Effects of Niobium, Titanium, Molybdenum, and Boron on Creep Rupture Strength of 16%Cr-10%Ni-V Steel*

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Synopsis

The influence of single and combined additions of molybdenum, niobium, boron, and titanium on the creep rupture strength and the rupture elongation of 16Cr-10Ni-0.4V heat resisting steel, and their correlation with microstructures have been studied.

Chemical compositions of the steels used are selected by the design of experiment and the relationship between the creep rupture strength and the chemical compositions is analysed by a computer. A regression equation is developed which satisfactorily predicts the 700°C-1 000 hr creep rupture strength from the chemical composition. The additions of molybdenum, niobium, and boron to this steel have a remarkable effect in increasing the creep rupture strength. Molybdenum, boron, and titanium additions are found to improve the creep rupture elongation.

It is presumed from electron microscopic studies that molybdenum accelerates slightly the coarsening of the precipitated carbide, but increases the creep rupture strength through the solid solution strengthening, and that niobium and boron make a distribution of precipitated carbide more uniform in matrix, this being one of the mechanisms of their strengthening effects.

1. Introduction

The increase of creep rupture strength by the addition of vanadium to 16Cr-10Ni austenitic steel has recently been reported by the authors.¹⁾ And it has also been found that a combined addition of titanium and niobium increases the creep rupture strength of the steel by the contents lower than that of stabilized steel, and that an addition of only 0.02% boron im-

proves both the strength and the rupture elongation.^{2,3)} Moreover, molybdenum has been known⁴⁾ as an alloying element which improves the creep rupture strength of the steel. A recent study⁵⁾ has pointed out that the decrease of the strength at the prolonged time in 18-10Mo (AISI 316) steel is correlated to the rapid coalescence of M₂₃C₆ carbide, and this decrease of the strength can be prevented by the combined addition of small amounts of titanium, niobium, and boron. From these aspects, the strength and the rupture elongation of the steels will be improved by the combined addition of those elements. The present paper is concerned primarily with the confirmation of the above prediction.

Many statistical methods⁶⁻⁹⁾ have been applied to the evaluation of the effects of combined additions. In this study, the statistical block arrangement method has been used to analyze the effect of the combined addition of these elements on the strength of the steel. For consideration on the fluctuation of the chemical compositions, the regression analysis has been made using a computer.

The observations of microstructures have also been made regarding the influences of alloying elements on the precipitated M₂₃C₆,¹⁰⁾ thread-like VC, and granular V₂C carbide.

Table 1. Chemical composition of steels used (wt%)

Steel	C	Si	Mn	Cr	Ni	V	Mo	B	Nb	Ti	N		
											Sol. N	Insol. N	Total N
A1	0.12	0.61	1.41	16.20	11.77	0.39	—	—	—	—	0.0458	0.0072	0.0530
A2	0.13	0.53	1.51	15.94	10.62	0.74	—	—	—	0.09	0.0121	0.0196	0.0317
A3	0.15	0.40	1.30	16.87	9.57	0.38	—	—	0.24	—	0.0272	0.0032	0.0304
A4	0.11	0.20	1.12	14.36	8.98	0.33	—	—	0.11	0.01	0.0477	0.0026	0.0503
A5	0.12	0.20	1.14	14.26	9.57	0.33	—	0.014	—	—	0.0303	0.0003	0.0306
A6	0.11	0.20	1.14	14.31	9.39	0.31	—	0.016	—	0.05	0.0450	0.0131	0.0581
A7	0.13	0.37	1.42	17.17	10.27	0.27	—	0.021	0.35	—	0.0513	0.0058	0.0571
A8	0.10	0.41	1.47	17.34	10.39	0.30	—	0.018	0.14	0.06	0.0252	0.0166	0.0418
A9	0.10	0.23	1.18	14.13	9.12	0.31	1.65	—	—	—	0.0255	0.0023	0.0278
A10	0.11	0.19	1.10	14.40	8.83	0.31	1.62	—	—	0.02	0.0477	0.0056	0.0533
A11	0.11	0.25	1.06	14.13	7.78	0.30	1.78	—	0.23	—	0.0359	0.0073	0.0432
A12	0.10	0.22	1.03	13.05	10.39	0.28	1.61	—	0.11	0.02	0.0372	0.0044	0.0416
A13	0.10	0.53	1.24	17.34	10.21	0.37	2.04	0.014	—	—	0.0331	0.0034	0.0365
A14	0.11	0.30	1.02	15.59	9.04	0.31	1.81	0.011	—	0.01	0.0446	0.0048	0.0494
A15	0.12	0.39	1.32	16.92	10.27	0.35	2.00	0.013	0.26	—	0.0451	0.0086	0.0537
A16	0.10	0.40	1.04	16.59	8.86	0.31	1.75	0.013	0.16	0.02	0.0336	0.0098	0.0444

