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Thermal Fatigue Resistance of Open Cell Ceramic Foams

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Abstract

A variety of open cell ceramic foams were subjected to rapid thermal cycles by infrared heating and forced air cooling to study the thermal fatigue behaviour of these materials. After cycling, the extent of damage in the samples was determined by measuring the elastic modulus using dynamic resonance (non-destructive test) and the retained strength in three-point bending (destructive test). It was found that the retained elastic modulus and strength gradually decreased with an increase in the number of cycles, followed by a saturation behaviour indicating a damage accumulation mechanism. The extent of damage was found to depend on the cellular structure parameters (i.e. cell size and density), composition, as well as thermal cycling variables, such as maximum temperature, cooling rate, etc.

1 Introduction

Cellular solids consists of an interconnected network of solid struts or walls which form the edges and faces of cells. These materials can be broadly divided into two groups, honeycombs and foams. In honeycombs, cells form a two dimensional array, whereas foams are comprised of a three dimensional array of hollow polygons. Foams are being considered for a variety of uses as a result of their low mass and large crushing strains. Applications of open cell ceramic foams usually exploit their high temperature stability and permeability, e.g. molten metal filters to remove any crass or non-metallic impurities from aluminum and iron based melts. These materials also have potential application as radiant burners in the natural gas industry. In a radiant burner, natural gas and air are fed from the back of a porous ceramic substrate with the combustion occurring on or near the front surface, thereby heating the substrate to very high temperatures. During operation, the heated surface can achieve temperatures close to 1300°C. However, significant thermal gradients can develop through the thickness of the substrate during operation and shut down.

As can be surmised, ceramic foams are often subject to high temperatures, large thermal gradients, and the thermal loading is often cyclic in nature, i.e. the material undergoes multiple heating and cooling cycles. The materials therefore require resistance to severe thermoelastic stresses and thermal fatigue during service. Currently the main drawback for the utilization of foams in these applications is the lack of knowledge about their high temperature behaviour and long term reliability in severe thermal environments.

The objective of the present study was to investigate the thermal fatigue behaviour of a wide range of open cell ceramic foams. The damage incurred by the foams was quantified by non-destructive (elastic modulus measurement) and destructive (strength measurement) test methods. Finite element analysis was also used to develop a thermoelastic model to predict the thermal stresses generated in the foams during cycling.

2 Background

A number of micromechanical models have been proposed to understand the mechanical behaviour of cellular materials. These models assume that the behaviour of the unit cell is representative of the bulk foam. There are a wide variety of cell shapes that can fill three-dimensional space, such as hexagonal and triangular prisms, dodecahedron, tetrahedron, etc. The shape of the cells is, however,

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governed by several factors besides space filling constraints, such as surface tension, processing variables, etc. Real foams, are thus, not regular arrays of identical unit cells, but instead exhibit variations in shape and cell size and the exact shape and size of the cells may influence the properties of foams. Several unit cells, such as simple cubic and tetrakaidecahedron have been proposed and the mechanical properties of foams derived in terms of the nature of cellular structure and the properties of solid struts.[1] Attempts have also been made to understand the thermal shock behaviour of cellular materials using these equations.

Gibson and Ashby[1] showed that for rapid quenches, thermal shock resistance (critical temperature change to initiate crack growth) of ceramic foams ΔT_{cf} can be expressed as;

$$\Delta T_{cf} = \frac{0{\cdot}65 \Delta T_{cs}}{(\rho/\rho_s)^{0{\cdot}5}} \quad (1)$$

where ΔT_{cs} is the resistance of the ceramic but in a non-porous form. The above expression includes the effect of density on the elastic modulus and strength of foams. Brezny and Green [2] have shown that the strength of struts can depend on the density and cell size of foams and this would need to be considered in a more exact analysis. In addition, the density and cell size are expected to control the heat transfer between the foam and surroundings. Hence, these variables are likely to have a stronger influence on the thermal stresses and thermal shock resistance of foams than is implied by eqn (1). More recently, a parameter R'_f has been proposed to predict the effect of density and cell size on the thermal shock resistance of foams.[3]

Previous work on thermal shock behaviour of open cell foams confirms that the thermal shock resistance of cellular ceramics is strongly dependent on cell size, increasing with increase in cell size, and being weakly dependent on density.[4] Orenstein and Green[4] pointed out that two different temperature gradients must be considered when an open cell foam is subjected to a rapid change in temperature. The first is a gradient across individual struts and the second is the gradient across the bulk section of the specimen. In their study, the specimens were quenched into oil or water to produce severe temperature gradients. The elastic modulus and strength were found to decrease gradually with increase in the quench temperature and it was determined that the retained foam stiffness was a good predictor of the retained foam strength after the thermal shock. In this study, it was assumed that the liquid quenching media could infiltrate the open-cell structure, which will reduce the bulk temperature gradients across the sample.

