognized except for low total strain condition.

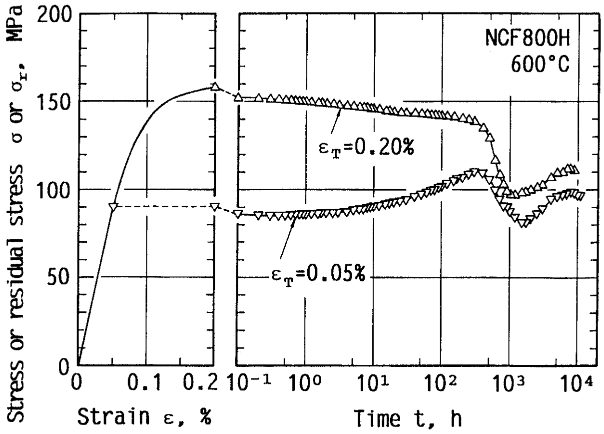

Fig.2. Effect of total strain on stress relaxation behavior for NCF 800H alloy at 600°C.

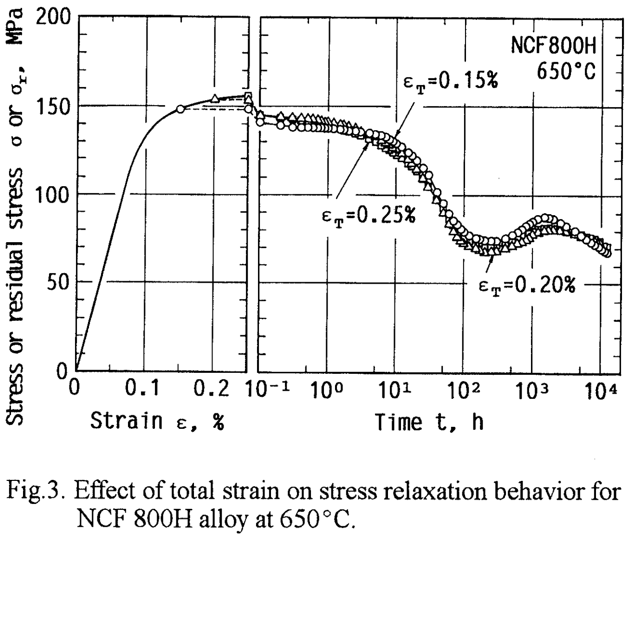

Fig.3. Effect of total strain on stress relaxation behavior for NCF 800H alloy at 650°C.

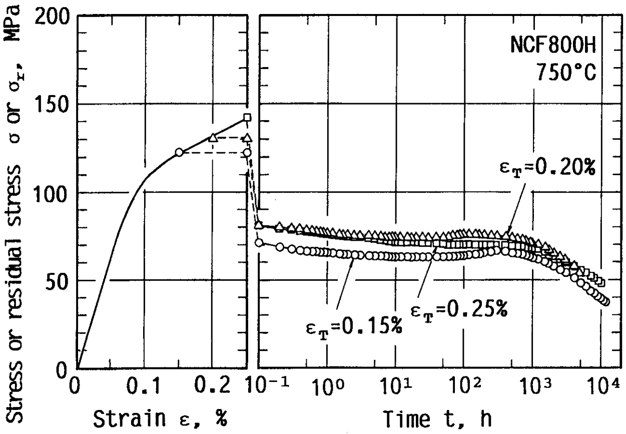

Fig.4. Effect of total strain on stress relaxation behavior for NCF 800H alloy at 750°C.

