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CREEP RUPTURE PROPERTIES UNDER VARYING
LOAD/TEMPERATURE CONDITIONS
ON A NICKEL-BASE HEAT-RESISTANT ALLOY
STRENGTHENED BY BORON ADDITION

September 1993

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and Hajime NAKAJIMA

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A series of constant load & temperature creep rupture tests and varying load & temperature creep rupture tests was carried out on Hastelloy XR whose boron content level is 60 mass ppm at 900 and 1000°C in order to examine the behavior of the alloy under varying load and temperature conditions.

The life fraction rule completely fails in the prediction of the creep rupture life under varying load and temperature conditions though the rule shows good applicability for Hastelloy XR whose boron content level is below 10 mass ppm. The modified life fraction rule has been proposed based on the dependence of the creep rupture strength on the boron content level of the alloy. The modified rule successfully predicts the creep rupture life under the test conditions from 1000°C to 900°C. The trend observed in the tests from 900°C to 1000°C can be qualitatively explained by the mechanism that the oxide film which is formed during the prior exposure to 900°C plays the role of the protective barrier against the boron dissipation into the environment.

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Keywords: HTGR, Nickel-base Alloy, Heat-resistant Alloy, Hastelloy XR,
Helium Coolant, Creep, Creep Damage Rule, Life Fraction Rule,
Modified Life Fraction Rule, Boron Addition, Boron Dissipation

ホウ素添加によって強化したニッケル基耐熱合金の
荷重／温度変動条件下におけるクリープ破断特性

日本原子力研究所東海研究所材料研究部

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(1993年8月12日受理)

60 mass ppmのホウ素を添加してクリープ強度を高めたニッケル基耐熱合金ハステロイ XR を供試材料として、一連の荷重及び温度一定クリープ破断試験並びに温度／応力変動を伴う2段クリープ破断試験を900～1000℃域の高温ガス炉1次冷却材模擬ヘリウムガス中及び大気中で行った。前報のホウ素含有量が10 mass ppm未満のハステロイ XRでの挙動とは大きく異なり、累積損傷和則は温度／応力変動を伴う条件下のクリープ破断寿命の予測に適用できなかった。これは、試験片から雰囲気中へのホウ素の散逸現象が生じたことに起因することを確認するとともに、この現象による材料のクリープ強度の低下を考慮した修正累積損傷和則を提案した。この修正和則は1000℃から900℃へと変化させた場合のクリープ破断寿命の予測が可能であった。900℃から1000℃へと変化させた場合の挙動については、900℃において形成された酸化被膜が1000℃の状態におけるホウ素の散逸現象を遅らせる防護効果があると考えたと定性的な説明がつく。

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1. Introduction

In the course of Japanese research and development of high-temperature gas-cooled reactors (HTGRs), the High-Temperature Engineering Test Reactor (HTTR) was planned to be constructed as the first reactor, and it is currently under construction at Oarai Research Establishment of the Japan Atomic Energy Research Institute. A nickel-base heat-resistant alloy, Hastelloy XR was developed as the structural material for high-temperature components of the HTTR [1-3]. Hastelloy XR has the common basal composition for the major constituents with Hastelloy X (i.e., nominally Ni - 22 mass% Cr - 18 mass% Fe - 9 mass% Mo), while minor elements such as Mn and Si are adjusted in the optimum ranges and Al, Ti and Co are eliminated to the lowest possible levels [1,3]. As the result of the above-mentioned modification, Hastelloy XR is known to have the substantially improved corrosion resistance in the simulated HTGR helium gas environment and the improved applicability to the HTTR relative to Hastelloy X [1-3]. It is also known that the creep properties of Hastelloy XR depend strongly on the boron content level [4,5]. The boron content level is determined to be equal or less than 100 mass ppm in the specification of Hastelloy XR [1]. Even within the range, Hastelloy XR shows the very different creep rupture strength according to the amount of boron content as indicated in Fig.1 [4].

Creep data of Hastelloy XR have been accumulated both in simulated HTGR helium gas and in air environments for the design and safety evaluation of the high-temperature components of the HTTR. Creep rupture properties under varying stress and/or temperature conditions have also been examined on the alloy, and it has been concluded that the life fraction rule [6,7] is applicable in the engineering design of high-temperature components made of Hastelloy XR [8-11]. This conclusion has been drawn based on the data for the version whose boron content level is below 10 mass ppm.

Considering the balance of the creep properties and weldability of

Hastelloy XR, the version whose boron content level is 40 to 60 mass ppm is the most preferable [2,4,5]. Since the applicability of the life fraction rule depends strongly on the material, different behavior may be observed under varying stress and temperature conditions between the versions whose boron content levels are different.

Hence in the present study, a series of constant load & temperature creep rupture tests and varying load & temperature creep rupture tests was carried out on Hastelloy XR whose boron content level is 60 mass ppm at 900 and 1000°C in order to examine the behavior of the alloy under varying load and temperature conditions.

2. Experimental Procedures

2.1 Material

The material tested in this study is Hastelloy XR bar commercially manufactured. The chemical composition and the tensile properties of the material are listed in Tables 1 and 2, respectively. As shown in Table 1, the boron content level of the material is 60 mass ppm. The material was prepared by hot forging followed by solution annealing at 1190°C and rapid cooling.

