DAMAGE DEVELOPMENT DURING THERMAL CYCLING OF METAL AND GLASS MATRIX COMPOSITES

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SUMMARY: Results of thermal fatigue studies in some fibre reinforced metal and glass matrix composites, MMCs and GMCs, respectively, are presented. The thermal cycling technique involved a computer controlled system of inserting the sample in the furnace, holding it there at elevated temperature (325 °C for the MMCs and 700 °C for CMCs) for 15 minutes, taking the sample out and cooling it in forced air. Microstructural changes resulting from thermal cycling and thermal fatigue damage were evaluated by employing a range of characterisation techniques, including measuring the change in elastic modulus and internal friction of the composites. In MMCs, damage was detected in the form of plastic deformation of the matrix and cracking, debonding at the fiber/matrix interface, followed by void formation at the interface. In GMCs, material degradation was attributed to phenomena related to the softening and cavitation of the glass matrix and to oxidation of the fibres.

KEYWORDS: metal matrix composites, glass matrix composites, thermal cycling, thermal stresses, microstructural damage.

INTRODUCTION

Metal, glass and ceramic matrix composites are being developed for applications involving elevated temperatures, where they will often experience transient thermal stresses due any temperature excursions (inadvertent or by design) during service [1]. Turbine blades, for example, are very much susceptible to thermal fatigue. Consequently, the behavior of metal, glass and ceramic matrix composites subject to abrupt thermal gradients, i.e. under thermal shock or thermal cycling conditions, is being given especial attention by researchers [2-8]. Metal matrix composites (MMCs) exploit the comparatively high stiffness of a ceramic component to reinforce a ductile metal matrix. In MMCs, there is generally a large mismatch between the coefficient of thermal expansion of the ceramic reinforcement and the metallic matrix. Thus, any temperature change will lead to thermal stresses, which can affect microstructural stability and alter mechanical properties. In glass matrix composites (GMCs), on the contrary, it is usually possible to match the thermal expansion of the matrix to that of the reinforcement. For example, when using SiC-Nicalon™ fibres as reinforcement, a borosilicate glass may be chosen as matrix to minimize the thermal expansion mismatch [3]. In

general, the magnitude of thermal stresses in composites is proportional to the thermal strain, $\Delta\alpha\,\Delta T$, where $\Delta\alpha$ is the difference in the expansion coefficients of the two components and ΔT is the amplitude of the thermal cycle. Thus, while in MMCs thermal cycling induced damage will be influenced by the development of internal stresses due to thermal expansion mismatch, in glass matrix composites this effect will not be relevant and other source of microstructural damage, e.g. fibre oxidation, may be active. In this paper we present results of thermal fatigue studies in both MMCs and GMCs. Microstructural changes resulting from thermal cycling were evaluated employing a range of characterisation techniques. The thermal fatigue damage in both types of composites was evaluated and compared.

EXPERIMENTAL PROCEDURE

Metal Matrix Composites (MMCs)

The investigated MMC was a magnesium-Al alloy matrix composite with continuous alumina fibers, unidirectionally aligned. The volume fraction of fibres was 35 vol. %. Details of this material have been given elsewhere [2]. Thermal cycling was done in a home-built apparatus shown in Fig. 1. It consists of a digitally controlled pneumatic piston and a tube furnace. The piston puts the sample into the furnace, holds it there for a preset time, and then withdraws it. After withdrawal the sample is cooled by an electric fan. Thermal cycling was done for a fixed number of cycles and the elastic modulus was measured. The number of thermal cycles performed was recorded by a cycle counter.