P: 0.004 to 0.006%, S: 0.007 to 0.012%

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II. Materials and Experimental Procedures

The base composition of the steels was chosen to be 16%Cr–10%Ni–0.1%C–0.4%V, as a result of balancing the high temperature oxidation with the creep rupture strength. The amounts of additional elements were determined by referring to the previous studies as follows, Nb: 0.3%, Ti: 0.05%, Mo: 2%, and B: 0.02%. The two levels of these four elements, with and without the addition, were combined to make 16 steels which were melted by using an induction furnace and were cast in 5 kg ingots. Table 1 shows the chemical compositions of the steels. Steel A1 has the base composition of a series of the steels tested. After forging and solution treatment of 1 100°C–1 hr, creep rupture tests were carried out at 650° and 700°C. Short term tests were made by lever type single testing machines and long term tests by spring type multiple testing machines.¹²⁾ Metallographic studies were primarily done by electron microscopic examination of replicas prepared by a carbon extraction method; the film had been made by etching with Villela solution for the first stage and with 10% bromine alcohol solution for the second stage.

Almost all the solution treated steels had a small amount of undissolved carbide and about the same grain size numbers (ASTM) of 4 to 6.

III. Experimental Results

1. Result and Analysis of Creep Rupture Test

The creep rupture tests were performed at 650° and 700°C up to several thousands hours. The creep rupture curves and the elongation curves of steel A1 are shown in Fig. 1, as a typical example. 100 and 1 000 hr creep rupture strengths of all the steels were determined by the interpolation and are listed in Table 2. The values of 700°C–1 000 hr rupture

strength range from 10.3 to 17.0 kg/mm² and those values are fairly higher than that of the conventional 18–8 stainless steels. As seen in Fig. 1, the rupture elongation varies with rupture life, and interpolated values of 1 000 hr rupture elongations are also listed in Table 2.

Table 3 shows the results of variance analysis, which was calculated from the values listed in Table 2, with regard to the main effects of various additional elements and their interactions. The addition of molybdenum appeared to be effective on the rupture strengths of 100 and 1 000 hr at 650° and 700°C, and increased the rupture elongation. Boron was also effective on the rupture strength except for the data of 100 hr at 700°C, and increased the rupture elongation. Niobium improved the rupture strength except for the data of 100 hr at 650°C, and titanium increased the rupture elongation at 650°C, whereas it was not effective on the rupture strength. Some interactions of boron and titanium, and molybdenum and boron, for instance, contributed to the rupture strength, although these vanished for the 700°C–1 000 hr strength.

Fig. 1. Stress and rupture elongation vs. rupture time curves of steel A1 obtained at 650° and 700°C testing

Table 2. Creep rupture strength and elongation of the steels tested

Property Steel	Creep rupture strength (kg/mm ²)				1 000 hr creep rupture elongation (%)	
	650°C		700°C		650°C	700°C
	100 hr	1 000 hr	100 hr	1 000 hr		
A1	21.0	15.0	16.7	11.2	13	30
A2	24.2	17.6	15.4	10.3	35	44
A3	23.7	15.5	16.8	10.8	11	20
A4	26.6	19.8	18.5	11.8	7	30
A5	27.0	17.7	16.4	10.5	10	20
A6	27.5	17.8	17.2	11.4	24	35
A7	27.5	21.5	19.0	14.2	20	36
A8	27.0	19.6	16.7	12.6	26	46
A9	32.5	21.4	19.5	11.3	25	55
A10	34.0	23.2	20.4	13.2	27	48
A11	30.5	24.0	20.7	14.5	15	56
A12	33.6	25.0	21.8	14.0	21	50
A13	37.0	24.0	18.0	14.3	20	60
A14	31.0	22.2	22.7	16.6	38	50
A15	37.5	24.4	28.0	17.0	46	62
A16	38.0	23.5	22.7	14.5	40	60

