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This is to certify that the
dissertation entitled
**Thermal Fatigue in Ceramics
and Ceramic Matrix Composites**
presented by
Won Jae Lee
has been accepted towards fulfillment
of the requirements for
Ph.D. degree in Materials Science

Eldon D. Case
Major professor

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**THERMAL FATIGUE IN CERAMICS
AND CERAMIC MATRIX COMPOSITES**

Volume I

By

WON JAE LEE

A Dissertation

**Submitted to
MICHIGAN STATE UNIVERSITY
in Partial Fulfillment of the Requirements
for the Degree of**

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Department of Metallurgy, Mechanics and Materials Science

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ABSTRACT

THERMAL FATIGUE IN CERAMICS AND CERAMIC MATRIX COMPOSITES

By

Won Jae Lee

Cyclic thermal shock damage by water quench in SiC whisker reinforced alumina ceramic-ceramic composites and polycrystalline alumina was monitored nondestructively via elastic modulus and internal friction measurements.

A power law relationship between the saturation damage level and thermal shock difference implies a fatigue-like power law relation in stress. The crack growth rates for the unreinforced polycrystalline ceramics are far more sensitive to ΔT than the composite materials used in this study. Exponents in the power law fatigue relations are functions of thermal shock resistance parameters such as R'''' and hence are likely also functions of materials parameters such as fracture toughness. The results of this study suggest the existence of threshold fracture toughness (or strength) above which thermal shock damage will not accumulate for a given ΔT .

A technique for measuring the surface heat transfer coefficient, h , is developed for quenched ceramics that combines a parameter estimation method with a fast-response capability of recently

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recently developed thin-film thermocouples. The surface heat transfer coefficient values were measured for alumina quenched into water, oil and liquid nitrogen. Measurements of heat transfer coefficient, h , for water, silicone oil and liquid nitrogen quenches show that h for silicone oil is an order of magnitude higher than for liquid nitrogen and an order of magnitude lower than for water.

The surface tensile stress developing during quenching was calculated using temperature-dependent function of the measured surface heat transfer coefficient, the Young's modulus, the coefficient of thermal expansion, Poisson's ratio, and thermal shock severity.

For polycrystalline alumina, plots of $\ln(A/E_o)$ versus $\ln(\sigma_{\max}/\sigma_f)$ for water and silicone quench show very different slopes. However these two different curves collapse into a single curve if we consider the duration time as well as the stress. A plot of normalized damage, A/E_o versus the product between stress $\sigma(f)$ and duration time Δf for the polycrystalline alumina quenched into water and oil bath shows a power law relation which is similar to the power law equation in A/E_o and $\Delta T/T_r$.

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1. INTRODUCTION

In recent years, ceramics have been studied extensively because of their applications as structural components, especially at elevated temperature. Mechanical properties such as fracture strength and creep properties have been investigated in considerable depth. However, comparatively a little is known about the fatigue behaviour of these materials under cyclic stress conditions.

Most ceramic material are extremely susceptible to thermal shock damage and can fail catastrophically under thermally induced stresses. Thermal shock may result from any rapid temperature changes which may cause restraints on the thermal expansion due to the thermal gradients developed within the material. This causes strength degradation. Nevertheless many significant applications of ceramics such as gas turbine components, high temperature valves, and heat engine components are subject to cyclic thermal shock. Hence a detailed investigation of the phenomena of cyclic thermal shock is necessary.

A number of difficulties are encountered in fatigue testing of brittle materials. In order to characterize the strength of brittle ceramic materials, a large number of test specimens is required [1-3]. Scatter in strength data may sometimes obscure the fundamental trends in the data [1-3].

Nondestructive techniques such as elasticity and internal friction can measure changes induced by thermal shock damage [4-14]. In addition, the crack-induced changes in the elasticity and internal

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friction reflect the entire flaw population [10], whereas the strength changes can be attributed to changes in the critical flaws lengths.