2.2 Creep Rupture Tests

Creep rupture tests were accomplished by using specimens with 6 mm in diameter and 30 mm in gauge length. The tests were performed with lever-type creep testers. Some tests were carried out in simulated HTGR helium gas environments, and others in air. The test section used for the tests in simulated HTGR helium gas environments was encased in a vacuum tight chamber connected to a circulating helium loop system. A series of constant load & temperature creep rupture tests and varying load & temperature creep rupture tests was conducted at 900 and 1000°C.

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Two types of simulated HTGR helium gas were employed, and the impurity contents in the gas are indicated in Table 3. The characterizations of these environments have been reported elsewhere [12], and they are essentially equivalent from the viewpoints of the chemical reactions between the material and the environment [12].

3. Results and Discussion

3.1 Constant Load and Temperature Creep Rupture Tests

Table 4 summarizes the constant load & temperature creep rupture test conditions and the test results. Figure 2 shows the relation between the applied stress, which is a nominal stress given by the load divided by the initial cross-sectional area, and the time to rupture obtained with the constant load and temperature creep rupture tests. In the figure, not only the data obtained in the present study but also those obtained for the same heat material in another work [13] are plotted. There is no significant difference in the creep rupture life among three test environments within the range tested. The relation between the applied stress and the time to rupture can be expressed as follows;

$$t_R = 3.849 \times 10^{13} \sigma^{-6.390} \quad \text{at } 900^\circ\text{C}, \quad (1)$$

$$\text{and } t_R = 1.225 \times 10^8 \sigma^{-3.964} \quad \text{at } 1000^\circ\text{C}, \quad (2)$$

where t_R is the time to rupture in hour and σ is the applied stress in MPa.

3.2 Varying Stress and Temperature Creep Rupture Tests

Table 5 summarizes the varying stress & temperature creep rupture test conditions and the test results. Figures 3 and 4 show the results of application of the life fraction rule to the varying stress and

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temperature test results. In these figures, the life fraction $\Delta t_1/t_R$ was obtained using the calculated values with eqs. (1) and (2) as t_R . As can be seen in these figures, the life fraction rule completely fails in the prediction of the creep rupture life under varying load and temperature conditions in contrast with the previous studies [8-11]. The data tend to locate in the higher portion than the line which shows the summation of the life fractions is equal to unity in Fig. 3, i.e., in the tests from 900°C to 1000°C. On the contrary, all the data are located in the lower portion than the line which shows the summation of the life fractions is equal to unity in Fig. 4, i.e., the tests from 1000°C to 900°C.

3.3 Possible Sources of Changes in Creep Strength

In general, the life fraction rule shows good applicability to the material whose creep strength is not strongly affected by the change of the chemical composition and/or the microstructure of the material which occurs during exposure to the high-temperature environment. In the present study, the followings may be taken into consideration as the change of the chemical composition and/or the microstructure which may occur during exposure to the high-temperature environment, and may affect the creep strength;

- 1) change of carbon content,
- 2) change of boron content,
- 3) change of chromium content,
- 4) precipitation of carbides, and
- 5) recrystallization.

It is known that the creep strength of Hastelloy XR strongly depends on the carbon content level [14,15], and that carburization or decarburization may occur in the helium gas environment which contains specific levels of impurities for the alloy [14,15]. However in the environments employed in the present study, Hastelloy XR indicates protective corrosion behavior, and only mild carburization, which does not affect the creep strength of the alloy significantly [14,15], occurs [12].

As already mentioned in section 1, the creep strength of Hastelloy XR depends strongly on the boron content level (see Fig. 1). If the amount of boron content decreases during exposure to the high-temperature environment, the creep strength of the alloy will reduce.

It is also known that the chromium depleted zone is formed near the surface region of the material by exposure to the high-temperature simulated HTGR helium gas environments [3]. Such a phenomenon might cause the creep strength reduction. The depth of the chromium depleted zone, however, is estimated below 200 μ m even in the condition where Δt_1 is the longest at 1000°C. The value is very small relative to the diameter of the creep specimen, i.e., 6 mm, employed in the present study [16]. The phenomenon, therefore, does not affect the creep strength of Hastelloy XR significantly [17].

During exposure to the high-temperature test environment, carbides precipitate and may grow in Hastelloy XR. Precipitation of carbides is considered to contribute the creep strength of the material, and there may be the most preferable amount and size of precipitates for the contribution. The amount and the size of precipitates are considered to depend on the exposed temperature more strongly than on the applied stress level. This mechanism, therefore, might play the role in impairing the reliability of the life fraction rule.

Recrystallization might occur in Hastelloy XR crept at 1000°C [18]. Such a phenomenon might cause the creep strength reduction. Recrystallization occurs more easily in the version whose boron content level is below 10 mass ppm than the material tested in the present study [19]. Since this mechanism did not affect the creep strength of the alloy significantly even in the version whose boron content level is below 10 mass ppm, the good applicability of the life fraction rule was observed in the previous studies [8-11].