The main effects of molybdenum, boron, and niobium on the 700°C–1 000 hr creep rupture strength of the steel are summarized in Fig. 2. In this figure, for example, the level 1 of molybdenum addition indicates the average value of 700°C–1 000 hr creep rupture strength of molybdenum free steels, and level 2 is that of molybdenum bearing steels. In this treatment, no influence of other factors is included on levels 1 and 2, because of the combination of factors by the statistical block arrangement. The same representations for the effects of boron and niobium are made in this figure. For molybdenum, the strength of level 2 is higher than that of level 1 by about 3 kg/mm². Similarly, the boron or niobium bearing steels have higher rupture strengths by 1.5 to 2 kg/mm² than that of the boron or niobium free steels.

By using Larson–Miller parameter, fairly long term strengths can be extrapolated from the short term strength at higher temperature. Percent increments in rupture strength resulted from the addition of alloying elements are shown in Fig. 3 as a function of Larson–Miller parameter, $P=T(20+\log t)$. It is obvious from this figure that the strengthening rate due to molybdenum addition is more than 10%, and this effect is larger than those of any other elements, though it decreases with increase of the value of P . Boron and niobium increase the rupture strength only about 5%.

As mentioned regarding Table 3, the 700°C–1 000 hr rupture strength can be represented only by main effects of molybdenum, boron, and niobium, and it is clear that the 700°C–1 000 hr rupture strength, $y(\text{kg/mm}^2)$ can be formulated by the following equation using average values of chemical compositions of steels, and the regression equation obtained is as follows:

$$y(\text{kg/mm}^2) = 10.25 + 1.36(\% \text{Mo}) + 117(\% \text{B}) + 6.7(\% \text{Nb}) \dots\dots\dots(1)$$

where (%Mo), (%B), and (%Nb) are the contents of

molybdenum, boron, and niobium in steel, respectively. A comparison of the experimental values and calculated values from Eq. (1) is shown in Fig. 4. Dotted lines indicate the confidence limits of 95%. As shown in the figure, the data of all the steels used in this study except for one case are well estimated by the above equation. Black symbols show the values of steels containing only molybdenum,⁵⁾ niobium,¹³⁾ or boron³⁾ without V as indicated in the figure. Since these steels contain no vanadium, the 700°C–1 000 hr

Fig. 2. Effect of molybdenum, niobium, and boron on the 1 000 hr creep rupture strength at 700°C (* 95%, ** 99%)

Fig. 3. Relationship between rate of increment in creep rupture strength due to molybdenum, boron, and niobium addition and Larson–Miller parameter

Table 3. Influence of chemical factors on the creep rupture properties

Property Factor		Creep rupture strength				1 000 hr creep rupture elongation	
		650°C		700°C		650°C	700°C
		100 hr	1 000 hr	100 hr	1 000 hr		
Main effect	Mo	**	**	**	**	**	**
	B		*		**	**	*
	Nb	**	**	**	*		
	Ti					**	
Interaction	Mo×B		*			*	
	B×Nb					**	**
	Mo×B×Nb	*					**
	Mo×Ti						**
	B×Ti	*	**				
	Nb×Ti					**	
	B×Nb×Ti			*			
	Mo×B×Nb×Ti					*	

* 95%, ** 99%

rupture strengths (y' kg/mm²) of these steels were calculated from the next equation instead of the Eq. (1), assuming that the effect of 0.3% vanadium corresponds to about +1 kg/mm².¹⁾

$$y'(\text{kg/mm}^2) = 9.25 + 2.9(\%V) + 1.36(\%Mo) + 117(\%B) + 6.7(\%Nb) \dots\dots\dots(2)$$

The strengths of niobium and boron bearing steels can be well estimated by this equation; that of molybdenum bearing steel, however, does not fit in this relation. This fact shows a probable interaction between molybdenum and vanadium. Therefore, the effects of this interaction and of other alloying elements on the rupture strength will be reestimated in the next section using a computer.