The limited information about the thermal shock resistance of ceramic foams suggests that the cell size and density of foams will play an important role in determining the extent of damage in these materials. There is practically no information available about the thermal fatigue behaviour of these materials. The objective of this research was to relate the damage induced by thermal cycling to baseline properties, such as elastic modulus, thermal expansion coefficient, thermal conductivity, and flexure strength and to determine if thermal fatigue might limit the lifetime of these foams.

3 Materials Studied

In this study, the thermal cycling behaviour of silica-bonded silicon carbide (SiC), alumina, alumina–11 wt% zirconia (AZ), and yttria stabilized zirconia–40 wt% alumina (YZA) was assessed. The AZ foams were obtained from Astro Met Associates, Cincinnati, OH, whereas all other materials were obtained from Selee Corporation, Hendersonville, NC. The cell size of the foams varied from 1·0 to 2·5 mm and the nominal relative density varied from 10 to 19%.

The reticulated structure of an open cell silica bonded silicon carbide foam is shown in Fig. 1. The microstructure of the as-received materials was characterized using scanning electron microscopy (SEM). In the as-received state, cracks were observed on the strut surface and along the cell edges in the AZ foams whereas in SiC and YZA foams, smaller scale microcracks were observed at the level of the grain structure. The microstructure of a strut in YZA foam is shown in Fig. 2, where both inter- and intra-granular cracks are seen in the as-received condition. All the foams used in this study were manufactured by using a technique

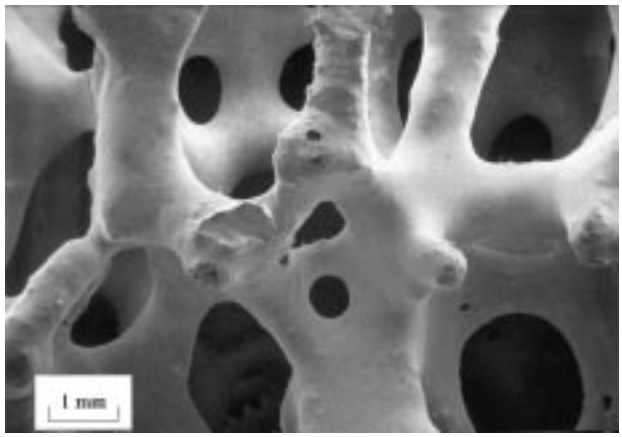

Fig. 1. Structure of an open cell silicon carbide foam.

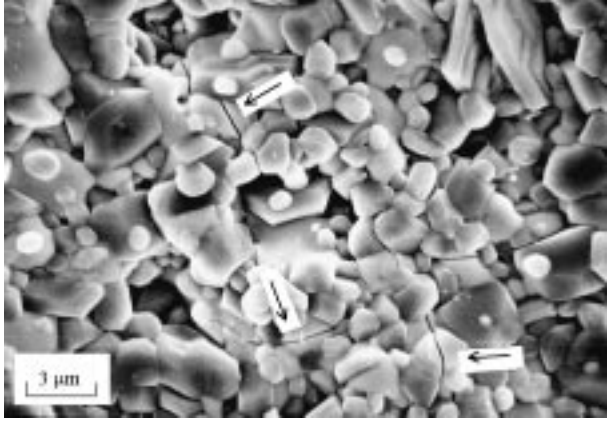

Fig. 2. Microstructure of as-received YZA foams showing inter- and intra-granular cracks.

in which an open cell polymer foam is coated by a ceramic slurry, which is following by drying and densification. The strut cracks in AZ foams are likely due to non-uniform coating of the slurry during the processing of the foams.

4 Experimental Procedure

An experimental approach based on infrared heating and forced air cooling was developed to perform systematic thermal cycling experiments. The details of the set-up have been presented elsewhere.[5] Foams were heated to the maximum temperature, held for about two minutes and cooled to room temperature by forced air jets directed on the surface of samples. Silica bonded silicon carbide (SiC) and alumina–zirconia (AZ) foams were heated to 1000°C whereas the yttria stabilized zirconia alumina (YZA) foams were heated to 1200°C. After thermal cycling, the elastic modulus was measured by dynamic resonance.