Among the above-mentioned five items, the items 1), 3), 4) and 5) are common to both the present and the previous studies [8-11], while the item 2) is specific to the present study. The item 2), therefore, is the most possible as the factor which caused the difference in the

applicability of the life fraction rule between the present and the previous studies [8-11], i.e., the versions whose boron content levels are different.

3.4 Results of Boron Analyses

In order to examine the changes in the amount of boron content, boron analyses were made for the crept specimens. In some cases the specimens for boron analyses were separated into two portions, i.e., the core and the surface portions as shown in Fig. 5, and in other cases the specimens were used for boron analyses in all. The specimens for boron analyses were selected from the crept ones indicated in Figs. 2 through 4, and some specimens were newly prepared conducting interruption creep tests under constant load and temperature conditions.

Figures 6 and 7 show the results of boron analyses as a function of the exposure time to the high-temperature environments at 900 and 1000°C, respectively. In the figures only one stage test results are shown, and no results under varying load and temperature conditions are indicated. It is clearly recognized that the amount of boron content reduces with increasing the exposure time at 1000°C in Fig. 7. Even at 900°C such a tendency is observed in Fig. 6. The boron content reduction rate at 900°C, however, is much lower than that at 1000°C. The amount of boron content at the surface portion tends to be smaller than that at the core portion, i.e., open symbols are generally located in the lower portion than solid ones in Figs. 6 and 7. The fact suggests that boron in the material dissipates from the surface as the result of the reaction with the high-temperature environments and that boron is supplied by the diffusion flow from the core portion to the surface.

Figure 7 suggests that the amount of boron content of the material crept at 1000°C in the first stage is lower than the original level at the point at which the test conditions were changed from 1000°C to 900°C. This mechanism may play the major role in impairing the reliability of the life fraction rule in Fig. 4, in which the creep lives in the

second stage are extremely short.

3.5 Modification of Life Fraction Rule

It is preferable to apply the evaluation method which takes account of the change of the chemical composition and/or the microstructure of the material which occurs during exposure to the high-temperature environment for the precise prediction of the creep rupture life under varying load and temperature conditions [20,21]. Since the above-mentioned mechanism seems to play the major role in impairing the reliability of the life fraction rule, the modification of the rule may be effective based on the dependence of the creep rupture strength on the boron content level of the alloy. In order to simplify the discussion, the followings are assumed:

- 1) The boron content reduction at 900°C is negligible.
- 2) There is no difference in the creep rupture life among three environments employed in the present study.
- 3) The bulk boron content level is a mean value of the boron analyzed results of the core and the surface portions.
- 4) The creep rupture life of the material whose boron content level is below 60 mass ppm becomes shorter along the line A linearly in a logarithm scale in Fig. 8. In the figure, the creep rupture data for Hastelloy XR whose boron content level is 1 to 2 mass ppm were obtained in the previous studies [8-11].

Adding the results of boron analyses for the specimens crept under varying load and temperature conditions to Fig.7 based on the assumption 1), Fig. 9 was obtained. According to the assumption 1), the amount of boron content after creep rupture tests under the conditions from 1000°C to 900°C can be regarded as the boron content level of the material at the point when the test conditions were changed from 1000°C to 900°C. Consequently the creep rupture life of the material whose boron content level is equal to that of the material at the point when the test conditions were changed from 1000°C to 900°C can be estimated based on the

assumptions 2), 3) and 4).

Figure 10 shows the result of the application of the above-mentioned method. Comparison of Fig. 10 with Fig. 4 clearly shows that the creep rupture life under varying load and temperature conditions is predicted more successfully by the modified life fraction rule based on the dependence of the creep rupture strength on the boron content level than by the life fraction rule.

By the way, the tendency is observed that the boron content reduction rate under the conditions from 900°C to 1000°C is low in Fig. 9. The tendency suggests that the prior exposure to 900°C could delay the phenomenon of boron dissipation during the following exposure to 1000°C. The oxide film which is formed on the surface of the specimen during the exposure to 900°C might play the role of the protective barrier against the boron dissipation into the environment. Since eq.(2) expresses the relation between the applied stress and the time to rupture in the case where the amount of boron content of the material reduces gradually, the creep rupture life would be longer in the case where the boron content reduction rate is low than that calculated with eq.(2). This mechanism can qualitatively explain the trend that the data are located in the higher portion than the line which shows the summation of the life fractions is equal to unity in Fig. 3, i.e., in the tests from 900°C to 1000°C.

4. Conclusions

A series of constant load & temperature creep rupture tests and varying load & temperature creep rupture tests was carried out on Hastelloy XR whose boron content level is 60 mass ppm at 900 and 1000°C in order to examine the behavior of the alloy under varying load and temperature conditions. Based on the results obtained the following conclusions are drawn:

- (1) The life fraction rule completely fails in the prediction of the

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- (1) The life fraction rule completely fails in the prediction of the

creep rupture life under varying load and temperature conditions though the rule shows good applicability for Hastelloy XR whose boron content level is below 10 mass ppm.

