

# Microstructural and Mechanistical Factors Affecting Creep-fatigue Properties of Heat-resisting Cast Steels

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## ABSTRACT

A small amount of Zr, Nb, and/or Ti addition has been found to improve the low-cycle-fatigue life of HK40 and HP cast alloys under creep conditions. In particular, the effect of Zr is marked. From a metallurgical viewpoint, the improving mechanism could be attributed to the interaction between dislocations and a number of fine MC-type carbides, such as ZrC. The interaction suppresses the formation of sub-boundaries and therefore reduces the precipitation of coarse  $M_{23}C_6$ -type carbides on the boundaries, which become crack propagation paths. Mechanistically, LCF life could be determined by partitioned strain-energy parameters, which are derived from fatigue and creep J-integrals for a cracked material.

## KEYWORDS

Creep-fatigue interaction; microstructure; carbide; strain-energy parameter; J-integral; heat-resisting centrifugal cast tube alloy.

## INTRODUCTION

Since tubes used in fuel-cell reformer furnaces are exposed to extremely high temperatures and subjected to frequent thermal cyclings, high creep-fatigue resistance is required for the tube materials. Although several new alloys have been developed for petrochemical reformers, such as HP-Nb, and HP-BST-M, mainly from the point of creep strength, there is little available data which supports the applicability of these materials for the fuel-cell reformer (Yokomaku et al., 1986, 1988).

In the present study, the creep-fatigue properties of various heat-resisting

cast alloys were investigated to find appropriate materials for the above purpose. As a result, Nb, Ti and, particularly, Zr were found to be effective in improving creep-fatigue properties. The mechanism of this improvement is discussed from both the metallurgical and the mechanistical aspects. Finally, a LCF life estimation method for these alloys, based on the fatigue and creep strain-energy density partitioning, is proposed.

### EXPERIMENTAL

#### Materials

Six cast alloys (see Table 1) manufactured by the centrifugal casting method were tested. Specimens were taken from the 200mm<sup>OD</sup> X 15mm<sup>t</sup> tubes in the longitudinal direction.

Table 1. Chemical compositions (wt%)

Materials	C	Si	Mn	Cr	Ni	Nb	Ti	Zr
HK40	0.43	0.88	0.52	24.4	21.3	--	--	--
HK-Zr	0.41	0.86	0.61	24.0	23.1	--	--	0.24
HP	0.48	1.19	0.78	25.5	34.3	--	--	--
HP-Nb	0.45	0.99	1.33	24.8	35.1	1.38	--	--
HP-BST-M	0.49	0.90	0.81	24.9	35.0	0.72	0.10	--
HP-Zr	0.48	1.18	0.54	25.7	34.8	--	--	0.35

#### Low-Cycle-Fatigue and Fatigue Crack Propagation Test

Low-cycle-fatigue tests were conducted on smooth specimens (8mm in diameter, 15mm in gauge length) at 800 and 1000 °C. Two types of strain waveforms were used; continuous cycles, and tension-hold cycles (hold period = 15min). A strain rate of 2%/sec and a strain ratio of -1 were used in both waveforms.

Crack propagation tests were performed on center notch specimens (16mm in width, 4mm in thickness) at 800 °C under load control mode. Continuous and tension-hold waveforms (hold period = 5 min) were used, which had a ramp time from peak to peak of 0.5 sec, and a load ratio, R = 0. Fatigue and creep J-integrals,  $\Delta J_r$  and  $\Delta J_c$ , were measured from load(P) versus load line displacement(V) curves (Dowling, 1976, Taira et al., 1981);

$$\Delta J_r = \Delta K^2/E + S_p / \{B(W-2a)\} \quad (1)$$

$$\Delta J_c = \{(n_c-1)/(n_c+1)S_c\} / \{2B(W-2a)\} \quad (2)$$

where,  $\Delta K$ : stress intensity factor range, E: Young's modulus, B: specimen

thickness, W: specimen total width, a: a half crack length,  $S_p$  and  $S_c$ : areas of fatigue and creep portion in P-V curve,  $n_c$ : stress exponent in creep equation,  $\dot{\epsilon}_c = k\sigma^{n_c}$ ;  $n_c=7.1$  for HK40 and HK-Zr, 11.1 for others.