One method of studying thermal shock damage of ceramic includes quenching a specimen from higher temperature into a lower temperature quenching media. Water is the popular quenching media used in thermal shock studies. Other quenching media can include oils [2, 6, 15, 16], liquid metals [17], salt mixtures [16], air [18, 19] or fluidized beds [20]. Each quenching media has different heat transfer characteristics since the surface heat transfer rates are different. The heat transfer rate at the surface of the body has a significant effect on the transient temperature and stress distribution [21]. Thus, the thermal shock damage may be different for different quench media even if the ΔT , the temperature difference between the specimen and the quenching media, is same.

The present study focuses on cyclic thermal shock damage by water quench in SiC whisker reinforced alumina ceramic-ceramic composites and polycrystalline alumina. The evolving crack damage was monitored nondestructively via elastic modulus and internal friction measurements. Thermal fatigue damage in SiC whisker reinforced alumina composites [13] and polycrystalline alumina [14] is compared to the thermal fatigue damage results for Macor machinable glass-ceramic (particulate reinforced composite in this case) [12], SiC fiber reinforced aluminosilicate composites [11] and titanium diboride in terms of fracture toughness, thermal shock damage parameters and experimentally determined thermal fatigue parameters.

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Young's modulus and internal friction measurements are presented for thermally cycled polycrystalline alumina quenched into a 20 C silicone oil bath and into a liquid nitrogen bath. The results are analyzed in terms of the variation of heat transfer coefficient for each quenching medium and temperature-dependent material properties.

2. LITERATURE

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2. LITERATURE REVIEW ON FATIGUE OF CERAMIC

The accumulation of damage during thermal shock cycling and cyclic indentation, as was observed in this study, implies the existence of a fatigue process. Before discussing the results of this study, we shall briefly review fatigue studies in ceramics.

Fatigue effects in metals, for example, are linked to plasticity and dislocation motion [22], but crack tip plasticity is generally absent in ceramics [23]. In ceramics, fatigue effects may result from a variety of energy dissipative processes although crack tip plasticity is the particular energy dissipative mechanism that has been documented for metal fatigue.

In metals, observed fatigue crack propagation rates, da/dN , typically depend on the stress and crack length, such as [22]

$$da/dn = W \Delta K^q \quad (1)$$

where a - the length of the fatigue crack

n - the cumulative number of stress cycles

W, q - the parameters W and q depend on the material, the test temperature, etc.

and ΔK - the stress intensity range $K_{\max} - K_{\min}$.

Recent work [24-28] has shown that crack extension due to repeated mechanical loading of ceramics can be described in equations

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that are similar in form to those developed for fatigue crack propagation in metals. Evans has reviewed some plausible forms for fatigue relationships in ceramics [29]. In this study, we develop a power law expression for thermal fatigue damage that is similar to equation 1.

Table 1 summarizes the materials, test geometries, crack propagation rates, and proposed energy dissipation mechanisms for studies of repeated mechanical loading of ceramics. The authors in each of these studies expressed their crack propagation data in terms of equation 1 (except in reference [25], as noted in Table 1). Note that while experimentally determined values of the fatigue exponent, q , for metals typically ranges from about 2 to 7 [22], q ranges from about 9 to 27 for the ceramic materials included in Table 1.

Several researchers have noted effects such as fatigue crack closure and crack branching in ceramics under repeated mechanical loading. Dauskardt et al. [27] measured the extent of fatigue crack closure in stabilized zirconia using a strain gauge. Magnesia stabilized zirconia compact tension specimens showed evidence of fatigue crack closure behavior similar to that observed in metals. Dauskardt et al. [27] suggested that the fatigue in these toughened ceramics may be related to several mechanisms including crack tip shielding by transformation [30, 31], crack deflection [32, 33], crack bridging [34] and crack surface asperity wedging [35-41]. Optical micrographs of the crack path support the crack deflection and crack bridging hypothesis [27].