3.3. Relationship Between Stress Relaxation Behavior and Creep Deformation Behavior

The stress relaxation behavior is closely related to the creep deformation behavior. The creep deformation behavior of this NCF 800H alloy was examined. Figures 5 and 6 show the creep curves which were obtained from the creep tests at 600 and 650°C. For 600°C, the rapid acceleration of creep deformation is seen at 300h after the short-term transient and steady-state creep regions, and then the creep rate gradually decreases and the creep curve represents again the steady-state aspect. This rapid increase of creep rate occurs at a specified time and the rapidly increasing time is independent of the stress condition in creep tests. The time at which the rapid increase of creep rate was started on creep curve t_{Ci}

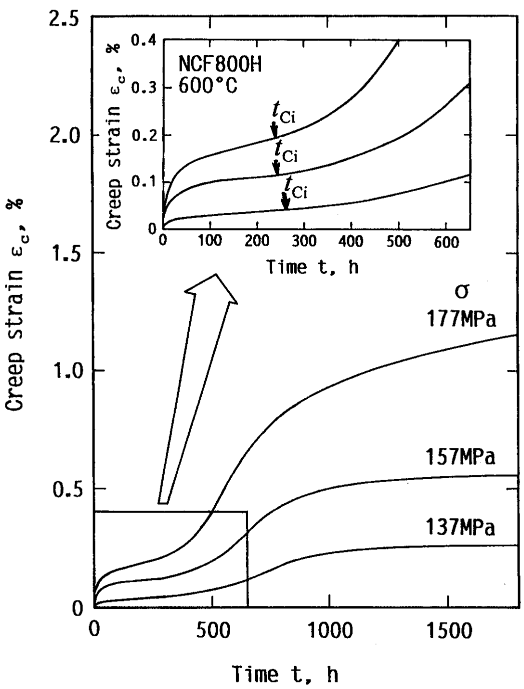

Fig.5. Creep curves of NCF 800H alloy at 600°C.

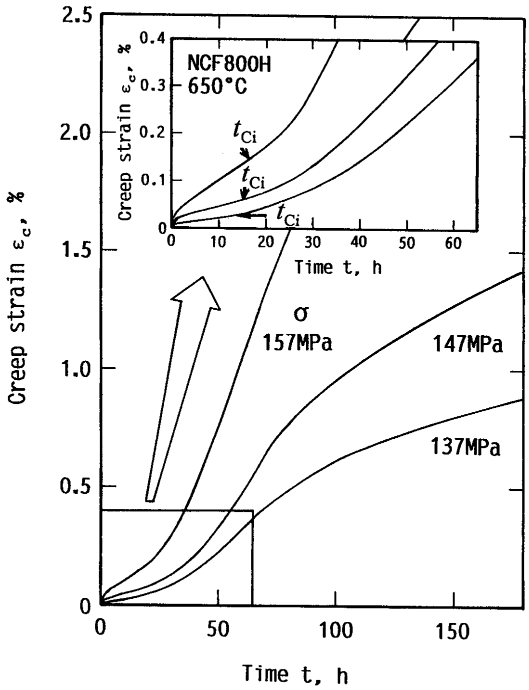

Fig.6. Creep curves of NCF 800H alloy at 650°C.

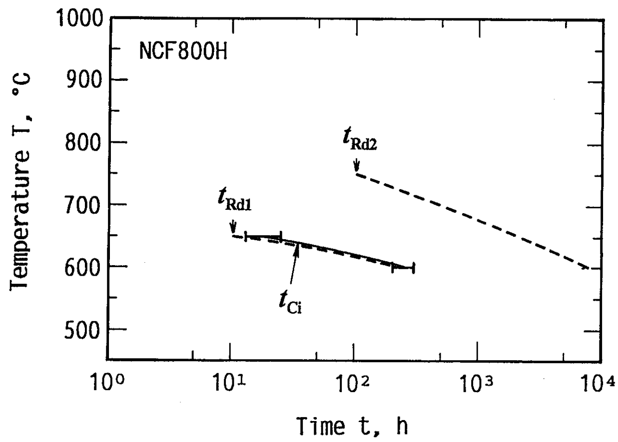

Fig.7. Comparison of rapidly decreasing time t_{Rd1} and t_{Rd2} of residual stress in stress relaxation curves with rapidly increasing time t_{Ci} of creep strain in creep curves.

was compared with the times corresponding to the rapid decrease of residual stress on stress relaxation curve t_{Rd1} and t_{Rd2}. Figure 7 shows the comparison of t_{Ci} with t_{Rd1} and t_{Rd2}. t_{Ci} of creep curve agrees well with t_{Rd1} of stress relaxation curve. The rapid decrease of residual stress in stress relaxation curve which was observed at 300h for 600°C and at 10h for 650°C was caused by the reduction of creep deformation resistance.