### RESULTS

#### Low-Cycle-Fatigue Properties

Under continuous cyclings, Zr-addition considerably improved LCF life of HK40 and HP, but Nb and Ti had small effect (see Fig.1(a)). On the other hand, all these elements showed marked life extension under tension-hold waveforms. Zirconium was the most effective for life improvement, followed by (Nb, Ti) and Nb (see Fig.2(b)).

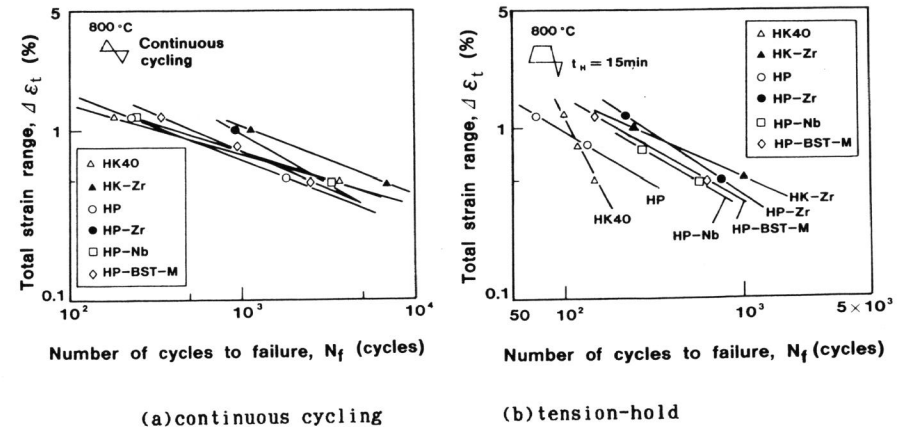


Fig.1 Total strain range vs LCF life

#### Fatigue Crack Propagation Properties

The relationship between  $da/dN$  and J-integral ranges,  $\Delta J_r$  and  $\Delta J_c$ , is shown in Fig.2. Almost the same  $da/dN$ - $\Delta J_r$  relations were obtained for all materials, but  $da/dN$ - $\Delta J_c$  relation was largely dependent on materials. In particular, Zr-containing alloys had an excellent crack propagation resistance under tension hold waveforms. The crack propagation rate can be expressed by;

$$da/dN = C_r \Delta J_r^{m_r} \quad (m_r = 1.5) \quad (3)$$

$$da/dN = C_c \Delta J_c^{m_c} \quad (m_c = 1.1) \quad (4)$$

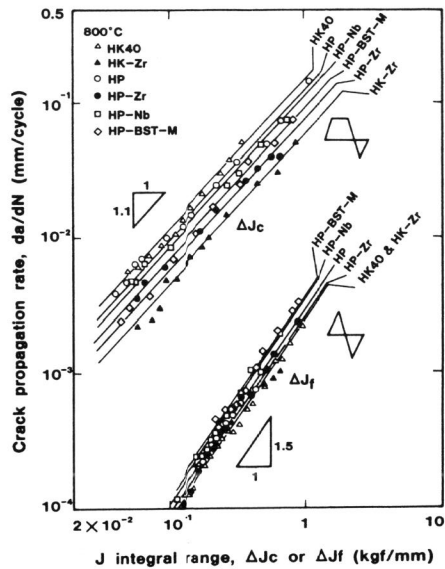


Fig. 2. Crack propagation rate,  $da/dN$ , vs fatigue and creep J-integral range,  $\Delta J_f$  and  $\Delta J_c$

## DISCUSSION

### Metallurgical Aspects in Creep-Fatigue Failure

The microstructures of tension-hold LCF test specimens are shown in Fig.3. Voids and cracks initiated at matrix/eutectic carbide boundaries and propagated along them. Cracks also propagated along sub-boundaries, on which coarse carbides ( $M_{23}C_6$ ) precipitated during testing. Few sub-boundaries were observed in Zr, Nb and Ti-containing materials with long LCF life.

