ELSEVIER

Journal of Nuclear Materials 278 (2000) 328–333

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

High temperature fatigue behaviour of TZM molybdenum alloy under mechanical and thermomechanical cyclic loads

H.J. Shi [a,*], L.S. Niu [a], C. Korn [b], G. Pluvinage [b]

[a] Department of Engineering Mechanics, Tsinghua University, Beijing 100084, People's Republic of China
[b] University of Metz, Ile du Saulcy, Metz 57045, France

Received 2 February 1999; accepted 23 August 1999

Abstract

High temperature isothermal mechanical fatigue and in-phase thermomechanical fatigue (TMF) tests in load control were carried out on a molybdenum-based alloy, one of the best known of the refractory alloys, TZM. The stress–strain response and the cyclic life of the material were measured during the tests. The fatigue lives obtained in the in-phase TMF tests are lower than those obtained in the isothermal mechanical tests at the same load amplitude. It appears that an additional damage is produced by the reaction of mechanical stress cycles and temperature cycles in TMF situation. Ratcheting phenomenon occurred during the tests with an increasing creep rate and it was dependent on temperature and load amplitude. A model of lifetime prediction, based on the Woehler–Miner law, was discussed. Damage coefficients that are functions of the maximum temperature and the variation of temperature are introduced in the model so as to evaluate TMF lives in load control. With this method the lifetime prediction gives results corresponding well to experimental data.

1. Introduction

The molybdenum-based alloy, TZM, is one of the best known of the refractory alloys for nuclear energy and aerospace systems such as for divertor of fusion reactors and spacecraft radiator panels [1,2]. With continued improvement in production and fabrication techniques, a wide variety of components can now be produced. Structural components of this alloy are often subjected to complex loads in different environment; however, additional information is required. Recently some studies of this aspect have been carried out. Filacchioni et al. [3] investigated the mechanical behaviour such as tensile strength, ductility and impact properties of the unirradiated TZM alloy. Singh et al. [4] investigated the mechanical behaviour and microstructural properties of the irradiated material. Hiraoka et al. [5] obtained the fracture and ductile-to-brittle transition characteristics of the material by impact and static bend tests. Maday [6] studied the low cycle fatigue behaviour of the material in high temperature water.

It is known that, in some structural applications, a component is subjected to the combination of cyclic mechanical loads with temperature variations. In this situation, temperature variations which produce damaging thermal stresses and strains across the component in complex ways can often limit service life of the component. For this reason, not only is knowledge of elevated temperature isothermal fatigue behaviour essential, but also a study of anisothermal fatigue behaviour is necessary. More recently, thermomechanical fatigue (TMF) tests were developed to estimate the fracture behaviour of components for a wide variety of engineering materials [7–11]. By using TMF technology, thermal fatigue and thermal shock resistance of materials can be studied more systematically and in a more refined nature. However, few references can be found about thermomechanical behaviour of TZM alloy [12,13].

The objective of this work is to investigate the high temperature fatigue behaviour of a molybdenum-based

* Corresponding author.

0022-3115/00/$ - see front matter
PII: S0022-3115(99)00240-8

alloy under isothermal and non-isothermal conditions. In-phase non-isothermal fatigue (TMF) tests at a temperature interval of 350–500°C and isothermal mechanical fatigue tests at two different temperature levels of 350°C and 500°C were performed. During TMF tests, loads and temperature applied were simultaneously varied within each fatigue cycle, and represented coupled changes. The cyclic stress–strain response was examined by different measuring parameters, and the analysis of microstructure of the fracture specimens was carried out to explain the damage mechanism. Especially, a lifetime predictive model was established to estimate both isothermal and thermomechanical fatigue life based on stress and temperature analysis.

2. Experimental

The composition of TZM alloy in the percent of mass is as follows: 99.3 Mo, 0.5 Ti, 0.08 Zr, 0.025 C, 0.025 O, 0.0005 H, 0.0005 N, 0.005 Fe, 0.001 Ni and 0.003 Si. It was provided by Metallwerk Plansee Company. The test alloy was produced with special high-grade molybdenum and the mixed metal powders were pressured to form a round rod, then the rod was wrought and finally treated in a stress-free annealed state. The annealing reduced the residual working stresses without destroying the forging or swaging texture or causing a decrease in hardness.

