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Cyclic Fatigue Behaviour of Eutectoid Aged Mg-PSZ Ceramics with Processing Flaws

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Abstract

The cyclic fatigue behaviour of Mg-PSZ in the as-fired state and after eutectoid aging (1400°C for 2 and 16 h) were investigated in four-point bending tests. A large number of specimens with processing flaws were used to measure the initial strength distribution and the cyclic fatigue lifetime at a stress ratio R = *0·3. From these experimental data a modified 'stress–time–probability' (S–T–P) diagram can be constructed using a statistical approach. The residual strength of the survivor specimens, which is lower than their corresponding initial strength, has also been measured, confirming that pronounced cyclic fatigue damage does occur in these Mg-PSZ ceramics. The experimental results are discussed in terms of transformation toughening, microcracking and precipitate hardening mechanisms.*

Das zyklische Ermüdungsverhalten von Mg-PSZ im gebrannten Zustand und nach eutektoidem Altern (1400°C für 2 und 16 h) wurde mit Hilfe von Vierpunktbiegeversuchen untersucht. Bei einer Vielzahl von Proben mit Prozeßdefekten wurden die Anfangsfestigkeitsverteilung und die zyklische Ermüdungslebensdauer für ein Spannungsverhältnis von R = *0·3 ermittelt. Aus den experimentellen Daten kann ein modifiziertes 'Spannungs–Zeit–Wahrscheinlichkeits' (S–T–P) Diagramm mit Hilfe eines statistischen Ansatzes erstellt werden. Die Restfestigkeit der nicht gebrochenen Proben, die kleiner als die Anfangsfestigkeit ist, wurde ebenso untersucht. Dies bestätigt, daß Mg-PSZ-Keramiken während der zyklischen Ermüdung erhebliche Beschädigung erfahren. Die experimentellen Ergebnisse werden in bezug auf Zähigkeitssteigerung durch Transformation, Mikrorisse und Ausscheidungshärtungsmechanismen diskutiert.*

Les auteurs ont étudié le comportement en fatigue cyclique, utilisant un test de flexion quatre points de Mg-PSZ, dans l'état obtenu après frittage et après un vieillissement eutectoïde (1400°C durant 2 et 16 h). Pour mesurer la distribution des résistances initiales et la durée de vie en fatigue cyclique pour un rapport de contrainte R = *0·3, on a utilisé un grand nombre d'échantillons présentant des défauts d'élaboration. A partir des résultats expérimentaux, un diagramme modifié de prédiction des durées de vie (S–T–P) peut être construit sur base d'une approche statistique. La résistance résiduelle des échantillons survivants a été également mesurée: elle est plus basse que la résistance initiale correspondante, ce qui confirme qu'une détérioration due à la fatigue cyclique se produit dans ces céramiques Mg-PSZ. Les résultats expérimentaux sont discutés en termes de mécanismes de transformation sous contrainte, de microfissuration et de durcissement par précipitation.*

1 Introduction

One of the most effective methods to increase strength and fracture toughness of ceramic materials is the stress-induced tetragonal to monoclinic (t–m) ZrO_2 phase transformation taking place in tetragonal precipitates, particles or grains near the crack tip in ZrO_2-containing ceramics.[1–3] Based on optimized powders and processing techniques, the microstructures such as grain size, flaw size and their distributions and the stress-induced t–m transformation behaviour have been systematically varied and controlled, leading to maximum values of flexural strength and fracture toughness near 2500 MPa and 30 MPa m$^{1/2}$, respectively, in ZrO_2 ceramics.[4–7]

Despite their high strength and fracture toughness, for nearly two decades many investigators have

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shown that ceramics do suffer from mechanical fatigue.[8,9] Efforts have been made to generate design-relevant fatigue data, to understand the micromechanisms of the fatigue effect, and to improve the fatigue behaviour of these ceramic materials.