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c. three point
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2. MgO-PSZ

a. compressive
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b. compact-ten
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[27]

3. Silicon Ni

a. three point
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4. Westerly Gr

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Table 1. Survey of Fatigue Crack Behavior for Ceramics Subjected to Cyclic Mechanical Loading

Test Specimen (material & geometry)	da/dN range (m/cycle)	Fatigue exponent q	Proposed Dissipation Mechanism	Starting Flaws
1. Alumina				
a. wedge opening loaded [24]	$10^{-9} - 10^{-7}$	14	wedging & sliding asperity	notch
b. tapered double cantilever beam [24]	$10^{-9} - 10^{-6}$	27	wedging & sliding asperity	notch
c. three point bend*** [25]	$10^{-8} - 10^{-5}$	9.12	* indentation	flaws

2. MgO-PSZ				
a. compressive loading [26]	$10^{-9} - 10^{-7}$	13**	*	hole
b. compact-tension (specimens loaded in tension- tension) [27]	$10^{-10} - 10^{-6}$	24	transformation, crack closure, crack deflection & crack bridging	notch

3. Silicon Nitride				
a. three point bend*** [25]	$10^{-10} - 10^{-6}$	21.1	* indentation	flaws

4. Westerly Granite				
a. three point bend [28]	$10^{-10} - 10^{-8}$	12	crack closure & crack branching	slot

* Dissipation mechanisms were not proposed for this study.

** Fatigue exponent calculated by the present authors from da/dn versus K plot of Grossmuller [26].

*** The authors in this study expressed their crack propagation rate in terms of K_{eff} instead of ΔK .

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Kim and Mubeen [28] attribute the fatigue in Westery granite to the crack closure due to crack face interference [35-41] and crack branching [42]. SEM micrographs of replicas taken from the near-notch area in the granite specimens [28] indicate crack branching along the grain boundaries. From the morphology of the observed cracks, Kim and Mubeen assumed that the crack arrested at several points. The repeated crack arrest was assumed to correspond to a temporary reduction in da/dn , where da/dn was inferred from compliance measurements.

Under cyclic compression loading, Suresh and co-workers observed stable crack growth for pre-notched specimens of unreinforced polycrystalline alumina [35-38], SiC whisker reinforced Si_3N_4 [39], and SiC whisker reinforced alumina [40]. For each material, the macrocrack propagating from the pre-notch was monitored (on both sides of the specimen) by a traveling optical microscope. The macrocrack growth rates decreased monotonically as the crack length increased. A graph of crack length versus the cumulative number of load cycles showed a series of plateaus which may indicate steady state behavior at over certain intervals of cyclic loading. The plateaus in crack length during cyclic loading, as observed by Suresh, perhaps may be similar to the crack damage saturation behavior observed in the present study for thermally fatigued monolithic polycrystalline alumina, SiC whisker reinforced alumina composites, Macor machinable glass ceramics and in SiC fiber reinforced aluminosilicate.

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Suresh and co-workers explained the fatigue crack growth by a combination of several plausible physical mechanisms. Unreinforced ceramics, ceramic-metal composites, and ceramic-ceramic composites may involve different mechanisms, at least in part due to differing microstructures. For unreinforced ceramics, such as the polycrystalline alumina in Suresh's study [35-38] and the polycrystalline alumina in the present study, the possible mechanisms for the fatigue crack growth include crack wedging by crack debris [24], tensile residual stress at the notch tip [35], frictional sliding and opening of microcracks [42], and grain boundary cracking induced by compressive loads and residual thermal stresses [43].

A number of researchers have obtained experimental evidence for fatigue effects in ceramics. For example, Lewis and Rice [44-46] have noted fatigue effects in ceramics under both purely mechanical loading and under thermomechanical loading (thermal shock). Fatigue effects were observed in tests of unreinforced polycrystalline alumina as well as particulate-based composites including alumina, silicon nitride, and BN-mullite specimens. Lewis and Rice [44-46] attribute the fatigue to residual stress effects, including thermal expansion anisotropy in unreinforced ceramics and thermal expansion mismatch in ceramic composites.