Cylindrical solid specimens were delivered with a gage diameter of 8.0 mm and a gage length of 20 mm. The specimen gauges were mechanically polished before fatigue tests to avoid reducing fatigue life due to premature crack initiation from surface machine marks.

The testing machine was based on an electro-hydraulic load frame equipped with an infrared heating furnace. Four radiant energy lamps situated in four semi-elliptic elements of the furnace, respectively, provided a 60 mm controlled uniform heated zone on the centre of the specimen. A special contact type of high temperature extensometer was employed for axial strain measurement. Displacement was measured between ridges of rod made of silica on a gage length of 12.5 mm in the middle of the specimen. The long rods permit the extensometer to keep out of the heating furnace. More information about the equipment was described in detail in Ref. [14].

To study the behaviour of the material in isothermal fatigue and thermomechanical fatigue, three kinds of tests were designed. They consisted of isothermal fatigue tests at 350°C and 500°C and in-phase thermomechanical fatigue tests at cyclic temperature between 350°C and 500°C with a triangular waveform. All tests were conducted under load control condition. Three cyclic load amplitudes (20.1, 25.1 and 30.1 kN) for isothermal fatigue tests and four cyclic load amplitudes (20.1, 22.6, 25.1 and 30.1 kN) for thermomechanical fatigue were chosen. All tests were run to failure, which defined as complete fracture. The cyclic load shape consisted of a triangular waveform with a 1-min period and a cyclic load ratio, $R = -1$. In thermomechanical tests cyclic temperature and mechanical load varied simultaneously with a combination of 'in-phase', which refers to the condition where the incurred maximum tensile load is coincident with the maximum temperature and the incurred maximum compressive load is coincident with the minimum temperature.

3. Experimental results

The fatigue life for the three kinds of the tests is shown in Tables 1–3. Between the two kinds of isothermal experiments at 350°C and 500°C, there are no obvious differences in the number of cycles to failure. The isothermal fatigue lives at 500°C are slightly lower than that at 350°C. It appears that in this temperature range, the material keeps a good high temperature cyclic strength. However, comparing with the thermomechanical fatigue data, it clearly indicates that the lifetime obtained under in-phase TMF condition is lower than the one obtained under isothermal condition at the same load amplitude.

During all the tests the ratcheting phenomenon was observed and a schematic showing this phenomenon is given in Fig. 1. The hysteresis loops move generally in the tensile direction in an increasing rate. It is known that the continuous movement of cycles towards the tensile direction indicates cyclic creep behaviour. In the tests, the specimen exhibits an increasing cyclic creep rate with cycling and the cyclic creep behaviour is found to be dependent on load and temperature. Under the same temperature condition, the larger the load amplitude is, the higher the cyclic creep of the specimen. At the same load amplitude, the highest cyclic creep occurs in the case of the 500°C isothermal test. The cyclic creep

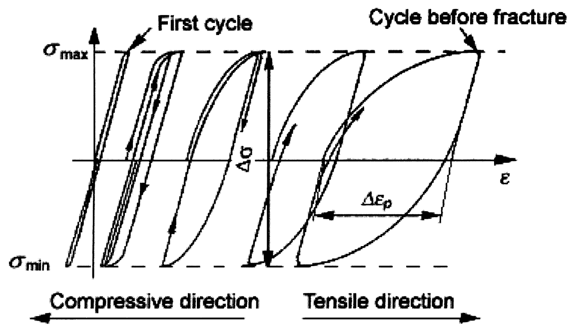

Fig. 1. Schematic showing the ratcheting phenomenon in load control fatigue.

in the 350°C isothermal test is lower than that in the 500°C isothermal test and the cyclic creep in the TMF test is lower than that in the two cases mentioned above.