2. Regression Analysis

From the above mentioned results, it is presumed that the 700°C–1 000 hr rupture strength y'' (kg/mm²) can be represented by an equation of the form:

$$y'' = \alpha_0 + \sum \alpha_i x_i + \alpha_{V \times Mo} \times x_V x_{Mo} \dots\dots\dots(3)$$

where α_0 : constant
 α_i : coefficient of i th alloying element
 x_i : content of i th alloying element, wt%
 $\alpha_{V \times Mo}$: coefficient of interaction between V and Mo
And i represents the chemical elements: C, Si, Mn,

Cr, Ni, V, Mo, B, Nb, Ti, and N. By using the rupture strength data of 23 steels, which are listed in Tables 1 and 4, the coefficients were determined by a computer as follows:

$$y'' = 15.2 + 2.5(\%C) + 6.2(\%Si) + 1.5(\%Mn) + 0.46(\%Cr) - 0.23(\%Ni) + 1.8(\%V) + 1.3(\%Mo) + 96(\%B) + 8.8(\%Nb) + 3.2(\%Ti) + 71(\%N) + 0.88(\%V \times \%Mo) \dots\dots(4)$$

(± 1.2)

(± 8.2)

(± 22)

(± 4.8)

(± 3.9)

(± 0.50)

(± 0.58)

(± 2.5)

(± 0.8)

(± 58)

(± 3.3)

(± 14)

(± 32)

(± 2.7)

where the values in parentheses are the standard deviations. A comparison between the creep rupture strengths observed and calculated from Eq. (4) is shown in Fig. 5, where dotted lines in the figure indicate the range of the standard deviation. The coefficient of chromium is negative, while those of C, Si, Mn, V, Mo, B, Nb, Ti, N, and $V \times Mo$ are positive, and this indicates that all of these latter elements contribute to the strengthening of the steel. But the standard deviation of the coefficient for interaction between molybdenum and vanadium is fairly large compared with the value of the coefficient itself, the reason of which is not clear. In Fig. 5, it is found that only one point (steel A14, 0.31%V, 1.18%Mo, 0.011%B, 0.01%Ti) is apart from the range of standard deviation, the cause of which can not be explained

Fig. 4. Calculated *vs.* observed 700°C–1 000 hr creep rupture strengths of 16Cr–10Ni–0.4V steel containing various alloying elements. Calculated values were obtained from Eqs. (1) and (2).

Fig. 5. Calculated (Eq. (4)) *vs.* observed 700°C–1 000 hr creep rupture strengths of 16Cr–10Ni steel containing various alloying elements

Table 4. Chemical composition and creep rupture strength of additional steels used for constructing Fig. 5

Steel	Chemical composition (wt%)											700°C–1 000 hr creep rupture strength (kg/mm ²)	References
	C	Si	Mn	Cr	Ni	V	Mo	B	Nb	Ti	N		
S1	0.10	0.53	1.24	17.98	9.92	—	0.44	0.009	—	0.03	0.0322	11.0	(3)
S2	0.10	0.60	1.57	17.73	10.00	—	—	—	0.29	—	0.0189	10.9	(13)
S3	0.09	0.61	1.57	17.81	10.00	—	—	—	0.29	—	0.0156	10.8	(13)
S4	0.08	0.45	1.56	18.98	10.51	—	2.40	—	—	0.01	0.0200	9.0	(5)
S5	0.06	0.57	1.48	18.49	11.15	—	—	—	—	—	0.0185	5.8	(14)
S6	0.11	0.77	1.53	18.66	11.26	—	—	—	—	—	0.0147	6.5	(14)
S7	0.11	0.62	1.45	16.20	11.66	0.75	—	—	—	—	0.0530	12.5	(1)

even by the following microstructural observations.

The coefficient for carbon in Eq. (4) seems to be too small; this is due to the narrow range of the carbon concentration as shown in Tables 1 and 4.