The extent of damage in the foams can be represented by the retained elastic modulus after cycling and was defined as,

$$D_E = \frac{E_0 - E}{E} \tag{2}$$

where E is elastic modulus after cycling, E_o is the modulus of as-received sample, and D_E is a damage parameter. D_E can vary from 0 in the as-received state to infinity after thermal cycling.

The extent of damage by thermal cycling was also determined by measuring the flexural strength of samples as a function of the number of thermal cycles. A damage parameter D_s can be defined to represent the extent of decrease in strength of foams after cycling, as,

$$D_s = \frac{\sigma_0 - \sigma}{\sigma} \tag{3}$$

where σ is the average strength after cycling and σ_0 is the average strength of samples in the as-received state.

Silica-bonded silicon carbide foams were rapidly heated to 1000°C, held for 2 min and then cooled to room temperature by forced air jets. Figure 3 shows the change in the retained elastic modulus and damage parameter D_E with increase in the number of cycles for 2·5 mm cell size silicon carbide foams. As can be seen, the damage increases with increase in the number of cycles, although most of the damage seems to occur in the first few cycles. Figure 4 shows the change in the retained strength and damage parameter D_S with increase in the number of cycles for these foams. The increase in D_S follows a similar trend to D_E with a marked increase in the first few cycles followed by saturation behaviour.

A decrease in the elastic modulus indicates an overall damage in the entire specimen, whereas the drop in the strength is usually related to the largest crack produced by the thermal shock. For every application, even if the primary function is not load bearing, the material needs to have a minimum stiffness and strength. Hence, for design purposes both parameters are required as a function of the number of cycles. A plot of D_E versus D_S in silicon carbide foams shows a very good correlation between the two parameters (Fig. 5). The figure indicates that the strength of foams is more sensitive to the damage occurring during thermal cycling than the elastic modulus. A good correlation between the two parameters suggests that the decrease in the elastic property may be used to predict a decrease in the strength of these foams.

5 Results and Discussion

5.1 Effect of cell size and density

Ceramic foams are available commercially in different cell sizes and densities. The effect of cell size on the thermal cycling resistance was analyzed

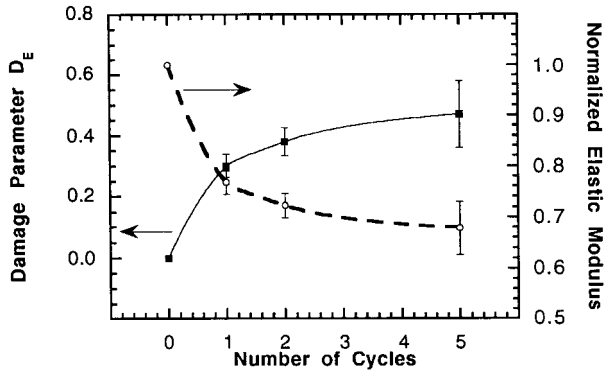

Fig. 3. Variation in retained elastic modulus and damage parameter D_E as a function of number of thermal cycles in 2·5 mm cell size silicon carbide foams.

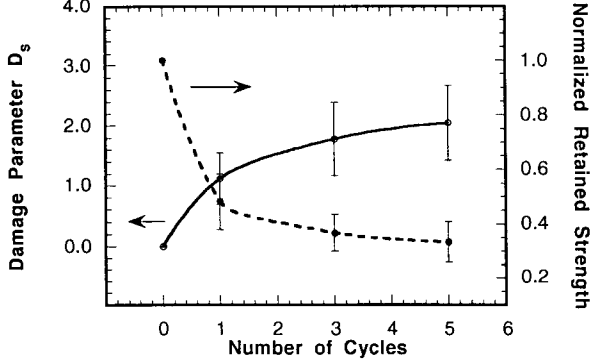

Fig. 4. Variation in retained strength and damage parameter D_S as a function of number of thermal cycles in 2·5 mm cell size silicon carbide foams.