The movement of cycles towards the tensile direction also indicates the elongation behaviour under cyclic loading. The final elongation before fracture for each test is listed in Tables 1–3. From the experimental data, it is found that the breaking elongation increases when the amplitude of cyclic loading rises. In the case of the isothermal tests at both 350°C and 500°C, it seems that there are similar values of the breaking elongation under the same amplitude of loading. However, in the case of the TMF tests at 350–500°C, except the test of the highest amplitude of loading (30.1 kN), the values of the breaking elongation are much lower than that of the isothermal tests. It appears that the material reveals lower tensile ductility under TMF condition.

The stress–strain response of fatigue can be obtained from the data registered successively during each cycle of the tests. The principal results are summarised in Tables 1–3, where the plastic strain $\Delta\varepsilon_p$ and the plastic strain energy density Δw_p taken from the cycle of half-lifetime and the cycle before fracture, meanwhile, the number of cycles to fail are listed for each isothermal and anisothermal tests. The definition of $\Delta\varepsilon_p$ is defined as in Fig. 1 and Δw_p is equal to the area of the hysteresis loop. It is found that, at the first cycle, there is a very small plastic strain level (< 0.01%) and the stress increases and decreases almost elastically under either isothermal or thermomechanical condition. Then the plastic strain increases continuously and it reaches a maximum level of more than 0.36% in the tests at 20.1 kN load amplitude and more than 1.0% in the tests at 30.1 kN before failure of the specimens. The variation of plastic strain presents a cyclic softening behaviour of the material under load control condition.

The variation of the plastic strain energy density has the same incremental tendency as the plastic strain versus the number of cycles. A very narrow hysteresis loop is observed at the first cycle. The greater the number of cycles is, the wider the hysteresis loop. The largest area of loop occurs before fracture.

Fracture surfaces of specimens for the different load amplitude tests were examined by scanning electron

Table 1
Main results of isothermal fatigue tests at 350°C

Specimen number	Load amplitude (kN)	Stress amplitude (MPa)	$\Delta\varepsilon_p$ at halftime (%)	$\Delta\varepsilon_p$ before fracture (%)	Δw_p at halftime (MJ m^{-3})	Δw_p before fracture (MJ m^{-3})	Breaking elongation (%)	N_f (cycles)
01	20.1	400	0.28	0.40	1.74	2.64	1.78	6600
02	25.1	500	0.35	0.72	2.51	5.92	1.98	380
03	30.1	600	0.55	1.05	3.72	8.66	2.46	36

Table 2
Main results of isothermal fatigue tests at 500°C

Specimen number	Load amplitude (kN)	Stress amplitude (MPa)	$\Delta\varepsilon_p$ at halftime (%)	$\Delta\varepsilon_p$ before fracture (%)	Δw_p at halftime (MJ m^{-3})	Δw_p before fracture (MJ m^{-3})	Breaking elongation (%)	N_f (cycles)
04	20.1	400	0.24	0.42	1.30	2.78	1.63	6020
05	25.1	500	0.33	0.75	2.38	6.14	2.02	410
06	30.1	600	0.60	1.25	4.35	10.85	2.82	26

Table 3
Main results of in-phase thermomechanical fatigue tests

Specimen number	Load amplitude (kN)	Stress amplitude (MPa)	$\Delta\varepsilon_p$ at halftime (%)	$\Delta\varepsilon_p$ before fracture (%)	Δw_p at halftime (MJ m^{-3})	Δw_p before fracture (MJ m^{-3})	Breaking elongation (%)	N_f (cycles)
07	20.1	400	0.26	0.36	1.38	1.86	0.42	5250
08	22.6	450	0.31	0.52	2.04	3.22	0.74	810
09	25.1	500	0.34	0.58	2.42	3.90	1.25	140
10	30.1	600	0.60	1.50	5.12	12.60	2.80	18

microscope. In the isothermal and TMF tests at 20.1 kN load amplitude, the fracture seemed to be characteristic of a small ductile fatigue crack propagation (FCP) zone and a large brittle zone. The FCP zone, where there was a lot of microcracks and inclusions, occupied about 10% of the failed area. In this zone many striations were found everywhere, indicating that the propagation of cracks occurred by fatigue. In the brittle zone no striations and less micro-cracks were observed.