3. Microstructures

The typical replica electron micrographs of steels A1 to A8 and A9 to A16 are shown in Photos. 1 and 2, respectively. Here, changes of only the precipitated carbides are mentioned as follows. The microstructure of A1 (Photo. 1 (a)) consists of carbide $M_{23}C_6$ on grain boundaries, and thread-like VC and granular V_2C in matrix. In steel A2 (Photo. 1 (b)) granular TiC carbides are observed, and the same shape carbides are also observed in the other titanium bearing steels, (b), (d), (f), and (h), the influence of these carbide on precipitation of $M_{23}C_6$ is, however, not so remarkable. Photograph 1 (c) of the niobium bearing steel shows the granular NbC carbides. By comparing the microstructures of niobium bearing steels ((d), (g), (h)) with those of niobium free steels ((a), (b), (f)), it can be seen that the niobium addition promotes the matrix precipitation of $M_{23}C_6$. The micrograph of boron bearing steel (Photo. 1 (e)) shows the finely

V_2C in matrix. In steel A2 (Photo. 1 (b)) granular TiC carbides are observed, and the same shape carbides are also observed in the other titanium bearing steels, (b), (d), (f), and (h), the influence of these carbide on precipitation of $M_{23}C_6$ is, however, not so remarkable. Photograph 1 (c) of the niobium bearing steel shows the granular NbC carbides. By comparing the microstructures of niobium bearing steels ((d), (g), (h)) with those of niobium free steels ((a), (b), (f)), it can be seen that the niobium addition promotes the matrix precipitation of $M_{23}C_6$. The micrograph of boron bearing steel (Photo. 1 (e)) shows the finely

(a) A1	11 kg/mm ²	946 hr	(b) A2 (Ti)	10 kg/mm ²	1 114 hr
(c) A3 (Nb)	12 kg/mm ²	676 hr	(d) A4 (Nb+Ti)	12 kg/mm ²	870 hr
(e) A5 (B)	12 kg/mm ²	457 hr	(f) A6 (B+Ti)	12 kg/mm ²	650 hr
(g) A7 (B+Nb)	12 kg/mm ²	624 hr	(h) A8 (B+Ti+Nb)	12 kg/mm ²	1 139 hr

Photo. 1. Electron micrographs of creep ruptured specimens at 700°C of steels A1 to A8 (extraction replica)

and uniformly dispersed $M_{23}C_6$, indicating that the boron also promotes the matrix precipitation of $M_{23}C_6$. By comparing the corresponding micrographs of Photo. 1 (molybdenum free) and Photo. 2 (molybdenum bearing), it appears that the effect of molybdenum on the precipitation behavior is not so clear, except that it slightly acceralates the coalescence of precipitated $M_{23}C_6$.

IV. Discussion

As expected, the strengthening effects of molybdenum, boron, and niobium on the 16Cr-10Ni-0.4V

austenitic steel have been found in the present study. These are, however, additional strengthening effects on the steel strengthened by vanadium addition, which is worthy of attention. The highest 700°C-1 000 hr rupture strength, 17 kg/mm², was obtained in steel A15; this value was fairly larger than that of the conventional 18-8 stainless steel.

From the results of metallographical examinations, some interpretations of the alloying elements will be deduced as follows. Molybdenum appears to contribute to the rupture strength by solid solution strengthening, while it has only a slight effect on the

(a) A 9 (Mo)	12 kg/mm ²	751 hr	(b) A10 (Mo+Ti)	15 kg/mm ²	420 hr
(c) A11 (Mo+Nb)	15 kg/mm ²	876 hr	(d) A12 (Mo+Nb+Ti)	15 kg/mm ²	583 hr
(e) A13 (Mo+B)	15 kg/mm ²	572 hr	(f) A14 (Mo+B+Ti)	15 kg/mm ²	2 070 hr
(g) A15 (Mo+B+Nb)	15 kg/mm ²	1 713 hr	(h) A16 (Mo+B+Nb+Ti)	15 kg/mm ²	861 hr

Photo. 2. Electron micrographs of creep ruptured specimens at 700°C of steels A9 to A16 (extraction replica)

precipitated $M_{23}C_6$ and does not form a special carbide. The contribution of niobium to the rupture strength is a promotion of matrix precipitation of $M_{23}C_6$ associated with NbC particles. As mentioned in section III. 3, the effect of boron also comes mainly from the uniformly precipitated $M_{23}C_6$. In this study, the effect of titanium was too small to be detected, a cause of which was probably due to the too small amount of titanium addition.