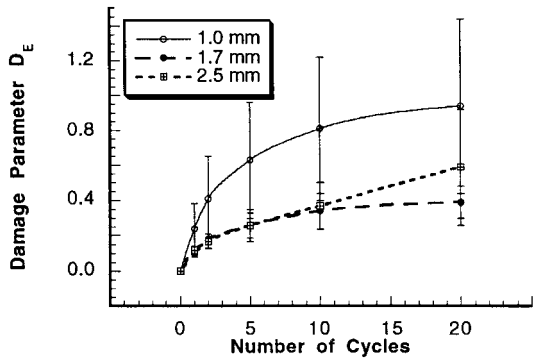

Fig. 6. Variation of damage parameter D_E with number of cycles for three different cell sizes in SiC foams.

using three different cell sizes of silicon carbide (SiC) and yttria stabilized zirconia-alumina (YZA) foams. The SiC samples were rapidly heated to 1000°C, held for 2 min, followed by forced air cooling. The variation in damage as a function of thermal cycles for 1·0, 1·7, and 2·5 mm cell size foams is shown in Fig. 6. As can be seen, more damage occurs in the samples with small cell size.

The effect of cell size on the thermal shock resistance was also analyzed in YZA foams. The samples were heated rapidly to 1200°C, held for 2 min, followed by forced convective cooling to room temperature. The variation in damage with the number of cycles for 1·0, 1·7, and 2·5 mm cell size foams is shown in Fig. 7. As can be seen, the extent of damage increases with decreasing cell size: similar behaviour to the silicon carbide foams. The thermal conductivity of foams depends very strongly on the cell size, decreasing rapidly with decrease in cell size at high temperatures.[3] This increase in heat transfer with increase in cell size leads to more severe macroscopic thermal gradients in samples with small cell sizes and hence, causes more damage.

The effect of density of foams on the thermal fatigue resistance was analyzed using three different densities of alumina–zirconia (AZ) foams. The samples were heated rapidly to 1000°C, held for 2 min, followed by forced convection cooling to room temperature. Figure 8 shows the variation of damage with the number of cycles for 0·10, 0·14, and 0·18 dense foams. An interesting effect was observed where the maximum damage was detected in 0·14 dense samples and was found to be similar for 0·10 and 0·18 dense foams. The effect of density on the thermal shock and fatigue resistance is complicated because density may play various roles in controlling the elastic modulus, strength, and thermal conductivity of foams and it is a combination of these parameters that controls the thermal shock behaviour. A careful thermal stress analysis showed that for 0·10, 0·14, and 0·18 dense samples, the induced thermal stresses were comparable to their respective bend strengths. This suggests that the density of foams may have a poorly defined role in determining the extent of damage.[6]

The thermal fatigue damage discussed above was also expressed in terms of an empirical equation given by:

$$\frac{D_E}{D_{ES}} = 1 - \exp(-\alpha N) \tag{4}$$

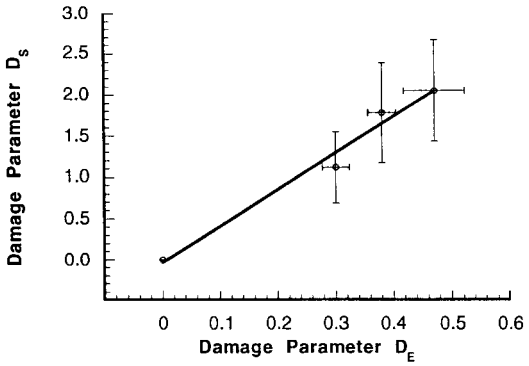

Fig. 5. Comparison between the modulus and strength damage parameters.

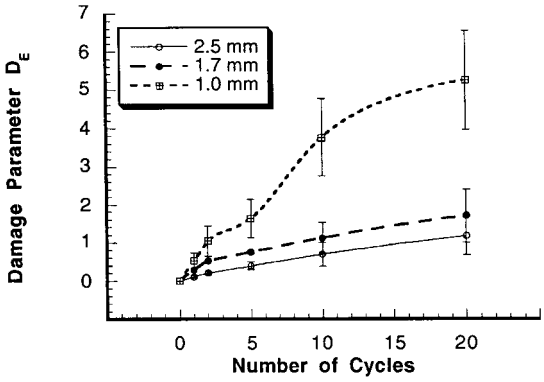

Fig. 7. Variation of damage parameter D_E with number of cycles for three different cell sizes in YZA foams.