In the isothermal and TMF test at the load amplitude of 25.1 and 30.1 kN, no ductile FCP zone could be found on the fracture surfaces and the rupture seemed to be entirely brittle. Some cleaved inclusions were visible on the fracture surface. The micro-cracks were also observed and many of them were larger than those in the case of the tests at 20.1 kN load amplitude. Fig. 2 shows the SEM images on the fracture surface of the specimen tested in TMF at 30.1 kN load amplitude. The brittle character of the fracture surface appearance can be easily recognised by the cleavage facetes with micro-cracks. Near these cracks, inclusions such as zirconia, titania were observed. More information on the microstructural analysis are described in detail in Ref. [13].

4. Fatigue-life modelling

In recent years, a number of techniques have been developed to predict the lives of materials subject to high temperature fatigue conditions. However, they have been assessed generally under isothermal situations. In the case of thermomechanical fatigue, less lifetime predictive models can be found, especially for TMF in cyclic load control.

In this study, we established a simple method of predicting isothermal and thermo-mechanical fatigue lives based on observations of the cyclic load control effects. It is apparent from TMF test results that the damage produced by the variation of temperature should be considered.

According to Woehler–Miner law, the damage per cycle can be described by a function with maximum stress $\sigma_{\max}$ and mean stress $\bar{\sigma}$ [14,15]

$$\frac{\delta D}{\delta N} = f(\sigma_{\max}, \bar{\sigma}). \tag{1}$$

One of many forms proposed for expressing the curves of the stress amplitude versus the number of cycles to fracture is given by [16]

$$\frac{\delta D}{\delta N} = \frac{\sigma_{\max} - \sigma_1(\bar{\sigma})}{\sigma_{\mathrm{u}} - \sigma_{\max}} \left(\frac{\sigma_{\max} - \bar{\sigma}}{M(\bar{\sigma})} \right)^{\beta}, \tag{2}$$

where σ_{u} is the tensile strength; $\sigma_1(\bar{\sigma})$ and $M(\bar{\sigma})$ are functions of the mean stress and they can be represented by following forms:

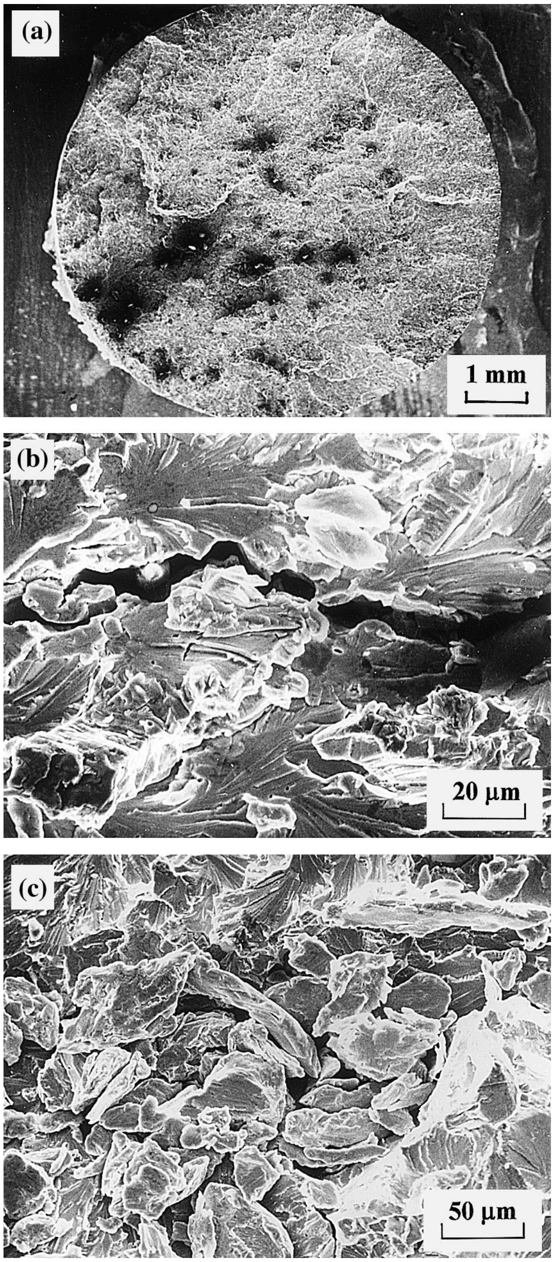

Fig. 2. SEM image on the fracture surface of the specimen tested in TMF at 30.1 kN load amplitude: (a) Total fracture surface; (b) cleavage facets with microcracks; (c) inclusions and cleavage facets on the fracture surface.

$$\sigma_1(\bar{\sigma}) = \bar{\sigma} + \sigma_{10}(1 - b\bar{\sigma}); \qquad M(\bar{\sigma}) = M_0(1 - b\bar{\sigma}), \tag{3}$$

where σ_{10} is the fatigue limit, b is a mean stress coefficient. In the formulas above β and M_0 are the coefficients that depend on the fatigue limit, the cumulate damage effects and of course the temperature.

In the case of our isothermal and thermomechanical fatigue tests, the mean external load is zero, but a complete reversal of external loading does not completely reverse the stress [17]. However, the mean stress is quite a small value in comparison with the stress amplitude. In this situation, the mean stress can be considered as zero and formula (2) is simplified as follows:

$$\frac{\delta D}{\delta N} = \frac{\sigma_{\max} - \sigma_{0l}(T)}{\sigma_u(T) - \sigma_{\max}} \left(\frac{\sigma_{\max}}{M_0(T)} \right)^{\beta(T)}, \tag{4}$$

where M_0 and β are considered as functions of the temperature so as to suit isothermal fatigue conditions. The number of cycles to fracture of isothermal fatigue can be calculated by integrating Eq. (4) for conditions of $N = 0 \rightarrow D = 0$ and $N = N_f \rightarrow D = 1$

$$N_f = \frac{\sigma_u(T) - \sigma_{\max}}{\sigma_{\max} - \sigma_{l0}(T)} \left(\frac{\sigma_{\max}}{M_0(T)} \right)^{-\beta(T)}. \tag{5}$$

The principal properties and damage coefficients of isothermal fatigue of the TZM alloy are given in Table 4.

For thermomechanical fatigue, the damage per cycle can be described by a function with not only maximum stress $\sigma_{\max}$ and mean stress $\bar{\sigma}$, but also maximum temperature $T_{\max}$ and temperature range ΔT

$$\frac{\delta D}{\delta N} = f(\sigma_{\max}, \bar{\sigma}, T_{\max}, \Delta T). \tag{6}$$

To simplify the model, we consider that formula (2) or (4) can be developed to suit the case of TMF conditions. The equation is given as follows if $\bar{\sigma} = 0$:

$$\frac{\delta D}{\delta N} = \frac{\sigma_{\max} - \sigma_{0l}(T_{\max})}{\sigma_u(T_{\max}) - \sigma_{\max}} \left(\frac{\sigma_{\max}}{M_0(T_{\max}, \Delta T)} \right)^{\beta(T_{\max}, \Delta T)}. \tag{7}$$

In this equation M_0 and β are functions of the maximum temperature and the temperature range. From the data of general metals, the value of coefficient M_0 reduces while the temperature increases for isothermal fatigue. Therefore, it is suggested that the value of $M_0(T_{\max}, \Delta T)$ under TMF condition can be replaced by M_0 at an equivalent temperature T_e, ($T_{\min} < T_e < T_{\max}$), that leads to the same thermal effects of fatigue as at the TMF testing

$$M_0(T_{\max}, \Delta T) = M_0(T_e)|_{T_{\min} < T_e < T_{\max}}. \tag{8}$$

Because of no great difference between the value of M_0 at maximum temperature and that at minimum temperature in isothermal fatigue of TZM alloy, we take a mean value of M_0 between $M_0(T_{\max})$ and $M_0(T_{\min})$ to represent $M_0(T_e)$ so as to simplify the calculation

$$M_0(T_{\max}, \Delta T) = \frac{M_0(T_{\max}) + M_0(T_{\max} - \Delta T)}{2}. \tag{9}$$