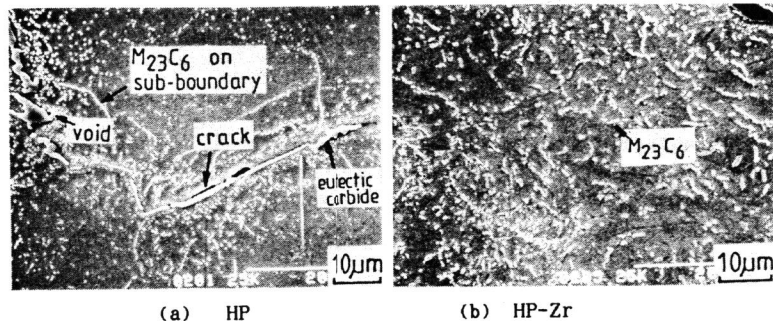


Fig. 3.  $M_{23}C_6$  precipitation and crack propagation during tension-hold LCF test at 800 °C

The role of Zr, Nb and Ti can be explained as follows; because these elements form a number of fine MC-type carbides which are dispersed evenly in the matrix, the uniform dislocation structure is maintained for a long time (see Fig.4). As a result, the number of intensive sub-boundaries and coarse  $M_{23}C_6$ -type carbides on the sub-boundaries is very small. Since ZrC is much smaller than NbC and TiC in size, Zr-addition is especially effective for improving creep-fatigue properties.

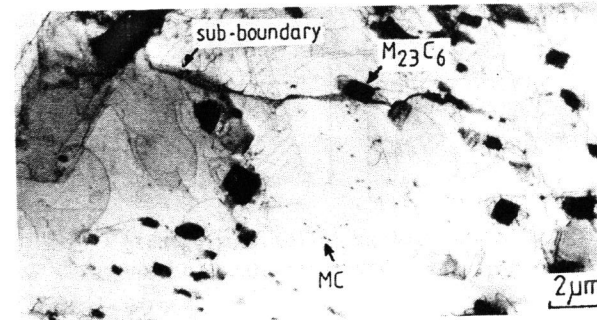


Fig.4 Dislocations, fine and coarse carbides after tension-hold LCF test at 1000 °C (HP-Zr)

### Mechanistic Aspects of Creep-Fatigue Failure

From the metallurgical observations mentioned above, crack propagation resistance is thought to be the main factor affecting LCF life. The crack propagation life in the LCF test was evaluated below, based on the fracture mechanics equations expressed by Eqs.3 and 4 and strain energy parameters derived from J-integral (Ohtani, et al., 1988).

Fatigue Strain Energy Parameter  $\Delta \tilde{W}_f$  and LCF Life The fatigue J-integral range,  $\Delta J_f$ , is given in terms of the fatigue strain energy parameter,  $\Delta \tilde{W}_f$ ;

$$\Delta J_f = 2 \pi M_J \Delta \tilde{W}_f a \quad (5)$$

$$\Delta \tilde{W}_f = \Delta W_e + \{ (n_f + 1) / 2 \pi \} f(n_f) \Delta W_p \quad (6)$$

where,  $M_J$ ; correction factor (0.51 for a semi-circular surface crack),  $n_f$ ; inverse of cyclic strain hardening exponent (5 for tested materials),  $a$ ; crack length. The function,  $f(n_f)$ , and elastic and plastic strain energy densities (see Fig. 5),  $\Delta W_e$  and  $\Delta W_p$ , are given by;

$$f(n_f) = 3.85 n_f (1 - 1/n_f) + \pi / n_f \quad (7)$$

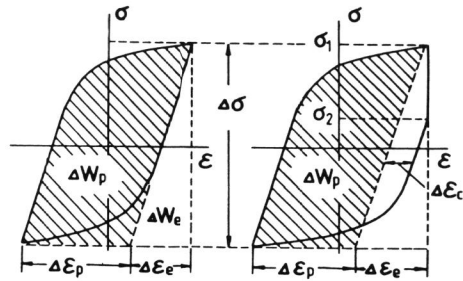
$$\Delta W_e = \Delta \sigma \Delta \epsilon_e / 2, \quad \Delta W_p = \Delta \sigma \Delta \epsilon_p / (n_f + 1) \quad (8)$$

From Eqs.3 and 5, crack propagation life,  $N_f$ , is derived as follows;

$$(\Delta \tilde{W}_f)^{m_f} N_f = D_f \quad (9)$$

$$D_r = \frac{a_0^{1-m_r} - a_r^{1-m_r}}{C_r (m_r - 1) (2 \pi M_J)^{m_r}} \quad (10)$$

where,  $a_0$ ; initial crack length,  $a_r$ ; final crack length.