- (2) The change of boron content level of the material during the tests is the most probable source of impairing the reliability of the life fraction rule.
- (3) The modified life fraction rule has been proposed based on the dependence of the creep rupture strength on the boron content level of the alloy. The modified rule successfully predicts the creep rupture life under the test conditions from 1000°C to 900°C.
- (4) The trend observed in the tests from 900°C to 1000°C can be qualitatively explained by the mechanism that the oxide film which is formed during the prior exposure to 900°C plays the role of the protective barrier against the boron dissipation into the environment.

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Table 1 Chemical composition of the material tested (mass%).

C	Si	Mn	P	S	Cr	Co	Mo	W	Fe	B	Al	Ti	N	Ni
0.07	0.33	0.88	<0.001	0.001	21.99	0.06	8.73	0.63	17.80	0.006	0.03	0.01	0.006	Bal.

Table 2 Tensile properties of the material tested at room temperature and at temperatures where creep rupture tests were conducted.

Test Temp.	Strain Rate	0.2 % Proof Stress	Ultimate Tensile Strength	Total Elongation	Reduction of Area
R T	5 × 10 ⁻³ %/s (first stage)	289 MPa	685 MPa	56.9 %	60.9 %
	1.25 × 10 ⁻¹ %/s (second stage)				
Strain rate was changed at a strain of 3 %.					
900°C	5 × 10 ⁻³ %/s	127 MPa	133 MPa	109.7 %	78.1 %
	2.5 × 10 ⁻² %/s	160 MPa	161 MPa	91.3 %	81.4 %
	1.25 × 10 ⁻¹ %/s	175 MPa	204 MPa	120.7 %	89.8 %
1000°C	5 × 10 ⁻³ %/s	72 MPa	75 MPa	93.0 %	66.6 %
	2.5 × 10 ⁻² %/s	89 MPa	109 MPa	119.3 %	79.3 %
	1.25 × 10 ⁻¹ %/s	116 MPa	145 MPa	104.2 %	82.1 %

Table 3 Impurity levels in simulated HTGR helium gas environments (vol ppm).

	H ₂	H ₂ O	CO	CO ₂	CH ₄
He-1	200	1	100	2	5
He-2	300	3	100	1	15

Table 4 Summary of constant load & temperature creep rupture test conditions and test results.

Temp. (°C)	σ (MPa)	Environment	t_R (h)	Ratio of t_R to value of regression equation
900	39.2	Air	3632.7	1.43
	40.0	Air	2001.4	0.90
	42.0	Air	1367.2	0.84
	42.0	He-2	1392.5	0.85
	46.0	Air	636.7	0.70
	46.0	Air	666.5	0.73
	46.0	He-1	899.2	0.98
	46.0	He-1	984.4	1.08
	46.0	He-2	792.5	0.87
	53.9	Air	374.8	1.13
1000	15.0	Air	2922.5	1.10
	15.0	He-2	4263.8	1.60
	20.0	Air	893.6	1.05
	20.0	He-2	724.5	0.85

Table 5 Summary of varying stress/temperature creep rupture test conditions and test results.

Test No.	Env.	1st stage			2nd stage			Time to rupture $\Delta t_1 + \Delta t_2$ (h)
		Temp. ($^{\circ}$ C)	σ_1 (MPa)	Δt_1 (h)	Temp. ($^{\circ}$ C)	σ_2 (MPa)	Δt_2 (h)	
1	Air	900	42.0	171.9	1000	15.0	2564.9	2736.8
2	Air	900	42.0	312.0	1000	20.0	1029.0	1341.0
3	Air	900	42.0	352.0	1000	15.0	2854.2	3206.2
4	Air	900	42.0	1016.7	1000	15.0	527.4	1544.1
5	He-2	900	42.0	312.0	1000	20.0	1132.5	1444.5
6	He-2	900	42.0	816.0	1000	15.0	3360.1	4176.1
7	Air	1000	15.0	391.4	900	47.0	478.8	870.2
8	Air	1000	15.0	1000.0	900	42.0	425.7	1425.7
9	Air	1000	15.0	1000.6	900	42.0	350.1	1350.7
10	Air	1000	15.0	1994.4	900	42.0	171.9	2166.3
11	Air	1000	20.0	446.8	900	42.0	303.8	750.6
12	He-2	1000	15.0	1200.0	900	42.0	369.4	1569.4
13	He-2	1000	15.0	1944.0	900	42.0	144.2	2088.2