Cyclic fatigue of transformation-toughened zirconia-based ceramics such as Mg-PSZ,[10-14] Y-TZP(A)[15-18] and Ce-TZP(A)[19,20] has been investigated from processing flaws, indentation cracks and long sharp cracks. Fatigue thresholds as observed on long sharp cracks in these ceramics are found to be as low as 40–50% of the fracture toughness and the cyclic fatigue crack growth rate can exceed those under static fatigue loading at the same stress intensity factor by several orders of magnitude. Fatigue lifetime under cyclic loading can be more than three orders of magnitude shorter than under static loading at the same stress levels.

While these experimental cyclic fatigue data have improved the understanding of the fatigue behaviour in ceramics to give a lifetime prediction and hence a reliable application of ceramics in engineering, there is still a lack of understanding of the micromechanisms of the cyclic fatigue in ceramics. Much more work is needed to understand the interrelations between the microstructure, the R-curve behaviour and cyclic fatigue effect of ceramic materials.

The main objectives of this paper are to investigate the cyclic fatigue of three Mg-PSZ ceramics with processing flaws and to construct modified S–T–P (stress–time to failure–probability of failure) diagrams based on a statistical approach for design applications.

2 Experimental Procedures

2.1 Materials and characterization

Commercial Mg-PSZ powders (ICI Advanced Ceramics Ltd, Melbourne, Australia) were used for this investigation. The MgO content was 9 mol.%. The powders were first compacted with a uniaxial die at 6 MPa followed by isostatic pressing at 200 MPa. The green compacts were sintered at 1700°C for 10 h. Specimens were then aged at 1400°C for 2 h and 16 h, respectively. The sintered and eutectoid aged bending bars were then ground with diamond tools to $3 \times 4 \times 60\,mm^3$ in size. The tensile surface was polished with 1 μm diamond paste. The phase composition of the materials was also characterized both by neutron and X-ray diffraction techniques. A four-point load fixture with 22 and 44 mm as inner and outer span widths, respectively, was used to determine the initial bending strength in air with a crosshead speed of $0{\cdot}1\,mm\,s^{-1}$. Fifteen specimens were tested for the Weibull evaluation of the initial strength.

2.2 Cyclic fatigue experiments

Four-point bending (22 mm inner and 44 mm outer spans) cyclic fatigue tests were conducted with an Instron machine model 8501 under load control with a stress ratio $R = 0{\cdot}3$. The frequency was kept constant at 30 Hz. Twelve to fifteen specimens were tested for each individual set of stressing conditions. While for the as-fired and the 16 h aged ceramics fatigue tests were conducted at two stress levels with corresponding failure probabilities of 3 and 10%, these tests for the 2 h aged ceramic were performed at three stress levels. The residual strengths of the survivor specimens were also measured and compared with their corresponding initial strengths.

3 Results and Discussion

3.1 Material characterization

Table 1 shows the phase content of the three Mg-PSZ ceramics from the neutron and X-ray diffraction in different states. According to the neutron diffraction, the as-fired material contained 25% tetragonal and 75% cubic ZrO_2 phase. No monoclinic phase was detected for the as-fired material by X-ray diffraction at polished, ground and fracture surfaces. The 2 h aged material contained 48% tetragonal and 52% cubic phase in the as-aged state. A stress-induced t–m transformation of 13%, 23% and 38% was measured by X-ray diffraction at polished, ground and fracture surfaces respectively. The 16 h aged material contained 42% monoclinic, 15% tetragonal and 44% cubic phase in the as-aged state. According to the X-ray diffraction the monoclinic phase content at the polished, ground and fracture surfaces was even lower than that from the neutron diffraction. This may be caused by the inaccuracy of the X-ray diffraction. However, the results of the phase analysis at the polished, ground and fracture surfaces indicate that no further stress-induced t–m transformation occurred for the 16 h aged Mg-PSZ ceramic.