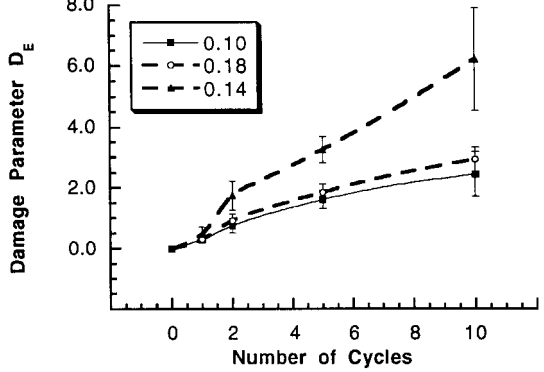

Fig. 8. Effect of density on the extent of damage in alumina–zirconia (AZ) foams.

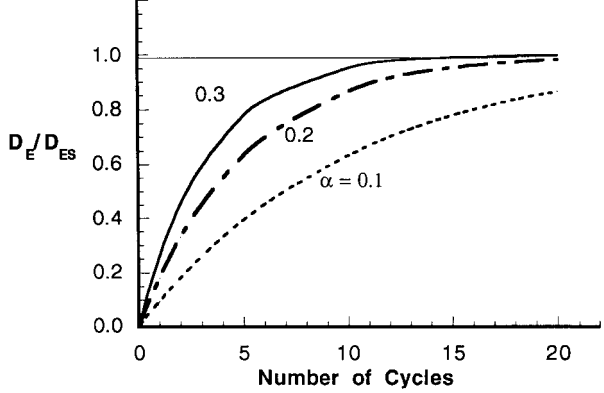

Fig. 9. Schematic of saturation of damage in foams.

Where D_E is the damage, D_{ES} is the damage saturation level, α is the rate constant, and N is the number of cycles. The thermal fatigue saturation behaviour observed in various foams under different thermal cycling conditions can be described by D_{ES} and α in eqn (4) and is shown schematically in Fig. 9 for various α values. The values of D_{ES} and α for SiC, YZA, and AZ foams for different cell sizes and density are shown in Table 1. As seen from the data, the damage saturation parameter D_{ES} depends on the cell size and density in the way discussed earlier and is, therefore, a measure of the overall thermal shock resistance of the particular foam. In contrast, the rate constant, α, seems to be invariant for a given material, suggesting that it may depend only on the fatigue damage mechanism associated with that material. Included in Table 1 are data obtained when the maximum temperature and heating or cooling rates were changed and these will be discussed in the next section.

5.2 Effect of maximum temperature and heating/cooling rates

A set of experiments were conducted on SiC and YZA foams to study the effect of maximum temperature and heating/cooling rates on the thermal fatigue behaviour of foams. The 2·5 mm cell size SiC foams were rapidly heated to 1000 and 1100°C, held for 2 min, and cooled to room temperature by forced air jets. The extent of damage for the two samples as a function of the number of cycles is shown in Fig. 10. As can be seen, the maximum temperature has a significant effect on the damage in silicon carbide foams with more damage occurring at higher temperatures. This is due to higher thermal stresses generated in the samples when cooled from a higher temperature.

To determine the effect of heating rate on the extent of damage, SiC foams were heated at 1, 10, and 100°C min. to 1000°C and cooled to room temperature by forced air jets. The heating rate did not have any affect on the damage behaviour of foams, as the extent of damage was found to be same in all three samples. The effect of cooling rate was determined by heating the SiC samples rapidly to 1000°C, holding for 2 min, followed by cooling to room temperature at three different cooling rates, as shown in Fig. 11. Cooling rate *A* was air cooling by natural convection, and *B* and *C* were cooling with forced air jets. The extent of damage as a function of thermal cycles for the three different cooling rates is shown in Fig. 12. There was no damage observed in the samples subjected to natural

Table 1. Thermal fatigue damage parameters for foams

Material	*Cell size (mm)*	*Density (%)*	*Max. temperature (°C) and cooling rate*	D_{ES}	α
SiC	1·0	0·14	1000 fast	0·81	0·34
SiC	1·7	0·14	1000 fast	0·34	0·34
SiC	2·5	0·14	1000 fast	0·37	0·30
YZA	1·0	0·12	1200 fast	5·25	0·11
YZA	1·7	0·12	1200 fast	1·70	0·13
YZA	2·5	0·14	1200 fast	1·17	0·10
YZA	2·5	0·14	1200 slow	0·26	0·13
YZA	2·5	0·14	1350 slow	1·61	0·16
AZ	1·3	0·10	1000 fast	2·44	0·22
AZ	1·3	0·14	1000 fast	6·21	0·18
AZ	1·3	0·18	1000 fast	2·93	0·21

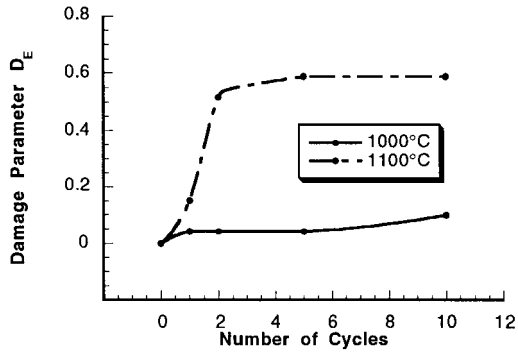

Fig. 10. Effect of maximum temperature on the damage in 2·5 mm cell size silicon carbide foams.