(a) continuous cycling (b) tension-hold  
Fig. 5 Stress-strain hysteresis loop in LCF test

Creep Strain Energy parameter  $\Delta \tilde{W}_c$  and LCF Life The creep J-integral range,  $\Delta J_c$ , in strain-hold cycle, can be derived by the analogy of elasto-plastic analysis. As the stress changes from  $\sigma_1$  to  $\sigma_2$  in strain-hold portion (see Fig.5), the elastic J-integral change,  $\Delta J_e'$ , is given by;

$$\Delta J_e' = 2 \pi M_J \Delta W_e' \quad (11)$$

$$\Delta W_e' = (\sigma_1^2 - \sigma_2^2) / 2E \quad (12)$$

The creep J-integral,  $J_c'$ , at time  $t$  under steady creep state is given by;

$$J_c' = M_J f(n_c) \sigma(t) \dot{\epsilon}_c(t) a \quad (13)$$

In the case of stress-hold (ie.  $\bar{\sigma} = \text{const.}$ ), the creep J-integral change,  $\Delta J_c'$ , can be readily obtained by integrating Eq.13 as follows;

$$\Delta J_c' = 2 \pi M_J \left\{ [(n_c+1)/2 \pi] f(n_c) \Delta W_c' \right\} a \quad (14)$$

$$\Delta W_c' = \bar{\sigma} \Delta \epsilon_c / (n_c+1) \quad (15)$$

In the case of strain-hold, however,  $\Delta J_c'$  cannot be obtained easily because stress changes with time, but it can be calculated approximately by applying average stress value during strain-hold,  $\bar{\sigma} = (\sigma_1 + \sigma_2) / 2$ , to Eq.14. Thus, the total J-integral change,  $\Delta J_c (= \Delta J_e' + \Delta J_c')$ , during strain-hold is given in terms of the creep strain energy parameter,  $\Delta \tilde{W}_c$ ;

$$\Delta J_c = 2 \pi M_J \Delta \tilde{W}_c a \quad (16)$$

$$\Delta \tilde{W}_c = \Delta W_e' + \left\{ (n_c+1) / 2 \pi \right\} f(n_c) \Delta W_c' \quad (17)$$

From Eqs.4 and 16, crack propagation life by strain-hold cycles,  $N_c$ , is derived as follows;

$$(\Delta \tilde{W}_c)^m N_c = D_c \quad (18)$$

$$D_c = \frac{a_0^{1-m_c} - a_r^{1-m_c}}{C_c (m_c - 1) (2 \pi M_J)^{m_c}} \quad (19)$$

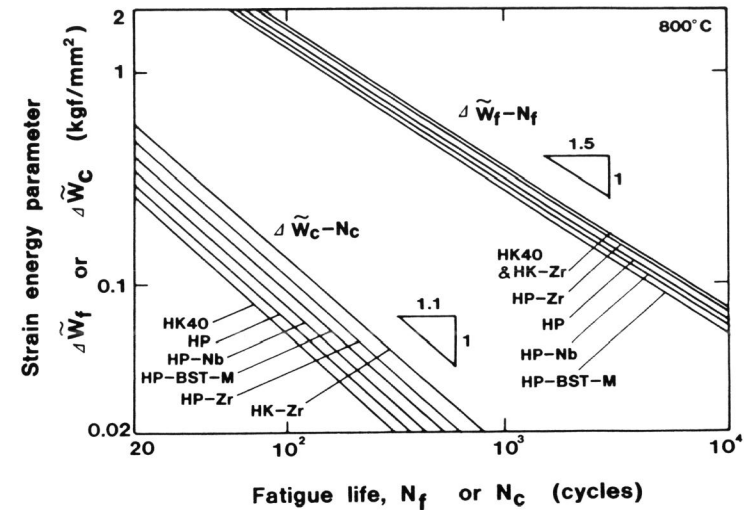


Fig. 6. Strain energy parameter vs life curves estimated by crack propagation law