Figure 1 plots the Weibull distribution of the initial bend strength σ_f for the three materials with processing flaws according to:

$$P = 1 - \exp\left[-\frac{\sigma_f}{\sigma_o}\right]^m \quad (1)$$

where m is Weibull modulus, σ_o is scale factor and P is probability of failure at σ_f. Thus, the following values may be obtained: $\sigma_o = 245$ MPa, $m = 11$ for the as-sintered Mg-PSZ-I; $\sigma_o = 585$ MPa, $m = 25$ for 2 h aged Mg-PSZ-II and $\sigma_o = 396$ MPa, $m = 19$ for the 16 h aged Mg-PSZ-III. The higher amount of

Table 1. Phase analysis of Mg-PSZ ceramics from neutron and X-ray diffraction

	Neutron diffraction								
	As fired			*2 h aging*			*16 h aging*		
	m (%)	*t* (%)	*c* (%)	*m* (%)	*t* (%)	*c* (%)	*m* (%)	*t* (%)	*c* (%)
Bulk composition	0	25	75	0	48	52	42	14	44

	X-ray diffraction					
	As-fired		*2 h aging*		*16 h aging*	
	m (%)	$(t+c)$ (%)	*m* (%)	$(t+c)$ (%)	*m* (%)	$(t+c)$ (%)
Polished surface	0	100	13	87	36	64
Ground surface	0	100	23	77	34	66
Fracture surface	0	100	38	62	39	61

the stress-induced t–m transformation led not only to an increase in the initial flexural strength but also to a higher Weibull modulus of the material.

3.2 Cyclic fatigue behaviour

Table 2 shows the stress levels and the number of the initial fractures (i), the fatigue fractures (f) and the survivors (s) under cyclic loading. For the as-fired and 16 h aged ceramics fatigue tests were conducted at two stress levels, which, according to Fig. 1, correspond to failure probabilities of 3 and 10% respectively. For the 2 h aged ceramics the fatigue tests were performed at three stress levels. The maximum number of cycles was fixed at $N_L = 2 \times 10^6$. Figure 2 shows the stress versus time to failure (s/t) curve for the three Mg-PSZ ceramics. Despite the large scatter in the fatigue lifetime it can be clearly seen that cyclic fatigue occurred at much higher applied stress levels for the 2 h aged Mg-PSZ-II ceramic with higher initial strength. The as-fired Mg-PSZ-I ceramic showed the lowest fatigue resistance caused by the very low initial strength. The Weibull distributions of the failure probabilities as a function of the lifetime are represented in Fig. 3. The specimens fractured during the fatigue tests are displayed here with their corresponding failure probabilities as determined on the basis of the sample size of 12–15 specimens.

From these cyclic fatigue data and the Weibull distribution of the initial strength a modified stress–lifetime (s/t) curve can be evaluated, as shown in Fig. 4, where both the Weibull distribution of the flexural strength σ_i and the applied stress level in the fatigue tests σ_a are represented for the as-fired Mg-PSZ-I ceramic. By using a large number of specimens it can be assumed that the two sample series A and B for the strength measurements and for cyclic fatigue tests ($R = 0{\cdot}3$), respectively, are equivalent with respect to the distribution of the initial flaws in the specimens, so that the initial strength can be described by the *same* Weibull distribution and the *same* Weibull parameters for the two series. Although the applied stress level σ_a in the fatigue test is kept constant, the loading situation for each individual specimen is different and can be described by the ratio σ_a/σ_i. It is this variable σ_a/σ_i ratio that

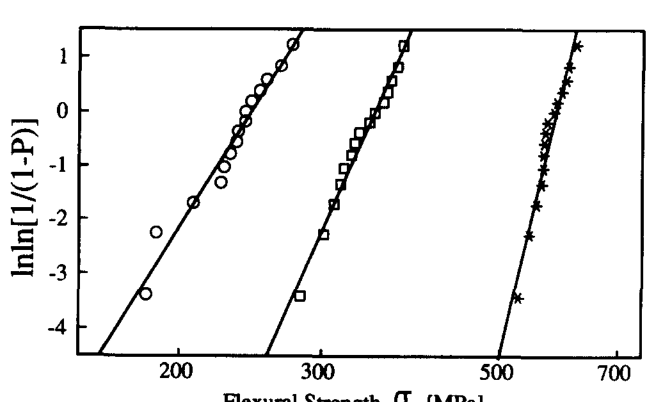

Fig. 1. Weibull distributions of the initial strength of Mg-PSZ ceramics with processing flaws. –⊖–, As-fired ($m = 11{\cdot}3$, $\sigma_o = 245$ MPa); –✕–, 2 h aging ($m = 25{\cdot}6$, $\sigma_o = 585$ MPa); –⊟–, 16 h aging ($m = 15$, $\sigma_o = 348$ MPa). $\ln\ln 1/1 - P = m \ln \sigma_f/\sigma_o$.