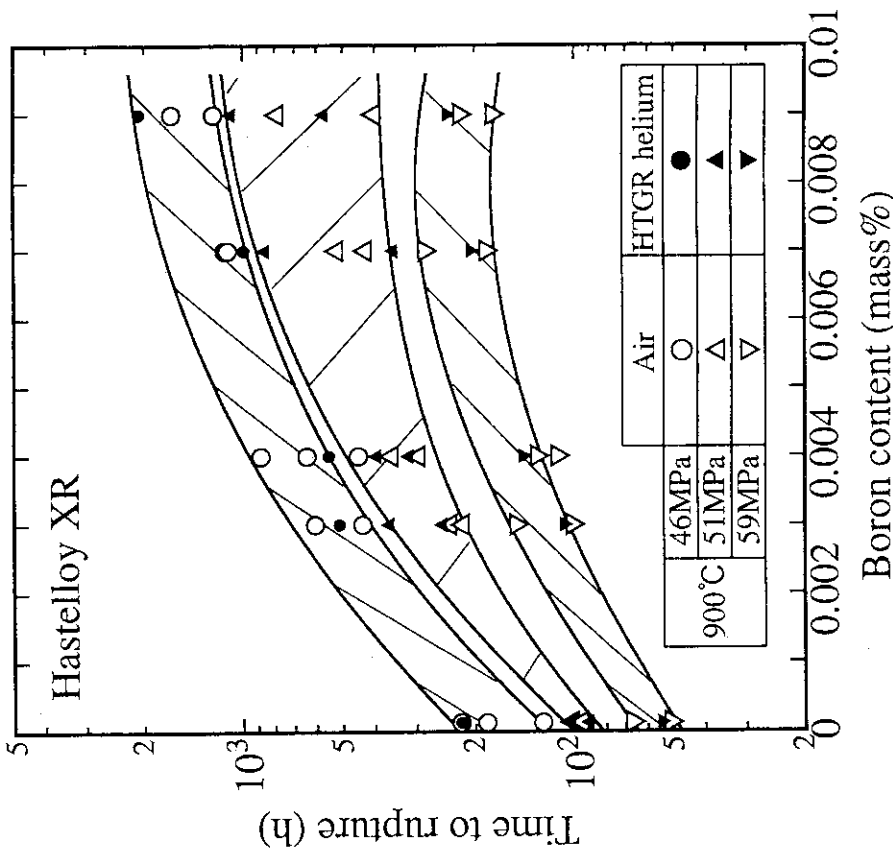


Fig. 1 Effect of the boron content level on the creep rupture life of Hastelloy XR [4].

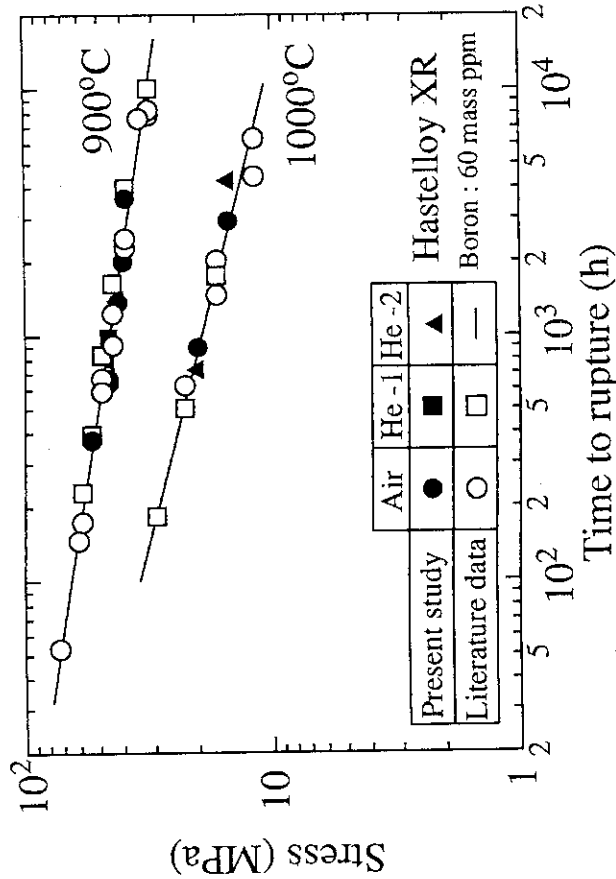
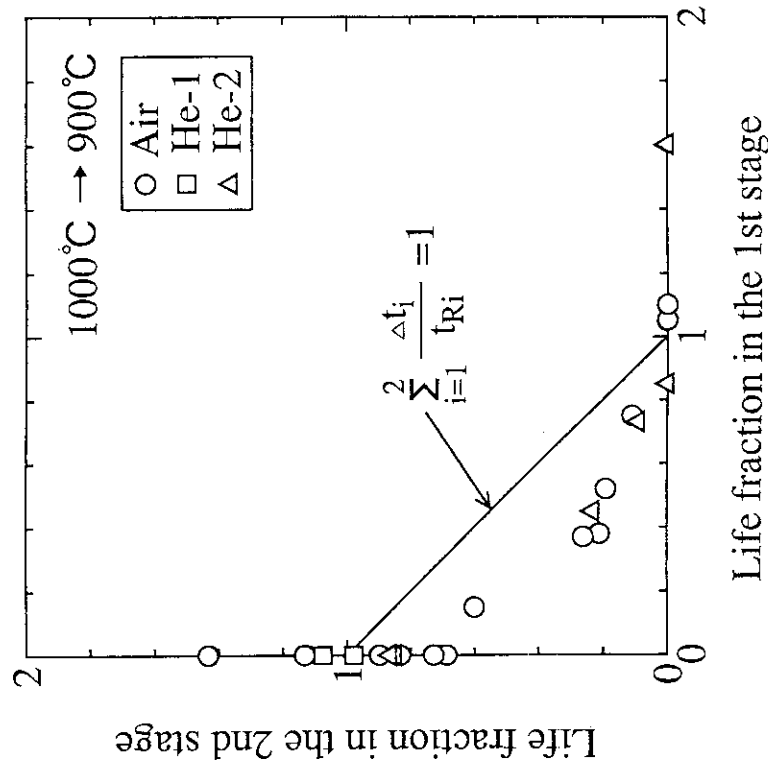
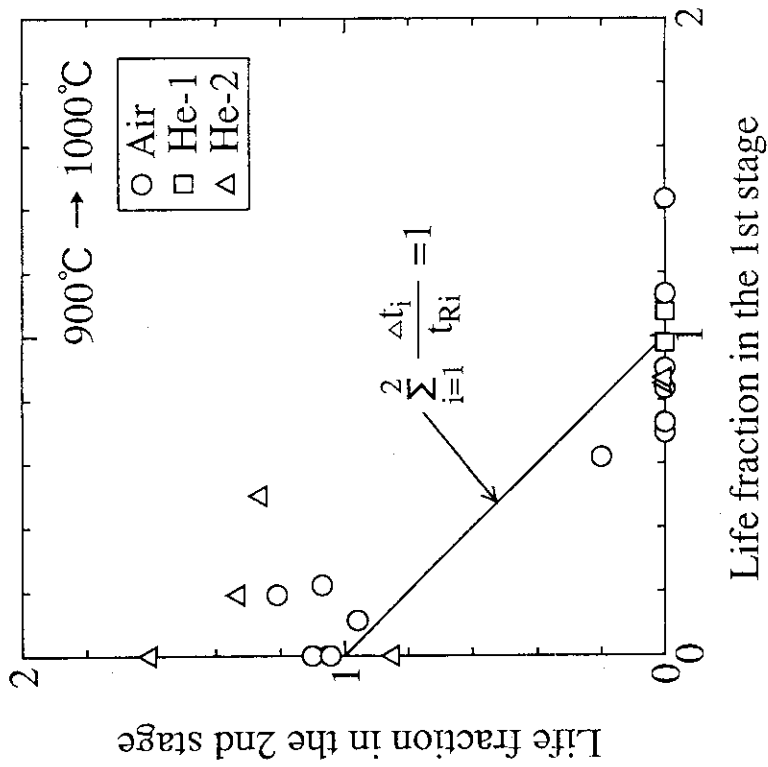