3.4. Microstructural Changes

The rapid acceleration of creep rate and the rapid decrease of residual stress might be dependent on microstructural changes[3]. The microstructure of the materials aged and tested was observed using transmission electron microscope. The precipitates were determined by X-ray diffraction analysis of electrolytically extracted residue. The results of microstructural examination were plotted on a Time-Temperature-Precipitation (TTP) diagram as shown in Fig. 8. The precipitates of TiC and $M_{23}C_6$ were identified by X-lay diffraction analysis. The result of TiC is not plotted in Fig. 8, because TiC was regarded as insoluble inclusions. The numerical value on each symbol means the amount of electrolytically extracted residue, which reduces the amount of electrolytically extracted residue of as-received material from the total amount of electrolytically extracted residue of aged materials. This material, NCF 800H alloy, is well-known as

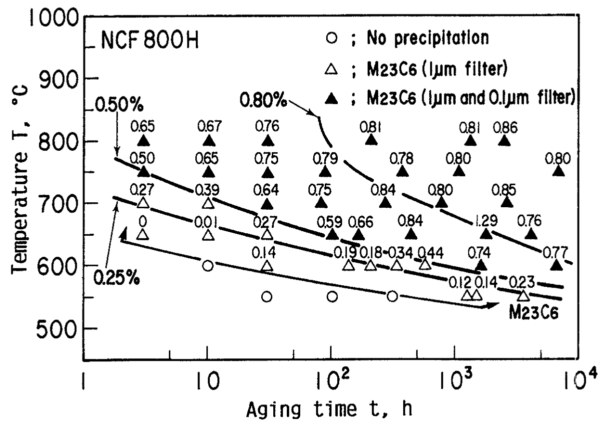

Fig.8. Time-Temperature-Precipitation diagram of NCF 800H alloy.

an alloy which is strengthened by the precipitation of γ' phases. Little γ' phase was observed in the specimens aged and tested, because the amount of solute Ti is not enough due to a lot of formation of Ti-nitride as inclusions[14].

The electrolytically extracted residue was collected at first using 1 μm filter, and then using 0.1 μm filter. Figure 8 also shows the type of filter which was used for the collection of electrolytically extracted residue. For low temperatures and short times, $M_{23}C_6$ was collected using 1 μm filter but not collected using 0.1 μm filter. On the other hand, with increase of aging time, $M_{23}C_6$ was collected using 1 μm filter and 0.1 μm filter. In this figure, the curves with equivalent amount of electrolytically extracted residue are shown based on the experimental result of $M_{23}C_6$ amount. Temperature-time curve of 0.5% in $M_{23}C_6$ amount of extracted residue is almost correspondent to the condition of temperature and time under which $M_{23}C_6$ can be collected using the 0.1 μm filter. The amount of residue is saturated at about 0.80%. Relationship between the change in stress relaxation behavior and creep deformation behavior and the equivalent curve of extracted residue were examined. The result is shown in Fig. 9. The time at which the residual stress of the stress relaxation curves is rapidly decreased, t_{Rd1}, and the time at which the creep rate is accelerated from the first steady stage to the second steady stage, t_{Ci}, agree with temperature-time condition of 0.25% in the amount of extracted residue. The time at which the residual stress is decreased again is corespondent to the temperature-time condition in 0.80% amount of extracted residue.