In a study of cyclic mechanical loading in soda-lime glass, Lu and Evans [47] used a Hertzian indentation system to apply a constant normal load to a soda-lime glass plate via a spherical indenter.

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Superimposed on the constant normal load was a cyclic tangential load, induced by periodically traversing the indenter across a fixed corridor on the plate. For cyclic tangential loadings of up to 100 cycles, the loading produced ring cracks with a series of striations marking the surfaces of the ring cracks. These striations were assumed to demark periodic growth and arrest of the cyclically loaded crack [47]. The spacing between the striations on the ring crack was observed to decrease with an increasing number of stress cycles at fixed load, indicating "the crack growth per cycle tends to saturate" [47].

In cyclic mechanical loading studies on silicon nitride, SiC, and glass ceramics, Brugger [48] observed a fatigue-like "stagnation" in macrocrack growth after an initial period of crack growth. As a physical mechanism for this crack damage saturation, Brugger cited evidence of frictional interaction of asperities on opposing macrocrack faces.

The experimental observations of Lu and Evans [47], Lewis and Rice [44-46], Brugger [48], Reece [24], Grossmuller [26], Hoshide [25], Dauskardt [27], Kim and Mabeen [28] and Suresh [35-41] indicate the presence of fatigue effects in glass [47], toughened ceramics [26, 27], polycrystalline ceramics [24, 25, 36-38, 44-46, 48], geological materials [28], and ceramic-ceramic composites [40, 41, 44-46] under cyclic mechanical loading. The cyclic mechanical loading research cited above involves measurements on a single advancing macrocrack, although microcracks may be generated in the vicinity of the

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macrocrack. However in this dissertation, thermal fatigue studies of SiC whisker reinforced alumina composites, polycrystalline alumina, Macor machinable glass ceramics, and SiC fiber reinforced aluminosilicate composites give evidence of fatigue in thermal loading. Thermal fatigue studies involve "distributed" damage, in that it is no longer limited to a single advancing crack.

Fatigue in this study and each of the other studies cited above might be accounted for by the concept of frictional forces between opposing crack faces, where such frictional energy dissipation provides the energy dissipation mechanism leading to fatigue. In addition, for thermal fatigue studies, the stress profile as a function of depth and the limited temporal duration of the stress pulse contribute to the limited crack growth (Section 4.1.4.). Furthermore, whisker and particulate reinforced composites, various whisker (particulate)-crack interactions may contribute to the energy dissipation, and hence the observed fatigue behavior.

3. EXPERIMENTAL

3.1. Materials

3.1.1. Substrates

All substrates were supplied by the manufacturer. The substrates were tested by the following methods:

The substrates of 11.5 cm (Coors) diamond SiC paper were not intercalated. The substrates were annealed in a vacuum furnace at 950 degrees Celsius for 24 hours to remove any residual stress or due to the annealing process. The substrates are given by the following methods:

An average of 100 Coors ve. techniques were used. The average

3. EXPERIMENTAL PROCEDURE

3.1. Materials

3.1.1. Polycrystalline Alumina

Alumina substrates (96% alumina) used for water quench tests were supplied by Saxonburg Ceramic Incorporated (Monroe, NC). Alumina substrates (AD-96) used for silicone oil and liquid nitrogen quench tests were supplied by Coors Inc.

The as-received specimens were rectangular plates with dimensions of 11.5 cm x 11.5 cm x 0.1 cm (Saxonburg) and 11.4 cm x 11.4 cm x 0.1 cm (Coors). Specimens were cut into prismatic bars using a low speed diamond saw. The edges of cut specimens were polished using 600 grit SiC paper to obtain a more uniform width however, the specimens were not intentionally edge beveled. The specimens for water quench tests were annealed in air at 1150 degrees Celsius for 12 hours. Specimens quenched into silicone oil and liquid nitrogen were annealed in air at 950 degrees Celsius for 12 hours. The pre-test anneal of the specimen remove residual stresses that may have been present due to processing or due to specimen preparation. The dimensions of annealed specimens are given in table 2.