Fig. 2 Relation between applied stress and time to rupture obtained with constant load and temperature creep rupture tests for Hastelloy XR whose boron content level is 60 mass ppm. The figure includes both of the data obtained in the present study and in another work [13] for the same heat material.



Life fraction in the 1st stage



Life fraction in the 1st stage

Fig. 4 Result of application of the life fraction rule to the test results under the conditions from 1000°C to 900°C for Hastelloy XR whose boron content level is 60 mass ppm.

Fig. 3 Result of application of the life fraction rule to the test results under the conditions from 900°C to 1000°C for Hastelloy XR whose boron content level is 60 mass ppm.

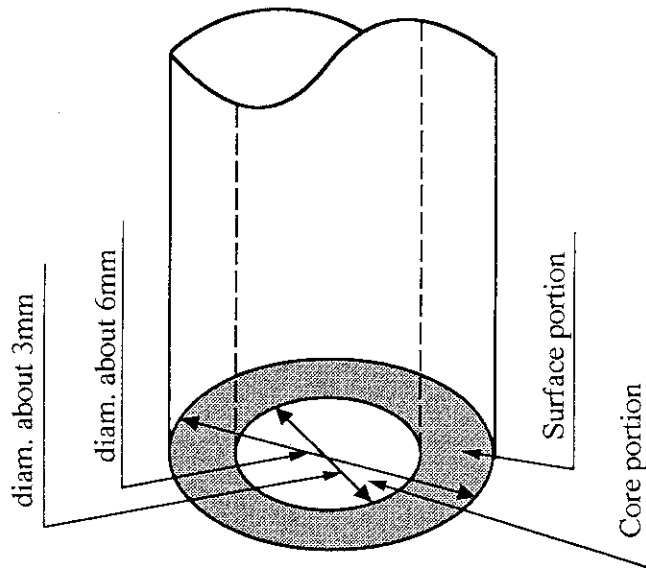
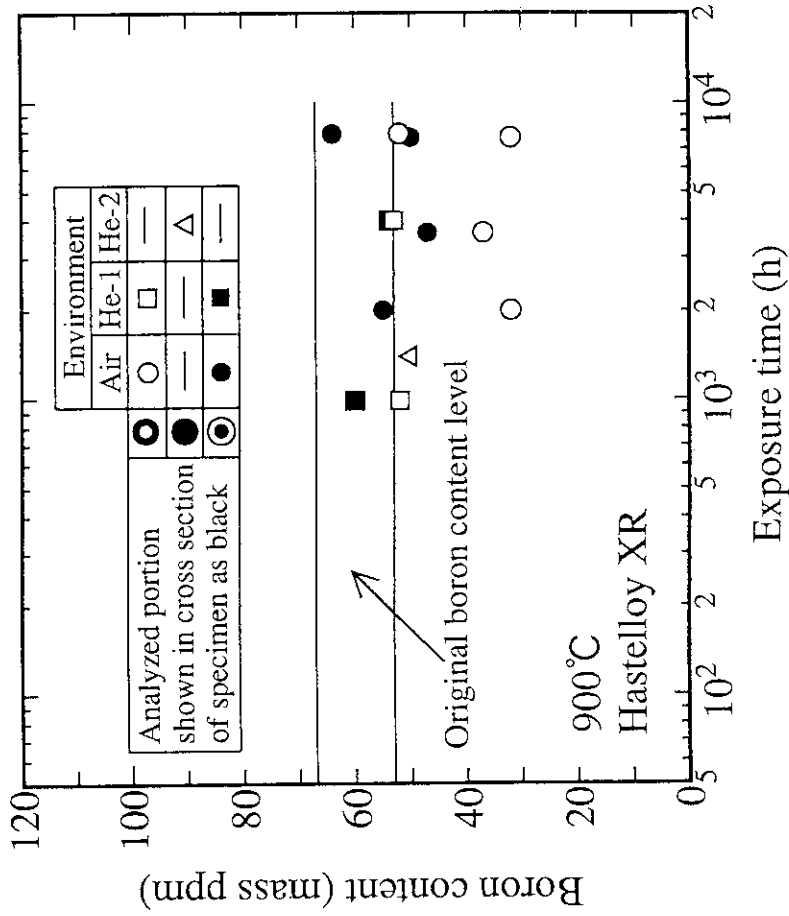