Figure 10 shows the change in the amount of residue which was electrolytically extracted from the specimens aged and tested at 600°C. The amount of residue starts to increase at 100h and is saturated at 10000h. The increase of the amount of residue was caused by the precipitation of carbide $M_{23}C_6$. The times t_{Rd1} and t_{Rd2} corresponding to the rapid decrease of residual stress at 600°C are also represented in Fig.10. t_{Rd1} and t_{Rd2} correspond to the early stage and the last stage of precipitation and coarsening of $M_{23}C_6$, respectively.

In order to examine the relationship between precipitation state and stress relaxation behavior, the stress relaxation tests were conducted at 600°C and interrupted at 215 and 993h. 215h is earlier than the time at which the residual stress rapidly decreases under the total strain condition of 0.20%, and 993h is later than the time at which residual stress

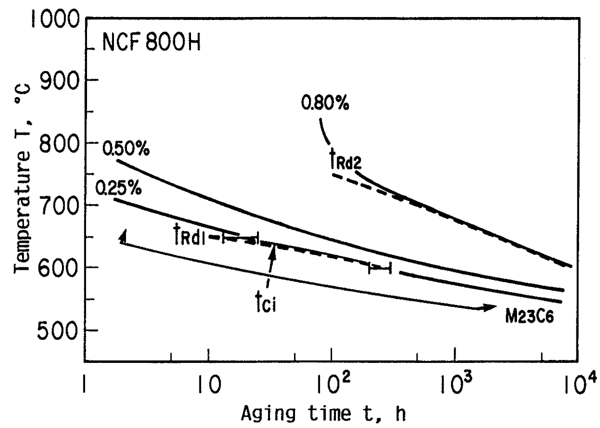

Fig.9. Comparison of t_{Rd1} and t_{Rd2} of relaxation curve and t_{Ci} of creep curve with amount of electrolytically extracted reside of $M_{23}C_6$.

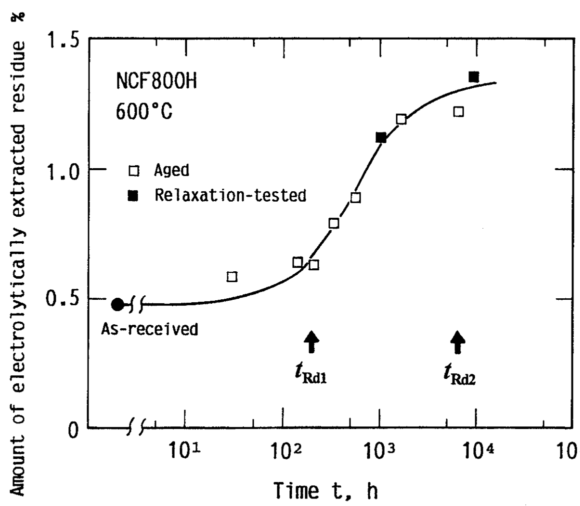

Fig.10. Change in amount of electrolytically extracted residue of materials aged and stress-relaxation-tested at 600°C, and t_{Rd1} and t_{Rd2} of stress relaxation curve at 600°C.

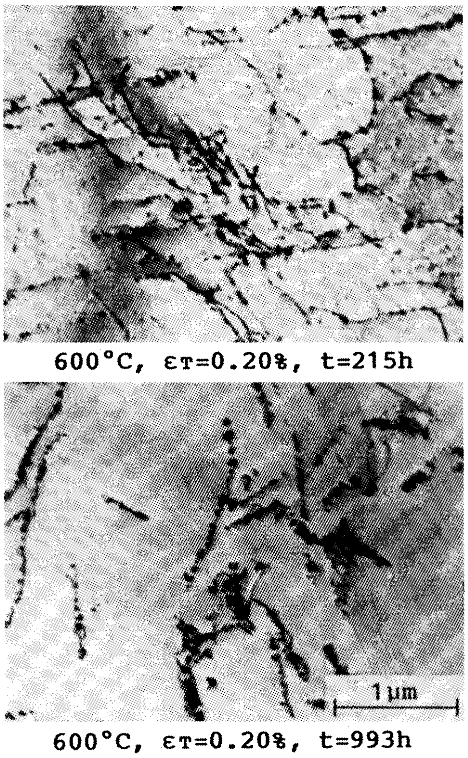

Fig.11. TEM micrograph for NCF 800H alloy stress-relaxation-tested at 600°C.