It is very difficult to obtain the exact chemical compositions that have been designed before melting. In addition to those deviations from the designed values, the contents of other minor elements not added are also different in each steel depending on melting conditions. In many previous studies, therefore the effects of the content fluctuations of minor elements were neglected. By the application of the regression analysis, however, it becomes possible to estimate the effects of an unintentional change in chemical composition. There are many problems in the method of estimation using the regression analysis, and the exactness of the estimation depends on the accuracies of chemical analysis and of creep rupture test and the number of data. It seems impossible to put many workers' data in this equation, because the properties of materials are influenced by production processes such as melting method, raw materials used, heat treatment, and so on. But, if a large number of data may be obtained with the same production process, this method of regression analysis becomes more useful for the estimation of the effects of the alloying elements on the creep rupture strength.

Many studies^{2,14,15)} have been reported, in which the estimations of the effects of alloying elements on creep rupture strength of the austenitic steels are done. Some of these results agreed with the regression equation obtained by the present study, but there are many exceptions. The causes of these exceptions are mainly attributed to the difference in base compositions and in content of alloying elements.

It has been known that in many cases the rupture strength has no linear relation with the amounts of alloying elements of the steel, and each element makes a different contribution to the carbide precipitation in matrix. These facts suggest that the relation between creep rupture strength and amounts of alloying elements is very complex.

V. Conclusions

Using the statistical method, the effects of addition of 2% molybdenum, 0.02% boron, 0.3% niobium, and 0.05% titanium on the creep rupture strength and rupture elongation of 16%Cr–10%Ni–0.1%C–0.4%V steel at 650° and 700°C have been analyzed and the conclusions obtained are as follows:

(1) The strengthening effects of molybdenum, niobium, and boron were confirmed in the vanadium bearing steel, and especially, addition of 2% molybdenum increased the rupture strength by about 10%, whereas additions of 0.3% niobium and 0.02% boron increased the rupture strength only about 5%. The strengthening effect of molybdenum decreased slightly

with increasing of Larson–Miller parameter $P=T(20+\log t)$. The highest value of 700°C–1 000 hr creep rupture strength obtained in present study was 17.0 kg/mm². The addition of titanium showed no influence to the rupture strength within the range of this investigation.

(2) To improve the rupture elongation, additions of molybdenum, boron, and titanium were found to be effective.

(3) A following regression equation was developed which satisfactorily predicted the 700°C–1 000 hr creep rupture strength from the chemical composition.

$$\begin{aligned} \sigma''(\text{kg/mm}^2) = & 15.2 + 2.5(\%C) + 6.2(\%Si) \\ (\pm 1.2) & (\pm 8.2) (\pm 22) (\pm 4.8) \\ & + 1.5(\%Mn) - 0.46(\%Cr) - 0.23(\%Ni) \\ & (\pm 3.9) (\pm 0.50) (\pm 0.58) \\ & + 1.8(\%V) + 1.3(\%Mo) + 96(\%B) \\ & (\pm 2.5) (\pm 0.8) (\pm 58) \\ & + 8.8(\%Nb) + 3.2(\%Ti) + 71(\%N) \\ & (\pm 3.3) (\pm 14) (\pm 32) \\ & + 0.88(\%V \times \%Mo) \\ & (\pm 2.7) \end{aligned}$$

(4) From electron replica microscopy, molybdenum was found to accelerate slightly the coalescence of the precipitated $M_{23}C_6$, and it was presumed that this element contributed to the strength of the steel by solid solution strengthening. Niobium precipitated in matrix as NbC, which contributed to the strength by promoting the matrix precipitation of $M_{23}C_6$. Boron improved also the distribution of carbides $M_{23}C_6$ in the matrix. Titanium showed no significant influence on the microstructure and creep rupture strength because of too small addition in this study.

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