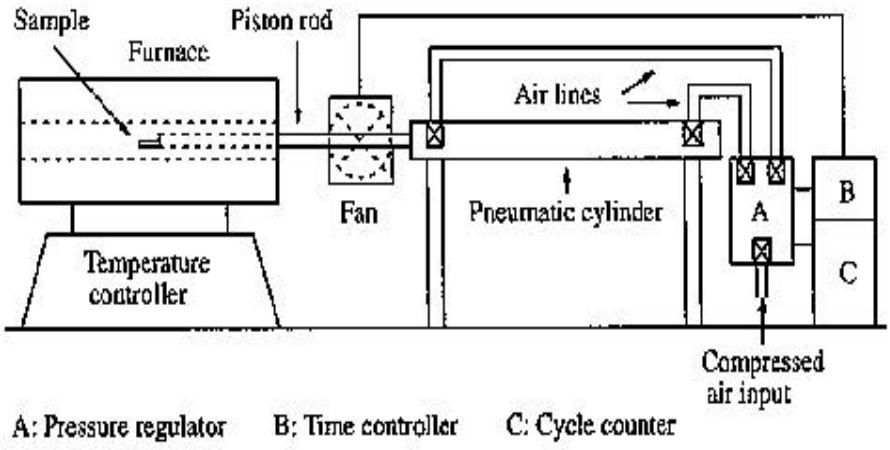

Figure 1: Schematic drawing of the thermal cycling rig used to test MMCs.

Glass matrix composites (GMCs)

The GMCs investigated was a commercially available SiC-Nicalon^{TM} fiber reinforced borosilicate (DURAN^{TM}) glass matrix composite (Schott Glaswerke, Mainz, Germany) [9]. Details about the composite fabrication are given in the literature [10]. The samples were received in the form of rectangular test bars of nominal dimensions (4.5 mm x 3.8 mm x 100 mm). The density of the composites was 2.4 g/cm^3, and the fiber volume fraction ~ 0.4. The composite exhibits a fairly homogeneous distribution of the fibers and absence of porosity in the glass matrix. For the thermal cycling tests, the samples were alternated quickly between high temperature (T = 700°C) and room temperature for different numbers of cycles (up to 1000 cycles). A computer program controlled the movement of the sample holder. The time at high temperature was set at 15 minutes, with 5 minutes being allowed for the sample to reach the target temperature of 700 °C, while the time at room temperature was fixed at 5 minutes.

At least two samples were considered for each number of cycles investigated. According to previous results [8], 700°C was considered to be the upper service temperature for these materials and therefore the thermal cycling behavior between this temperature and room temperature was of particular interest in this study. Damage development in the GMCs was evaluated by monitoring the variation in the Young's modulus and internal friction of the material as a function of the number of thermal cycles it was subjected to. The Young's modulus and internal friction measurements were performed on both as-received and thermally-cycled samples using a forced-vibration resonance technique, similar to that described by Förster [11]. This technique used a suspended bar configuration and has been used in a previous study to characterize the microstructural degradation in similar materials subjected to thermal shock treatments [8]. Scanning electron microscopy (SEM) examination of polished sections of the thermally-treated samples was used to characterize the microstructures.

RESULTS AND DISCUSSION

MMCs

In MMCs thermal fatigue induced damage can take the form of plastic deformation of the matrix, damage at fiber/matrix interface, such as microvoid formation or cracking at the interface, interfacial sliding (in fiber reinforced composites), or in some cases even fiber fracture. As mentioned above, thermal stresses will be generated because of a temperature change due to the mismatch in expansion coefficient between the components. The special aspect of a metal matrix composite is that the metal matrix in general is soft enough to undergo plastic deformation under the action of the thermal stresses generated in the matrix. Such plastic deformation of the matrix will tend to work harden the matrix.