decreases rapidly. The sample was cut down from the interrupted test pieces to observe using transmission electron microscopy. The results are shown in Fig.11. From TTP diagram of Fig. 8, the electrolytically extracted residue at 215h was regarded to be only $M_{23}C_6$ which might be collected by 1μm filter. However, very small $M_{23}C_6$ was observed. These precipitates are deposited on dislocations. On the other hand, $M_{23}C_6$ at 993h was more coarse than that at 215h, and the dislocation tangle seems to be less. From these results, the rapid decrease of residual stress for 300h at 600°C and for 10h at 650°C is caused by the reduction of creep deformation resistance due to coarsening of $M_{23}C_6$ carbides.

3.5. Minus Relaxation Behavior

The increase of residual stress was observed after the rapid decrease of residual stress at 600, 650 and 700°C, as shown in Fig.1. This minus relaxation behavior was reported also on SUS 316 steel[5]. It was caused by shrinking of the testing material due to the drop of carbon content in matrix accompanying the formation of $M_{23}C_6$.

The amount of electrolytically extracted residue increases during aging and testing as shown in Fig.10. This increase of the residue results from the precipitation of $M_{23}C_6$. The minus relaxation behavior is observed during the increasing period of the residue. The minus relaxation behavior of this material tested must be related to shrinking due to the drop of solute carbon content. The increase of residual stress was calculated from the change in lattice parameter due to the solution of carbon. The increase of lattice parameter of γ phase due to the solution of carbon is estimated by 0.0033wt%C(nm)[15,16]. The amount of $M_{23}C_6$ which was precipitated during the testing period of 1000 to 7000h at 600°C is estimated to be 0.17% from Fig.10. If all of the carbide are $Cr_{23}C_6$, the reducing amount of solute carbon content due to this precipitation is 0.0097wt%. As the lattice parameter and Young's modulus of this material are 0.36 nm and 150GPa, the increase of stress due to shrinking is estimated to be 13MPa. The observed increase of residual stress was 13.5MPa for 0.20% in total strain at 600°C, and the estimated value agreed well with the experimental one. The minus relaxation behavior was caused by shrinking of material due to the drop of solute carbon content due to the precipitation of $Cr_{23}C_6$.

3.6. Effect of Aging on Stress Relaxation Behavior

The residual stress was affected by the microstructural changes during stress relaxation tests. The stress relaxation behavior was tested on the material which had been aged previously. Figures 12 and 13 show the comparison of stress relaxation curve for aged material with that for as-received material. For the aged material, no rapid decrease of residual stress due to coarsening of $M_{23}C_6$ and little minus relaxation behavior are observed. The long-term residual stress for as-

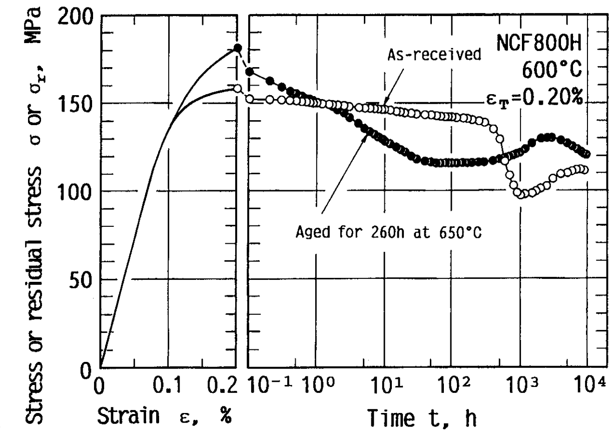

Fig.12. Effect of aging for 260h at 650°C on stress relaxation behavior at 600°C.