It is noted that TMF tests of TZM alloy lead to more serious damage than isothermal fatigue tests at the maximum temperature. It is considered that this additional damage produced by the variation of temperature, ΔT, may be simulated by exponential coefficient $\beta(T_{\max}, \Delta T)$, as shown below:

$$\beta(T_{\max}, \Delta T) = \alpha \left(\frac{T_{\max} - \Delta T}{T_{\max}} \right) \beta(T_{\max}). \tag{10}$$

In this formula the maximum temperature is given by Kelvin temperature; the parameter α depends on the phasic relation of thermal cycles and mechanical cycles (in-phase or out-of-phase, for example). In our in-phase cases, α is decided from the experimental data and is equal to 1.16.

Similar to the calculation of the number of cycles to fracture of isothermal fatigue, we integrate Eq. (7) for conditions of $N = 0 \rightarrow D = 0$ and $N = N_f \rightarrow D = 1$

$$(N_f)_{TMF} = \frac{\sigma_u(T_{\max}) - \sigma_{\max}}{\sigma_{\max} - \sigma_{10}(T_{\max})} \left(\frac{\sigma_{\max}}{M_0(T_{\max}, \Delta T)} \right)^{-\beta(T_{\max}, \Delta T)}, \tag{11}$$

where $(N_f)_{TMF}$ is the number of cycles to fracture of thermomechanical fatigue. With formulas (9), (10) and (11), the lifetime of TMF can be evaluated.

A comparison between the experimental results and the calculation by this simple model is presented in Table 5, where $(N_f)_{exp.}$ is the experimental lifetime and $(N_f)_{cal.}$ is the calculated lifetime. It appears that the proposed method to predict TMF lives gives acceptable results. However, it will be noted that this improved method is only verified by TMF tests in which the temperature range, ΔT, is not very large. More information should be obtained to examine the efficiency of the model in more extensive range.

Table 4
Physical properties and damage coefficients of isothermal fatigue

Temperature (°C)	Thermal expansion coefficient (1/K)	Modulus of elasticity, E (MPa)	Tensile strength, σ_u (MPa)	Minimum elongation (%)	Fatigue limit σ_{l0} (MPa)	Damage coefficient, M_0 (MPa)	Damage coefficient, β
20	5.3×10^{-6}	3.2×10^5	785	2.0	250	–	–
350	5.5×10^{-6}	3.1×10^5	680	–	220	1120	8.20
500	5.6×10^{-6}	3.0×10^5	650	–	200	1160	8.05

Table 5
Data of the experimental and calculated lifetime

Fatigue at 350°C, $M_0 = 1120, \beta = 8.20$			Fatigue at 350°C, $M_0 = 1160, \beta = 8.05$			TMF of 350–500°C, $M_0 = 1140, \beta = 7.53$		
σ_{max} (MPa)	$(N_f)_{exp.}$ (cycles)	$(N_f)_{cal.}$ (cycles)	σ_{max} (MPa)	$(N_f)_{exp.}$ (cycles)	$(N_f)_{cal.}$ (cycles)	σ_{max} (MPa)	$(N_f)_{exp.}$ (cycles)	$(N_f)_{cal.}$ (cycles)
400	6600	8030	400	6020	6590	400	5250	3320
–	–	–	–	–	–	450	810	872
500	380	480	500	410	438	500	140	250
600	36	34	600	26	25	600	18	16

5. Conclusions

Isothermal mechanical fatigue and in-phase TMF tests in load control were performed on a molybdenum-based alloy, TZM. By comparing with the yield strength and the tensile strength of the alloy, the experiments can be considered as low-cycle fatigue tests. When the cyclic stress amplitude approaches the material strength limit during the tests of 30.1 kN load amplitude, the fatigue lives of about 10–40 cycles are reasonable.