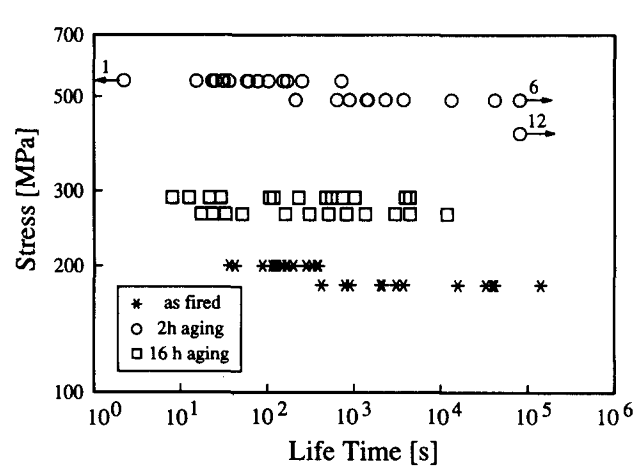

Fig. 2. Stress versus time to failure (s/t) curve of Mg-PSZ ceramics with processing flaws. Frequency: 30 Hz; $R = 0{\cdot}3$.

Table 2. Compilation of experimental fatigue data

Materials	*Heat treatment*	*Stress* σ_{max} (*MPa*)	*Failure probability* *P* (%)	*Number of specimens*[a] (*i*)	(*f*)	(*s*)
Mg-PSZ-I	As fired	180	3	0	13	0
		200	10	1	14	0
Mg-PSZ-II	2 h aging	400	0·01	0	0	12
		480	0·6	0	9	6
		530	8	1	14	0
Mg-PSZ-III	16 h aging	275	3	0	12	0
		300	10	1	14	0

[a] (i) = Failed during the first loading cycle; (f) = failed during the fatigue test; (s) = not failed after the limit number of cycles ($N_L = 2 \times 10^6$). The fatigue stress corresponds to the failure probability calculated from the Weibull distribution of the initial strength.