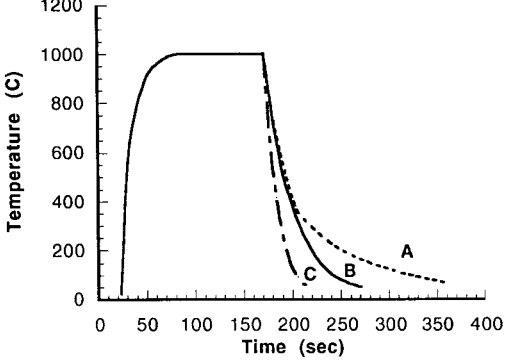

Fig. 11. Temperature profiles showing three different cooling rates used for cycling silicon carbide foams. *A* was air cooling by natural convection, and *B* and *C* were cooling with forced air jets.

convection (cooling rate *A*). In the samples subjected to cooling rates *B* and *C*, the damage seemed to occur in a step-function like fashion, where it appears to be saturated for a few cycles, and then suddenly increased. During these intermediate saturation periods, localized damage might have occurred in small regions but was presumably not enough to decrease the elastic modulus of the entire sample. However, after a few cycles the damage accumulated, resulting in a drop in elastic modulus and increase in the damage parameter D_E. The other possibility is that the elastic modulus measurement technique is not sensitive enough to detect small decreases in elastic modulus. Lee and Case[7] observed a similar behaviour in polycrystalline alumina samples subjected to thermal shock by oil quenching.

The effect of maximum temperature and cooling rate was also investigated on the yttria stabilized zirconia–alumina (YZA) foams using two different maximum temperatures (1200 and 1350°C) and two different cooling rates (natural convection and forced air cooling). It was found that the maximum temperature and cooling rate also have a significant influence on the extent of damage in these foams, with more damage occurring at the higher

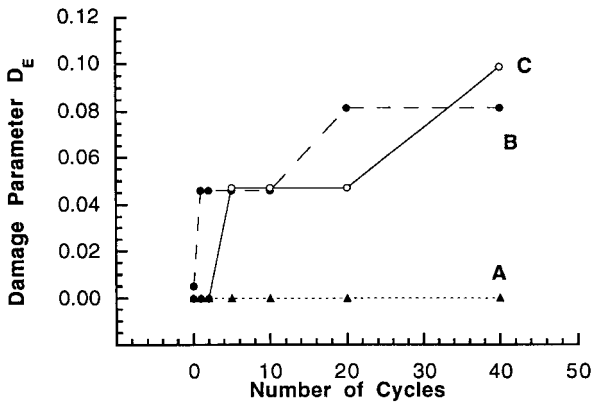

Fig. 12. Effect of cooling rate on the damage in 2·5 mm cell size SiC foams.

hold temperature and faster cooling rate. Such behaviour is consistent with the expected changes in the thermal stress.

5.3 Thermal fatigue in ceramic foams

Clearly, it is of interest to determine the cause of the fatigue effect in these foams. In order to determine the source of the damage due to thermal cycling, foams were analyzed by scanning electron microscope and X-ray diffraction. In the as-received silicon carbide foams, a number of cracks were observed at the microstructual level. An increase in the volume of the sample after thermal cycling indicated a possible increase in microcrack density of foams, but an increase in the surface microcrack density was not observed. In silica-bonded silicon carbide foams, quantitive XRD showed that the as-received materials consists of approximately 6 wt% cristobalite and the percentage of cristobalite was also found to increase after thermal cycling. To confirm the crystallization of silica, a few samples were annealed at 1000°C for 6 h and then cooled to room temperature. In these samples cristobalite was found to be high as 12 wt%.[6] The transformation of silica to cristobalite involves a change in volume and suggests that this could lead to a decrease in the elastic modulus due to microcracking. This is an additional factor which could contribute to damage in SiC foams, besides thermal stress.