Fig. 5 Schematic illustration of the sampling method of specimens for boron analyses.

Fig. 6 Results of boron analyses as a function of the exposure time to the high-temperature environments at 900°C. The figure does not include the results under varying load and temperature conditions.

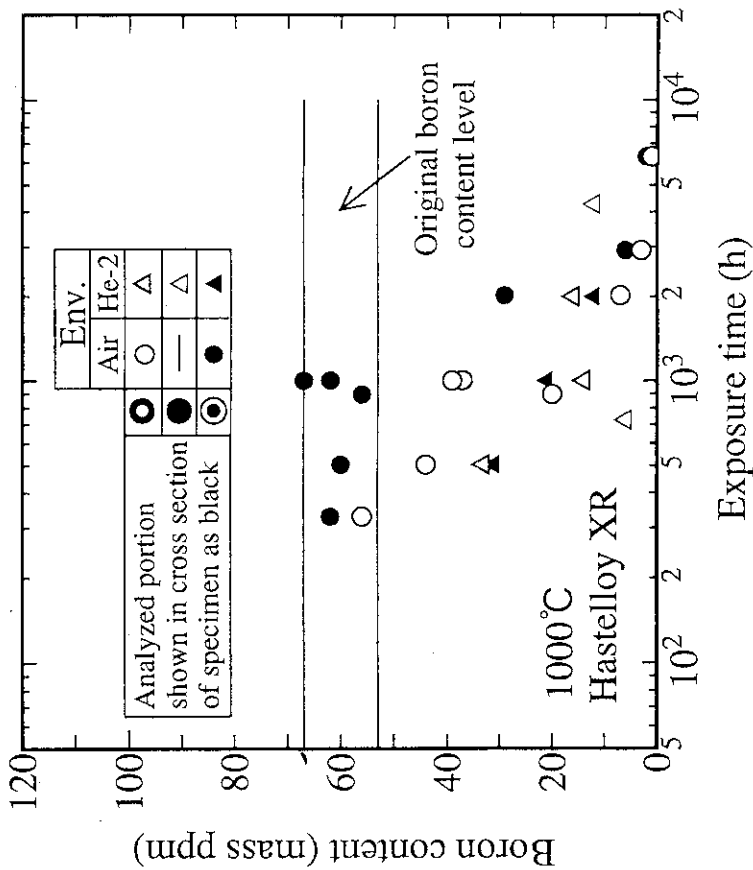


Fig. 7 Results of boron analyses as a function of the exposure time to the high-temperature environments at 1000 °C. The figure does not include the results under varying load and temperature conditions.

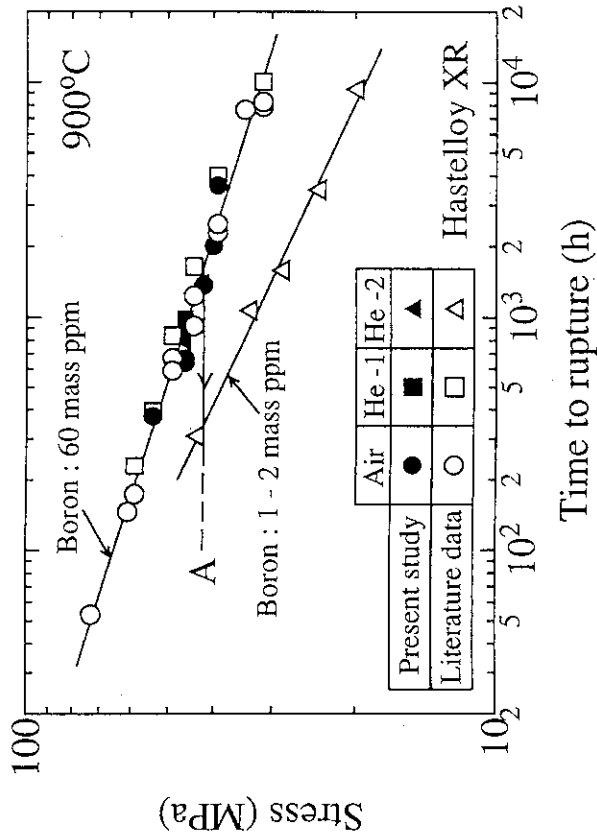


Fig. 8 Relation between applied stress and time to rupture obtained with constant load and temperature creep rupture tests for Hastelloy XR whose boron content levels are different [8-11,13].