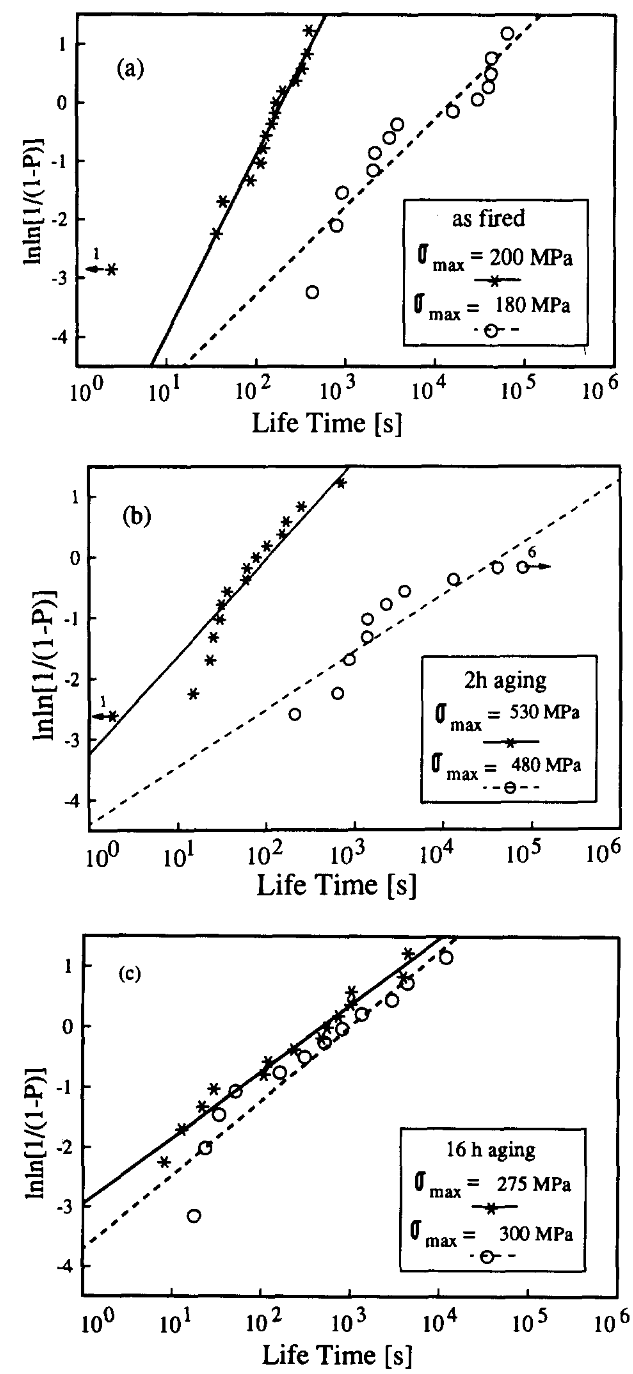

Fig. 3. Weibull distribution of the lifetime under cyclic loading at different stress levels for (a) as-fired, (b) 2 h aged and (c) 16 h aged Mg-PSZ ceramics.

leads to a Weibull distribution of the time to failure for a constant applied stress level in the fatigue test (see Fig. 3). The modified stress–lifetime (*s/t*) curves of the three materials are shown in Fig. 5, where only the specimens which failed during the fatigue tests are considered (see also Table 2); their fatigue stress σ_a ($\sigma_a = \sigma_{max}$) is correlated with the initial strength σ_i of the individual specimens (Fig. 1) with the same failure probability. While for the as-fired Mg-PSZ-I ceramic the σ_a/σ_i–t_f curves derived from the fatigue tests at two stress levels showed an experimental error of one order of magnitude in the fatigue lifetime, they agreed much better with each other for the 2 h and 16 h aged Mg-PSZ ceramics. From Fig. 5 it also becomes evident that the stress induced t–m transformation toughening in Mg-PSZ ceramics increases not only the initial strength of the materials but also the fatigue resistance of these ceramics.

This modified σ_a–t_f curve provides a new effective method to evaluate the dependence of the lifetime on the fatigue stress levels (so-called *s/N* or *s/t* curve) from the Weibull distribution of the initial strength and the fatigue data measured at one stress level. In the 'conventional' *s/N* or *s/t* curve the changing fatigue stress levels lead to a variation of fatigue

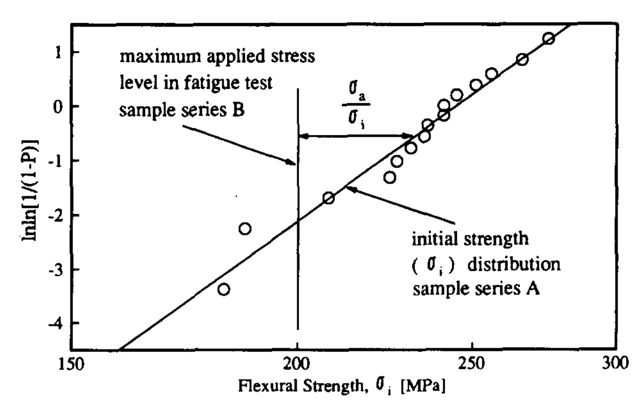

Fig. 4. Representation of the loading situations for each individual specimen of the fatigue test series B at fixed maximum stress level. Their initial strength can be derived from the Weibull distribution of the flexural strength of the reference sample (series A).