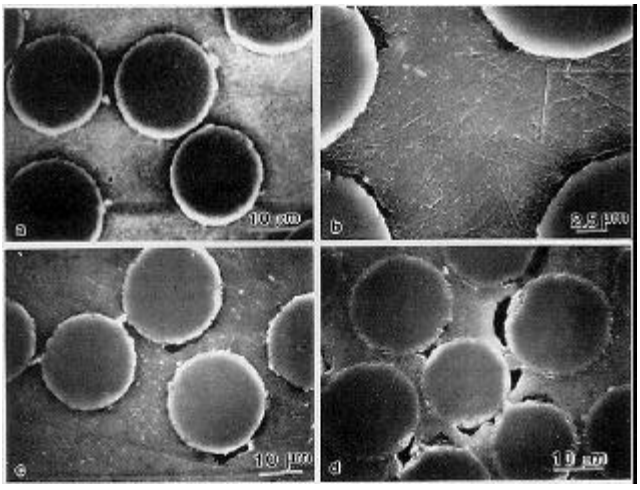

Figure 2: Development of microstructural damage in Mg-Al alloy matrix composite with increasing number of thermal cycles

Continued thermal cycling will lead to further damage in the composite involving fiber/matrix debonding, cavitation at the interface and growth of cavities. The development of such microstructural damage with increasing number of thermal shock cycles is shown in Figure 2. Such damage will result in a reduction of strength of the composite, but, more importantly, it leads to a loss of stiffness.

In order to evaluate the damage caused by thermal cycling, in terms of the elastic modulus, we can be express the damage in elastic modulus, D_E, as

$$D_E = 1 - (E_n / E_o) \quad (1)$$

where E_n is the elastic modulus after n cycles and E_o is the initial elastic modulus before cycling. Thus, when the number of cycles is zero, i.e., $E_n = E_o$, $D_E = 0$, i.e., there is no damage. At the failure point of the material, the modulus goes to zero, i.e., $E_n = 0$, $D_E = 1$, i.e., maximum damage has occurred. Here, we have implicitly assumed that damage responsible for the stiffness loss consists of cavitation and cracking. It turns out that it is indeed so. This allows to define another damage parameter in terms of density of the composite, D, as follows

$$D_\rho = 1 - (\rho_n / \rho_o) \quad (2)$$

where ρ_n is the density after n cycles and ρ_o is the initial density before cycling.
In the as-received specimen, the fiber and matrix are in intimate contact with no apparent voids. The microstructures after thermal cycling showed microvoids at the fiber/matrix interface after 1000 cycles (Figure 2). These microvoids grew with increasing thermal cycles, indicating that the cavitation was not only a surface phenomenon, but characteristic of internal damage. Such microvoid formation was almost absent in the same composite material which had been isothermally treated at 300 ^oC up to 410 hours. Thus, cavitation at the interface resulted from the cyclic thermal stresses. Figure 3 shows the damage in elastic modulus, D_E, and in density, D_ρ, vs. number of cycles. A significant damage in elastic modulus and density with increasing thermal cycles can be seen. No weight loss was observed in the composites after thermal cycling, i.e., the mass was conserved. However, a measurable increase in the sample dimension after thermal cycling was observed. This corresponding dimension change was due to the microvoid formation in the sample. This indicates that the decrease in composite density was associated with microvoid formation rather than loss of matrix material due vaporization. Damage in terms of cavitation leads to a loss in modulus and density. Since the same physical phenomenon is responsible for both D_E and D_ρ, it is instructive to investigate a correlation between the two. The effect of porosity on the elastic modulus can be described by Mackenzie's equation [12]:

$$E_n = E_o (1 - bV_v - b_1V_v^2) \quad (3)$$

where E_n is the Young's modulus, V_v is the volume fraction of voids in the composite after N cycles, E_o is the elastic modulus of the composite after zero cycles and, b and b_1 are material constants of a fully dense material. For most materials, b = 2 and b_1 = 0.5 [12]. Substituting Eq. (1) into Eq. (3), one can get

$$D_E = bV_v + b_1V_v^2 \quad (4)$$

The density of composite, ρ_o and ρ_n, before and after thermal cycling, respectively,can be written in terms a rule of mixtures relationship as

$$\rho_o = \rho_m V_m + \rho_f V_f \quad (5)$$
$$\rho_n = \rho_m V'_m + \rho_f V'_f \quad (6)$$