In YZA foams, cracks were observed in alumina and zirconia grains in the as-received samples. The average length of cracks was found to increase from 3·7 to 10·1 μm after 20 cycles to a maximum temperature of 1200°C. In alumina–zirconia (AZ) foams, the extent of cracking on the stuts was found to increase after thermal cycling.[6] Dense YZA samples, of the same composition as the foams, were subjected to isothermal annealing to determine if any phase changes occur in theses samples during thermal cycling. XRD patterns for the samples annealed for a period of 3 h at 1000 and 1100°C are shown in Fig. 13. The percentage of monoclinic phase was found to increase with increase in annealing temperature. This shows that the phase stability in these materials could be an important issue. Thermal expansion behaviour of YZA suggests that during cycling, monoclinic zirconia transforms to tetragonal around 800–1000°C. Thus one concludes that repeated phase transitions during cycling can lead to additional damage in these materials. The transformation of tetragonal zirconia to monoclinic is accompanied by a volume change and might lead to microcracking in the material.

In order to determine if any damage is occurring due to thermochemical effects, i.e. due solely to the fact that the material is at a high temperature,

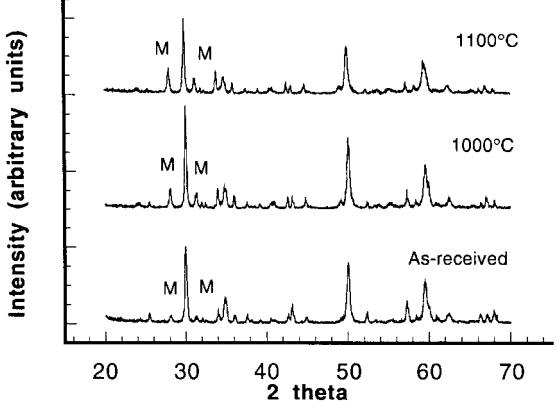

Fig. 13. X-ray diffraction patterns of YZA showing an increase in monoclinic phase (M) with isothermal annealing.

foams were heated slowly in a box furnace to the maximum temperature, held for a period of time, and cooled slowly to room temperature. Silicon carbide foams were heated at 5°C min^{-1} to 1000°C, held for 80 min, and cooled to room temperature. The decrease in the modulus and extent of damage was compared to samples subject to 40 cycles to 1000°C with a 2 min hold at the maximum temperature. Yttria stabilized zirconia-alumina foams were heated at 5°C min^{-1} to 1350°C, held for 40 min, and cooled slowly to room temperature. The extent of damage was compared to YZA foams subject to 20 cycles to 1350°C with 2 min hold at the maximum temperature. The data for SiC and YZA foams are shown in Table 2. The extent of damage in foams subject to isothermal heat treatment is much less than those subject to thermal cycling, suggesting that thermal stress is the major cause of the damage in these foams and not the phase changes.

The silica-bonded silicon carbide foams may be susceptible to creep damage at high temperatures due to the presence of high amount of glass phase (15 wt%). To determine the degree of such inelastic behavior, foams were subjected to constant loads corresponding to an initial stress of 0.18, 0.35, and 0.53 MPa in four point flexure at 1000°C. Creep rates of the order of 10^{-4}–10^{-3} sec were observed in the foams. The stress exponent for creep rate was also calculated and found to be 2.1. The creep rates observed were significantly higher than those found in dense ceramics and suggests that large strains during the hold periods at the maximum temperature could lead to additional damage in these materials. More detailed analysis is required to determine the mechanism and the extent of creep damage. The data also suggest that relaxation of thermal stresses may be possible during the thermal shock of these foams.

Table 2. Comparison of damage during thermal cycling and isothermal heat treatments

	Thermal cycling		*Isothermal heat treatment*	
Material	E/E_o	D_E	E/E_o	D_E
SiC	0·88	0·14	0·95	0·05
YZA	0·53	0·89	0·96	0·04

It is also important to determine whether the thermal stress changes during thermal cycling. A detailed thermal stress analysis showed that the bulk thermal gradients across the thickness of foams were the main source of damage in the foams and that microscopic gradients across the individual struts were not likely to induce the damage observed in the current experiments.[6] A close correlation between the increase in damage parameters and thermal stresses during the first cycle indicated that the foams were damaged by the thermal stresses. The observed decreases in the elastic properties, discussed earlier, can be used to predict the variation in thermal stresses with the number of cycles.