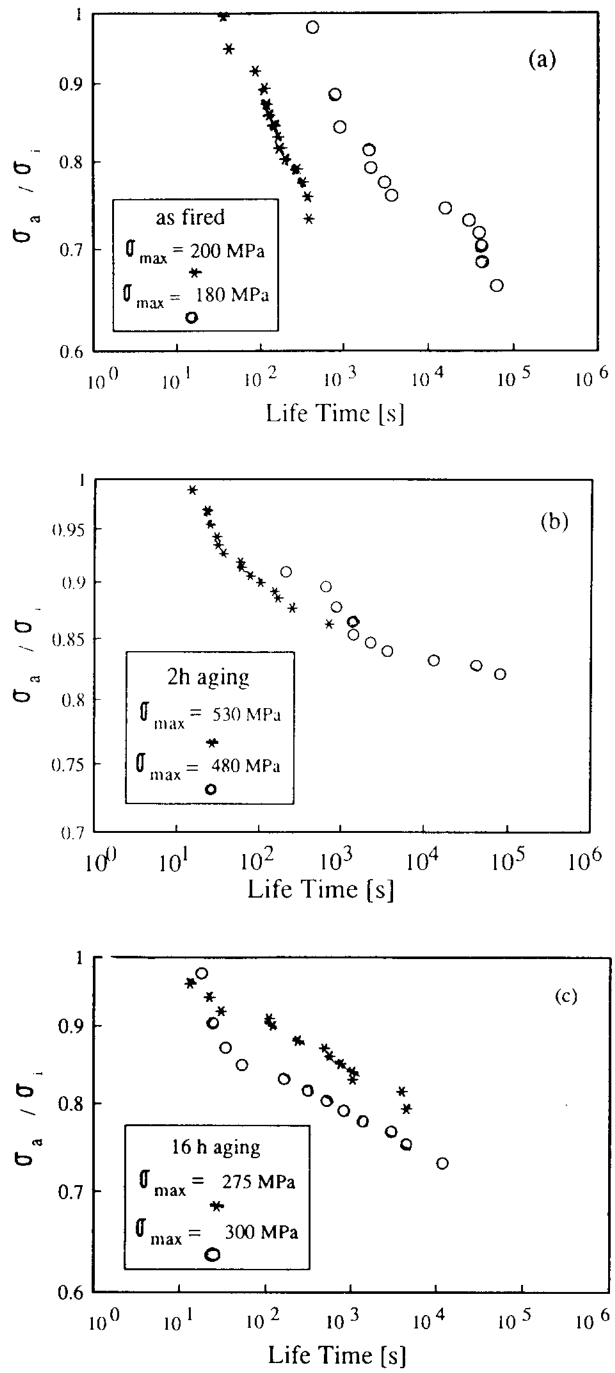

Fig. 5. Modified stress–lifetime (σ_a/σ_i–t_f) curves under cyclic loading for (a) as-fired, (b) 2 h aged and (c) 16 h aged Mg-PSZ ceramics. For fatigue conditions see Table 2.

lifetime for a constant flaw size expressed as a particular failure probability. This means nothing else than the dependence of the lifetime on the fatigue stress levels for a particular flaw size which can be represented at constant failure probabilities (for example 10%, 50% or 90% in the distribution of the lifetime in the fatigue tests). For ceramic materials a large number of specimens at each fatigue stress level must be used in order to get such an s/N or s/t curve with acceptable statistical accuracy.

In the modified (σ_a–t_f) curve as shown in Fig. 5, however, only two series of specimens are needed with one series for the initial strength distribution (see Fig. 1) and the other series for the fatigue tests at one stress level. Here the stress in the fatigue test is chosen at one level but the flaw sizes of the specimens are naturally changing, which can be evaluated from the Weibull distribution of the initial strength. These two approaches for getting s/t or s/N curves, i.e. the change of the stress levels for constant flaw sizes, i.e. failure probabilities, or the change of the flaw sizes at constant stress levels, can be regarded as equivalent. For a particular σ_i value with a corresponding failure probability, which can be calculated from the Weibull distribution of the initial strength, the modified σ_a–t_f curve can then be transferred to an S–T–P curve by multiplying this σ_i value with σ_a/σ_i. The S–T–P curve obtained in this way represents then the dependence of the lifetime on the stress levels at a failure probability which is equal to the failure probability of σ_i. Figure 6 shows such an S–T–P curve for the three ceramics at 50% failure probability. It is evident that fatigue occurred at much lower applied stress levels for the as-fired Mg-PSZ ceramic, which is caused by the lower initial flexural strength. From Fig. 6 it can be concluded that a higher initial flexural strength is also connected with an increased fatigue resistance of the Mg-PSZ ceramics with processing flaws. It has to be noted here, however, that the present method to obtain the modified S–T–P curve has two limitations: (1) the flaw populations which lead to failure of the ceramics are the same under monotonic loading and under cyclic loading; (2) there can be no significant R-curve behaviour of the ceramics.