where V_m and V'_m are the volume faction of matrix before and after thermal cycling, V_f and V'_f are the volume faction of fiber before and after thermal cycling, and ρ_m and ρ_f are density of matrix and fiber, respectively. Since the volume fraction of voids is very small and voids only

exist on the matrix side of the fiber/matrix interface, $V_f = V'_f$, $V_m = V'_m + V_v$, i.e, $V_v = V_m - V'_m$. Therefore, subtracting Eq. (6) from (5), one can get

$$V_v = (\rho_o - \rho_n)/\rho_m \qquad (7)$$

Accordingly, Eqs. (2), (4) and (7) can be combined as

$$D_E = b(\rho_o/\rho_m)D_\rho + b_1[(\rho_o/\rho_m)D_\rho]^2 \qquad (8)$$

Plotting the experimental D_E data and the calculated D_E from Eq. (8), see Fig.4, a reasonable match between the experimental and calculated values is found. The plot of D_E vs. D_ρ via Eq. (8) and D_E vs. D_ρ from experimental data in Fig. 4 indicates that density decrease (i.e., cavitation) was responsible for the decrease in the elastic modulus. Thus, Eq. (8) can be used to predict the damage in elastic modulus from the measurement of the damage in the density of the composite. This can be done, however, only when chemical influence is not a factor in the change of elastic modulus of the composite during thermal cycling.

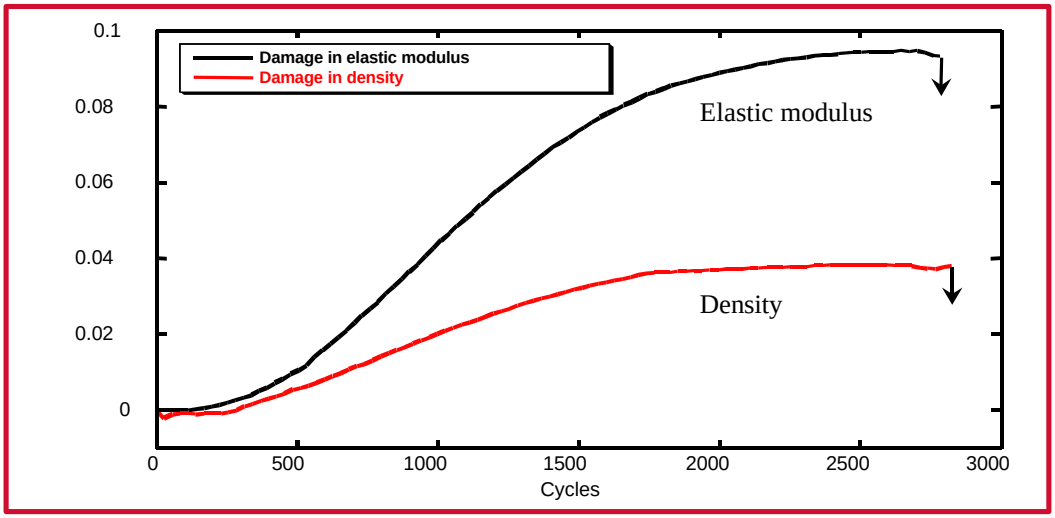

Figure 3: Damage in elastic modulus, D_E, and in density, D_δ, vs. number of thermal cycles in MMC.