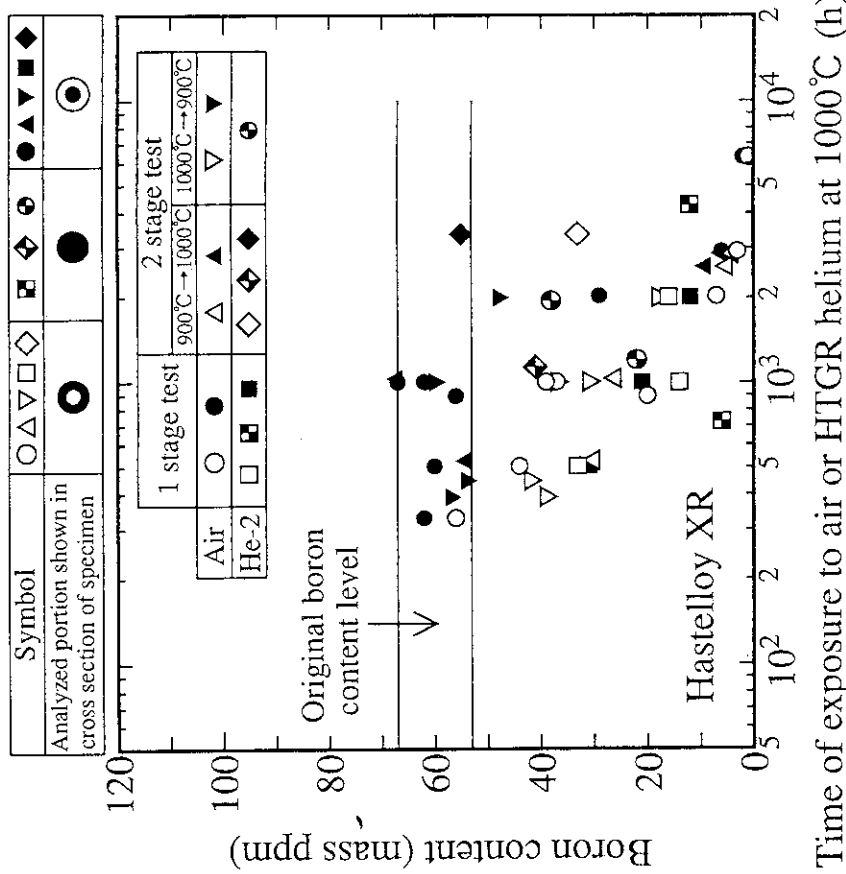


Fig. 9 Results of boron analyses as a function of the exposure time to the high-temperature environments at 1000°C. The figure includes the results under varying load and temperature conditions.

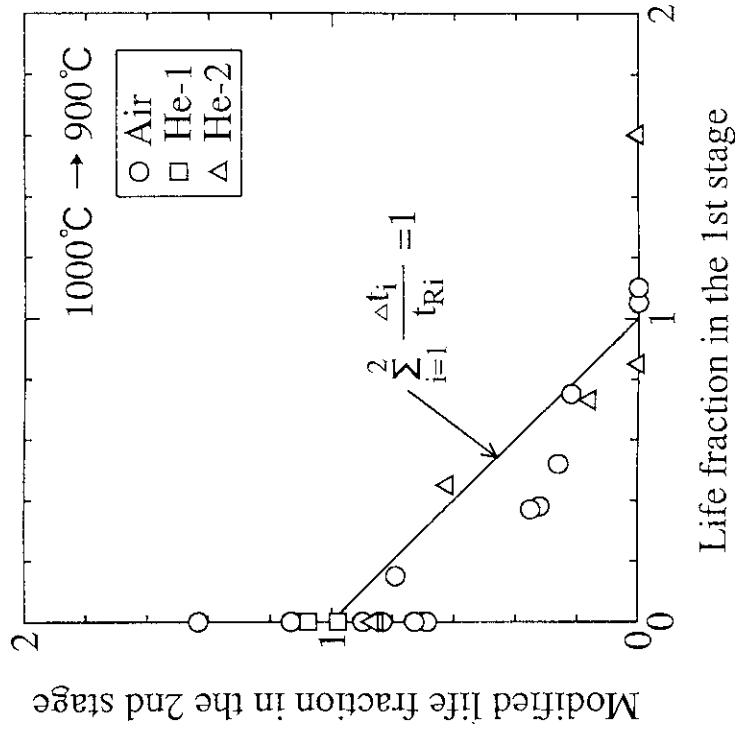


Fig. 10 Result of application of the modified life fraction rule to the test results under the conditions from 1000°C to 900°C for Hastelloy XR whose boron content level is 60 mass ppm.