The residual strengths of the survivor specimens (2 h aged ceramic) were also measured after the fatigue tests. The results are represented together with the Weibull distribution of the initial strength in Fig. 7. Necessarily, the residual strength values appear as truncated strength distributions. They are compared with the corresponding initial strength values with the highest failure probabilities, since it

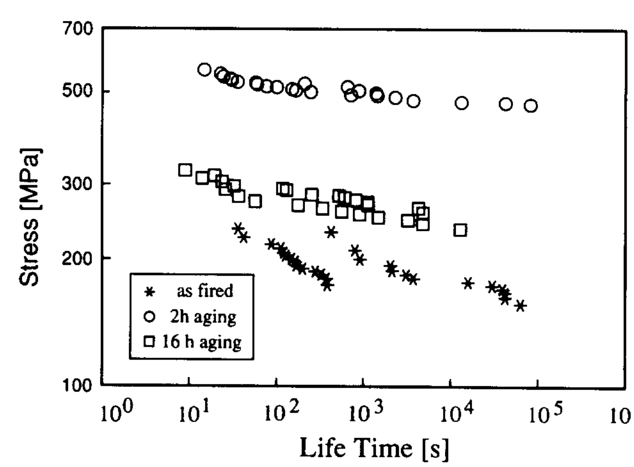

Fig. 6. Fatigue lifetimes of the three Mg-PSZ ceramics as represented in the S–T–P curves at $P = 50\%$ under cyclic loading.

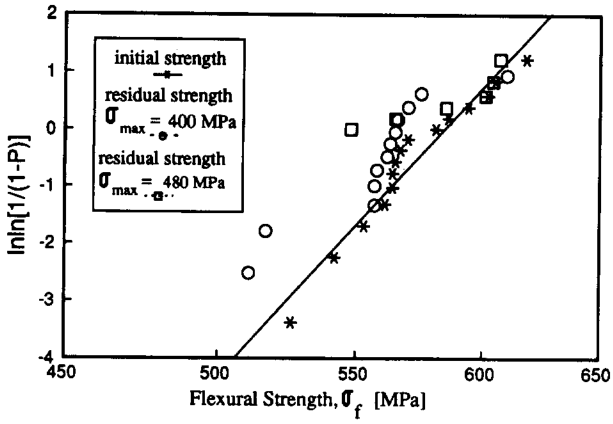

Fig. 7. Weibull distributions of initial strength (sample A) and the residual strength of survivor specimens (samples B and C, $R = 0{\cdot}3$, $f = 30$ Hz, $N_L = 2 \times 10^6$) after cyclic loadings of the 2 h aged Mg-PSZ ceramic.

can be conjectured that these survivor specimens would have had a higher failure probability in their initial state than the specimens which failed during the fatigue tests. As shown in Fig. 7, the residual strength of the survivor specimens after long-term fatigue tests is clearly lower than the initial strength. This indicates that damage has occurred to the specimens during the long-term cyclic fatigue test.

4 Conclusion

Three 9 mol.% Mg-PSZ ceramics (as-fired, 2 h and 16 h aged) were studied with respect to the phase compositions, the stress-induced t–m transformation, the Weibull distribution and cyclic fatigue behaviour. Pronounced stress-induced t–m transformation by the 2 h aged Mg-PSZ leads not only to an increase in flexural strength and a higher Weibull modulus, but also to higher fatigue resistance under cyclic loading. Cyclic fatigue damage exists in the Mg-PSZ ceramics which can be seen also in the decrease of the residual strength of the survivor specimens after cyclic loading compared with their corresponding initial strength.

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