#### Life Estimation by Strain Energy Partitioning Method

The  $\Delta \tilde{W}_r - N_r$  and  $\Delta \tilde{W}_c - N_c$  life relationships, calculated from Eqs.9 and 18 using material constants,  $C_r$ ,  $C_c$ ,  $m_r$ , and  $m_c$ , are shown in Fig.6. Here, we assumed  $a_0 = 0.2\text{mm}$  and  $a_r = 2\text{mm}$ . Applying  $\Delta \tilde{W}_r$  and  $\Delta \tilde{W}_c$ , measured by stress-strain loops in the LCF test, to these relationships, the partitioned lives,  $N_r$  and  $N_c$ , can be obtained. In the case of the tension-hold test, the life can be estimated by the linear damage rule on partitioned lives;

$$1/N = 1/N_r + 1/N_c \quad (20)$$

The comparison between the measured and estimated LCF life is shown in Fig.7. Nearly all of the results fall within a factor of two of the observed lives, regardless of waveform types. This result indicates that the main part of LCF life is a crack propagation life and that the mechanical factors determining LCF life are  $\Delta \tilde{W}_r$  and  $\Delta \tilde{W}_c$ . The effect of  $\Delta \tilde{W}_c$  on life is so marked that the improvement of crack propagation resistance under creep conditions will lead to the LCF life extension of the material. More correct evaluation of  $\Delta \tilde{W}_c$  and the physical meaning of initial crack length,  $a_0$ , should be examined in the future.

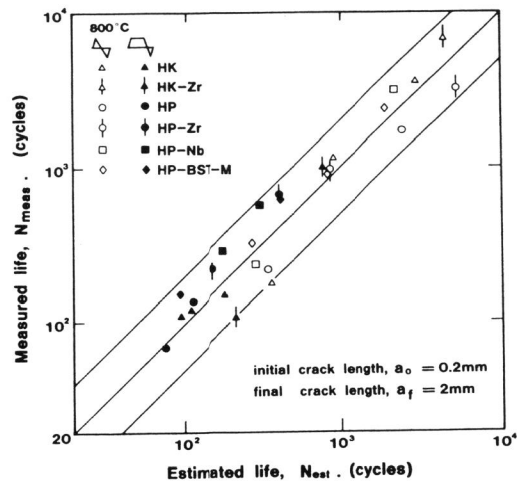


Fig. 7. Comparison between measured life and estimated life by strain energy partitioning method

#### CONCLUSIONS

Creep-fatigue properties of HK40, HP and the modified alloys were investigated. The main results are as follows;

- (1) Creep-fatigue properties were markedly improved by Zr, Nb, and/or Ti addition. The effect of Zr-addition was especially remarkable.
- (2) These elements formed fine secondary carbides (MC) in the matrix, and, accordingly, they suppressed the formation of intensive sub-boundaries and the precipitation of coarse carbides ( $M_{23}C_6$ ) on the sub-boundaries. As these boundaries can be crack propagation paths, Zr, Nb, and/or Ti addition will lead to excellent creep-fatigue properties.
- (3) Low-cycle-fatigue life could be well estimated by the strain energy partitioning method based on the fatigue and creep crack propagation laws.

#### REFERENCES

- Dowling, N.E. (1976). ASTM STP 601, pp. 19-32.
- Ohtani, R. and T. Kitamura (1988). ASTM STP 942, pp. 1163-1180.
- Taira, S., R. Ohtani, T. Kitamura and K. Yamada (1981). J. of Soc. Mat. Sci., Japan, **28**-308, pp. 414-420
- Yokomaku, T., M. Saori, T. Okuda, A. Nohara, H. Tai and Y. Imoto (1986). 1986 Fuel Cell Seminar, Tucson, Arizona, pp. 284-287.
- Yokomaku, T., M. Saori, T. Okuda and A. Nohara (1988). J. of Soc. Mat. Sci., Japan, **37**-414, pp. 308-314.