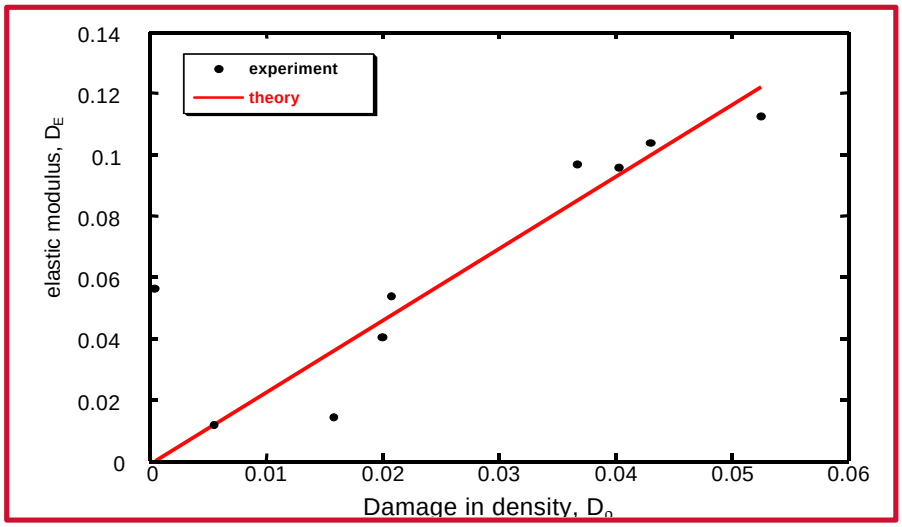

Figure 4: Plot of the experimental D_E data and the calculated D_E from Eq. (8) for thermally cycled MMCs showing reasonable match between the experimental and calculated values.

GMCs

Figure 5 shows the typical variation in the non-destructively-determined Young's modulus (E/E_0) and internal friction (Q^{-1}/ Q_0^{-1}) of the samples cycled thermally between room temperature and 700 °C as a function of the number of thermal cycles. The Young's modulus is given as relative values, i.e. normalized to the values measured in the as-received material. (E_0 = 120.1 ± 0.1 GPa). The magnitude of the change in internal friction is much greater than that of the Young's modulus. This is in agreement with the findings of previous studies on thermally-shocked samples [8] and also with trends found in the literature [13]. This marked difference in behavior suggests that internal friction is the more sensitive parameter by which to assess microstructural damage. An increase in the internal friction, coupled with a simultaneous decrease in the Young's modulus, has been shown by several authors to be indicative of the development of microstructural damage in materials subjected to thermal shock [8, 13-15]. In particular, the generation of porosity and/or microcracking has been correlated with the significant increase in internal friction observed in damaged materials. Thus, the results in Figure 5 are indicative of microstructural degradation in the present samples as a consequence of the thermal cycling. This microstructural damage is most likely due to the creation of new internal surfaces within the composite, such as cavities, interfacial debonding and microcracking.

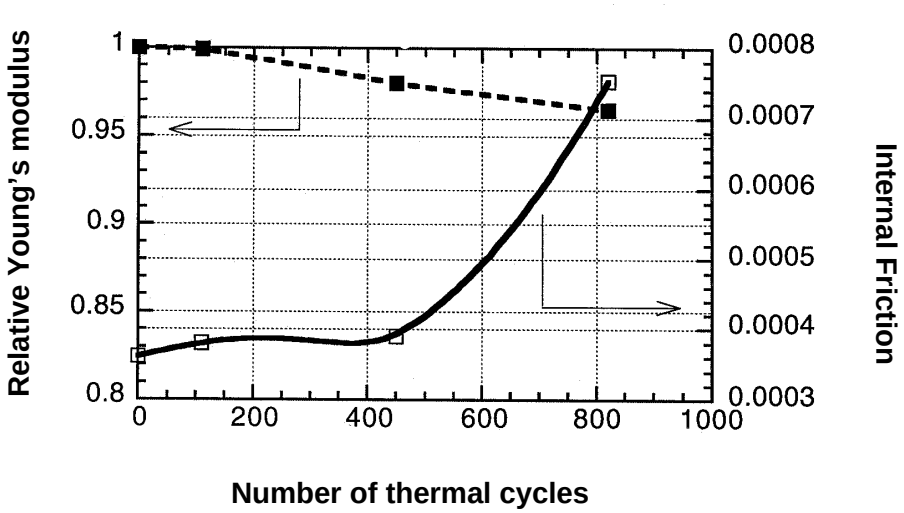

Figure 5: The effect of thermal cycling on the Young's modulus (■) and the internal friction (❒) of SiC-fiber reinforced glass matrix composites

These forms of microstructural damage have been observed in other fiber-reinforced glass and glass-ceramic matrix composites aged at intermediate temperatures (in the range 400 - 800 °C) for different times (from a few hours to over 500 hours) [4,16,17].