The isothermal mechanical fatigue lives at 500°C are slightly lower than that at 350°C. However, the fatigue lives obtained in the in-phase TMF tests are obviously lower than the ones obtained in the isothermal tests at the same load amplitude. A reduction of lifetime in TMF tests is produced by the reaction of the variation of temperature and the cyclic mechanical stresses that leads to an additional damage.

Continuous cyclic creep represented by ratcheting occurred during the tests with an increasing creep rate, which is dependent on temperature and load amplitude. The breaking elongation will increase when the amplitude of cyclic loading rises and the material reveals lower tensile ductility under TMF condition. The variation of plastic strain presents a cyclic softening behaviour of the material under load control condition. The greater the number of cycles is, the larger the plastic strain.

The fracture of the alloy is, on the whole, a brittle process under both isothermal and thermomechanical fatigue conditions. A typical cleavage fracture with microcracks is observed and some inclusions can be seen on the fracture surface.

A model of lifetime prediction is developed and it is based on Woehler–Miner law. Damage coefficients that are functions of the maximum temperature and the variation of temperature are introduced in the model to evaluate TMF lives in load control. With this method the lifetime prediction gives results corresponding well to experimental data.

Acknowledgements

We are grateful for the financial support provided partly by the European Atomic Energy Community and partly by the National Natural Science Foundation of China under grant No. 59571037 and No. 59871022.

References

[1] H.A. Calderon, G. Kostorz, Mater. Sci. Eng. A 160 (1993) 189.
[2] W. Jakobeit, J.P. Pfeiffer, G. Wlrich, Nucl. Technol. 66 (1984) 195.
[3] G. Filacchioni, E. Casagrande, U. deangelis, G. Desantis, D. Ferrara, J. Nucl. Mater. 215 (1994) 1288.
[4] B.N. Singh, A. Horsewell, P. Toft, J.H. Evans, J. Nucl. Mater. 215 (1994) 1292.
[5] Y. Hiraoka, H. Kurishita, M. Narui, H. Kayano, Mater. Trans. JIM 36 (1995) 504.
[6] M.F. Maday, J. Nucl. Mater. 233–237 (1995) 1397.
[7] H. Sehitoglu, M. Karasek, J. Eng. Mater. Tech. ASME 108 (1986) 192.
[8] T. Nicolas, M.L. Heil, G.K. Haritos, Int. J. Fract. 41 (1989) 157.
[9] S. Kraft, R. Zauter, H. Mughrabi, Fatigue Fract. Eng. Mater. Struct 16 (1993) 237.
[10] K.A. Hart, S. Mall, J. Eng. Mater. Technol.–Trans. ASME 117 (1995) 109.
[11] H.J. Shi, Z.G. Wang, H.H. Su, Scr. Mater. 35 (1996) 1107.
[12] A. Cardella, M. Akiba, R. Duwe, E. Dipietro, S. Suzuki, K. Satoh, N. Reheis, J. Nucl. Mater. 209 (1994) 117.
[13] H.J. Shi, C. Korn, G. Pluvinage, Mater. Sci. Eng. A 247 (1998) 180.
[14] R.W. Landgraf, Fundamentals of Fatigue Characterizations of Metals, SAE, 1982, pp. 11–18.
[15] D.F. Socie, J. Morrow, in: J.J. Burke, V. Weiss (Eds.), Risk and Failure Analysis for Improved Performance and Reliability, Plenum, New York, 1980, p. 141.
[16] J. Lemaitre, J.L. Chaboche, Mécanique des Matériaux Solides, Bordas, Paris, 1985, pp. 361–367.
[17] S.S. Manson, Thermal Stress and Low-Cycle Fatigue, McGraw-Hill, New York, 1981, pp. 187–191.