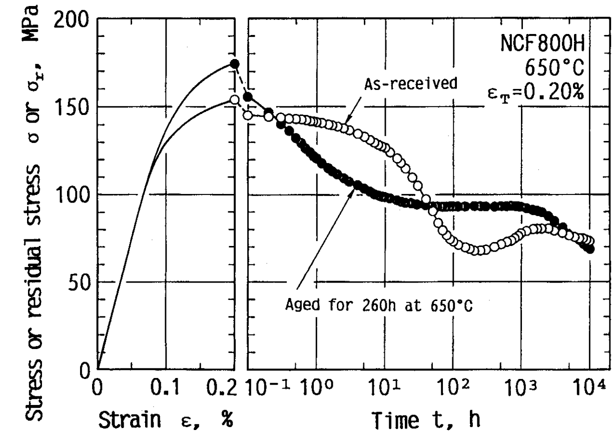

Fig.13. Effect of aging for 260h at 650°C on stress relaxation behavior at 650°C.

received material becomes equal to that for aged material. The decrease of residual stress at longer times, i.e., the behavior corresponding to longer than t_{Rd2} which is represented in Fig.1, is considered to be the behavior under stable condition after the transient microstructural change.

4. CONCLUSIONS

The long-term stress relaxation behavior was investigated on NCF 800H alloy at 600 to 750°C. The results are summarized as follows :

(1) The complicated behavior such as the rapid decrease of residual stress and the minus relaxation was observed.

(2) The rapid decrease of residual stress at 600 and 650°C was caused by the reduction of creep deformation resistance due to coarsening of $M_{23}C_6$.

(3) The minus relaxation behavior was caused by shrinking of material due to the drop of solute carbon content because of the precipitation of $Cr_{23}C_6$.

(4) The complicated stress relaxation behavior resulted from the transient phenomena due to precipitation and coarsening of $M_{23}C_6$. Therefore, it is difficult to extrapolate a long-term stress relaxation curve from the result of short-term test. The long-term residual stress value should be predicted using aged material.

REFERENCES

1. M. Schirra and K. Anderko, Steel Research, No. 6, (1990)242.
2. M. Sakamoto, K. Yagi, H. Morisita, K. Kubo, Y. Monma and C. Tanaka, J. Soc. Mater. Sci. Japan, **39** (1990)674.
3. F. E. Asbury, Mater. Sci. and Tech., **2** (1986)1123.
4. B. Weiss and R. Sticker, Metal. Trans., **3** (1972)851.
5. T. Ohba, K. Yagi, C. Tanaka and K. Kubo, J. Soc. Mater. Sci. Japan, **36** (1987)117.
6. K. Yagi and F. Abe: Proc. of 6th Int. Conf. on Creep and Fatigue, IMechE, London, (1996)41.
7. K. Yagi and F. Abe, J. Japan Soc. for Simulation Technology, **15** (1996)3.
8. ASME Boiler and Pressure Vessel Code, Section III, Division I, Case N47-17(1979).
9. K. Aoto, T. Koakutsu, Y. Wada and M. Hirano, Proc. of Int. Conf. on Creep, Tokyo, (1986)495.
10. K. Kussmaul, W. Gaudig and K. Maile, Proc. of ASME Conf. on High Temperature Constitutive Modeling, Theory and Application, Atlanta, December 1-6, (1991).
11. T. Ohba, O. Kanemaru, K. Yagi and C. Tanaka, J. Soc. Mater. Sci. Japan, **39** (1990)888.
12. A. A. Tavassoli and G. Colombe, Metal. Trans. A, **8A** (1977)1577.
13. C. Tanaka and T. Ohba , Trans. NRIM, **20** (1978)138.
14. J. Orr, Proc. of Petten Int. Conf. on Alloy 800, (1978)25.
15. N. Ridley, H. Stuaurt and L. Zwell, Trans. Metal. Soc. of AIME, **245** (1969)1834.
16. N. Ridley, J. Iron & Steel Inst., **209** (1971)396.