In the absence of matrix microcracking, the only possibility for oxygen ingress and interface degradation is through the sample surfaces, where the fiber ends are directly exposed to hot air. This form of microstructural damage, characterized by the formation of annular porosity around the fibers, has been observed in aging experiments of similar composites and has been referred to as "pipeline" oxidation [16,17]. Exposure of the material at a temperature well above the glass transition temperature of the glass matrix, T_g = 525 °C [8], can also result in viscous flow of the glass with consequent cavity or porosity formation, which is exacerbated by the presence of the rigid, constraining fibers [17]. This form of damage can be deduced from the SEM micrographs in Figure 6 showing the polished sections of a sample that had been

subjected to 700 thermal cycles. It is evident that viscous flow of the matrix at 700 °C has resulted in the formation of cavities, as well as local interfacial separation and interfacial cracking. It may also have caused some displacement and rearrangement of the fibers within the composite. These thermal effects, which are not connected with oxidation mechanisms, have also been found in similar composites aged thermally in non-oxidizing argon atmospheres [18]. These mechanisms, in particular the formation of porosity, are likely to lead to a loss of composite stiffness and may also explain the observed increase in internal friction as the number of thermal cycles increased [16,17]. In summary, it can be stated, that for these composite materials with matched thermal expansion coefficients, thermal-cycling in air has no significant effect itself since relatively low thermal stresses are developed, rather, it is the high-temperature exposure to the oxidizing environment that is responsible for the observed deterioration in the material's behavior. Similar results were found in borosilicate glass matrix composites reinforced by carbon fibres [19].

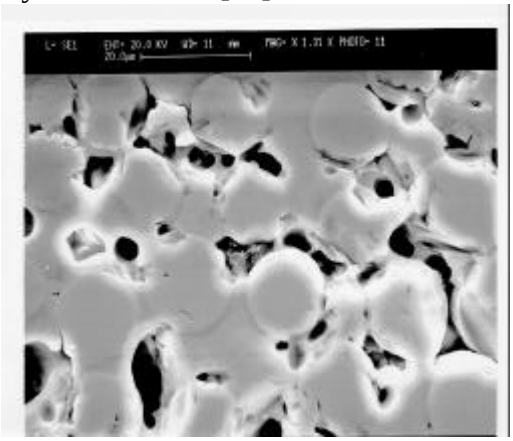

Figure 6: SEM micrograph of a GMC sample that was thermally cycled 700 times from 700 °C to room temperature, indicating matrix cavity generation adjacent to the fibers.

CONCLUSIONS

Thermal stresses in an MMC can be relieved by one or more of the following means: plastic deformation of the matrix and cracking, debonding at the fiber/matrix interface, followed by void formation at the interface. Plastic deformation of the ductile matrix in an MMC is the first manifestation of damage under thermal fatigue. On continued cycling, damage takes the form of microvoids at the interface and/or cracking in the matrix. Under these conditions, the elastic modulus and density of the composite decrease with thermal cycling. The suggested damage parameters in terms changes in the elastic modulus and density can be used to describe the evolution of damage. In GMCs, thermal cycling in air from 700 °C to room temperature resulted in the generation of microstructural damage, which was detected by the simultaneous decrease in the Young's modulus and increase in the internal friction of the composite as the number of thermal cycles increased. Microstructural damage was mainly attributed to partial fiber oxidation and softening of the glass matrix during the exposure to a high temperature oxidising environment. Matrix softening caused cavity formation within the matrix and at the fiber/matrix interfaces.

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