Budiansky and O'Connnel[8] determined the effect of randomly oriented microcracks on the elastic properties of brittle materials. For a first approximation, the elastic modulus of a material containing elliptical cracks can be expressed as;

$$\frac{E}{E_0} = \frac{1}{1 + Na^3} \tag{5}$$

where N is the microcrack density per unit volume and a is the major axis crack length.

The thermal conductivity of a material will also be influenced by the presence of microcracks. The maximum effect will be at elevated temperatures when the crack spacing limits the phonon mean free path. The effect of statistically randomly oriented microcracks on thermal conductivity can be expressed as[9]

$$\frac{K}{K_0} = \frac{1}{1 + (8Na^3/9)} \tag{6}$$

After thermal cycling, the microcracks in foams were assumed to be randomly oriented. This is a reasonable assumption considering the random orientation of struts and the complicated thermoelastic stress state in the interconnected structure.

Based on the above discussion, an increase in microcrack density will decrease both the elastic modulus and thermal conductivity of the material. These two properties will affect the thermal gradients and thermoelastic stresses induced in the foams. The expressions for the change in elastic modulus and thermal conductivity are very similar, and by knowing the decrease in elastic modulus, the variation in thermal conductivity can be estimated using eqns (5) and (6). For each thermal cycle, using the measured decrease in elastic modulus values, $Na^3 (= D_E)$ was calculated. The Na^3 values were then used in eqn (6) to predict the decrease in

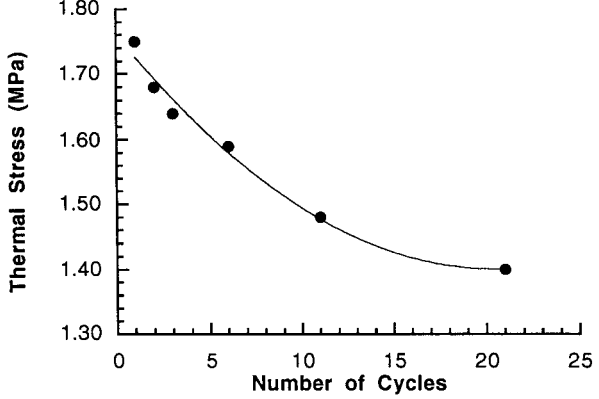

Fig. 14. Variation in thermal stress with number of cycles in YZA foams.

the thermal conductivity. The bulk thermal stresses in foams were calculated as a function of cycles using finite element analysis. The variation in thermal stresses with cycling is shown in Fig. 14. As can be seen, the thermal stresses in foams decrease gradually with an increase in the number of cycles. The thermal stress calculations were based on the assumption that during thermal cycling damage occurs uniformly through out the sample leading to a decrease in the elastic properties. However, the finite element calculations showed that the maximum tensile thermal stresses occur only at the foam surface. Therefore after the first cycle, foams are like a layered composite, where the properties of core or interior remain the same with little or no damage and only the outer layers are damaged. The lower modulus outer layers will yield significantly lower thermal stresses than shown in Fig. 14, so that the thermal stresses are expected to decrease more rapidly with the number of cycles than predicted. Alternatively, damage may be spreading through the outer layers until it saturates. The gradual decrease in the thermal stresses is a possible explanation for the saturation of damage in foams after cycling. After a few cycles, the thermal stresses are presumably significantly lower than the stress required for crack propagation in the foams, causing no further damage in the samples. The major question is why the damage continues after the first cycle. This suggest that either sub-critical crack growth occurs in these materials or that the damage becomes more homogeneous as the thermal stress is repeated. For the SiC foams, it is also possible that creep damage may be involved in the fatigue process.

6 Summary

A number of experiments were performed on silica bonded silicon carbide (SiC), alumina, and alumina–zirconia foams to determine the extent of damage due to various sources during thermal cycling. The damage in foams was found to be strongly dependent on the cell size, increasing with a decrease in the cell size and weakly dependent on density, initially increasing with an increase in density and then decreasing. The extent of damage also increased with an increase in maximum temperature and cooling rate. It was determined that the damage in foams was mainly due to propagation of pre-existing cracks under thermal stress. The thermal fatigue behavior in foams was expressed in terms of damage saturation value and rate constant, using an empirical equation. It was found that the damage saturation value depends very strongly on the cell size, density, maximum temperature, and cooling rate during thermal cycling whereas the rate constant seemed to be invariant for a given material, suggesting that it depends on a particular damage mechanism for a given material.

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