An average grain size of 10 μm and 7 μm for Saxonburg vendor and Coors vendor, respectively, were determined by the linear intercept technique on SEM micrographs on fracture surfaces (figures 1a and 1b). The average grain size was computed by multiplying the average

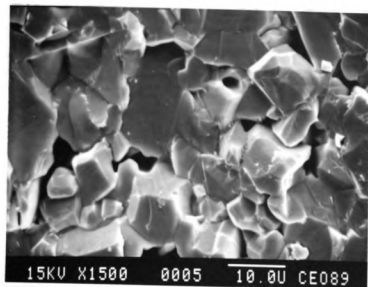


Figure 1a. A SEM micrograph showing a fracture surface of the polycrystalline alumina specimens (Saxonburg).

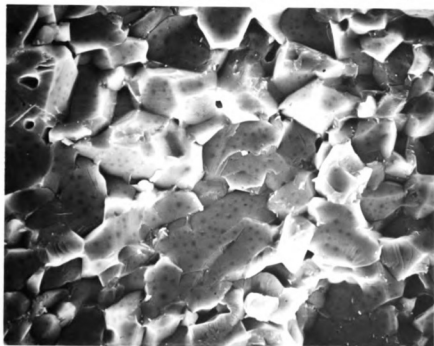


Figure 1b. A SEM micrograph showing a fracture surface of the polycrystalline alumina specimens (Coors) ($\times 2000$).

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Table 2. Dimensions, Mass, and Mass density of Polycrystalline Alumina Specimens

Specimen Label	Dimensions (cm)	mass (g)	density (g/cm ³)
*	A1	11.48 x 1.23 x 0.1	5.6741
	A2	11.45 x 1.27 x 0.1	5.8513
	A3	11.39 x 1.28 x 0.1	5.7738
	A4	11.47 x 1.28 x 0.1	5.8162
	A5	11.48 x 1.26 x 0.1	5.8148
	A6	11.49 x 1.24 x 0.1	5.7210
**	AN1	6.97 x 1.20 x 0.1	3.1359
	AN2	6.96 x 1.20 x 0.1	3.1394
	AN3	6.98 x 1.20 x 0.1	3.1599
	AN4	6.98 x 1.20 x 0.1	3.1511
***	A01	10.00 x 1.27 x 0.1	4.8690
	A02	10.00 x 1.28 x 0.1	4.9692
	A03	10.51 x 1.29 x 0.1	5.1715
	A04	10.00 x 1.29 x 0.1	4.9090
	A05	8.50 x 1.28 x 0.1	4.1300
	A06	9.14 x 1.31 x 0.1	4.6523
	A07	8.97 x 1.33 x 0.1	4.5368
	A08	10.00 x 1.27 x 0.1	4.8295

* Water quenched specimens (Saxonburg)

** Liquid nitrogen quenched specimens (Coors)

*** Silicone oil quenched specimens (Coors)

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intercept length by a correction factor of 1.5 [49]. The mass density of the polycrystalline alumina used in this study, as determined from the mass and dimensions of each specimen, ranged from 3.68 to 3.82 g/cm³. This corresponds to a volume fraction porosity of approximately 3 to 7.4 percent.

3.1.2. SiC Whisker Reinforced Alumina Composites

SiC Whisker reinforced alumina composites used for this research were produced by Alcoa.* The material consists of A-16 SG alumina (a super ground calcined alumina), reinforced with both the 20 volume percent Arco and Tokai carbon SiC whiskers. Processing was carried out using pH stabilization in a wet procedure. The material was then dried and hot pressed.

The dimensions of the as-received specimens for modulus and internal friction measurements are shown in table 3. Table 4 lists some properties of the samples used for this research. Mass density of the specimens ranged from 3.76 to 3.80 g/cm³. The average grain size of the alumina matrix was approximately 4 to 6 microns, as determined by linear intercept technique on SEM micrographs of fracture surfaces, again computing the average grain size by

* The whisker reinforced ceramic composites were obtained from D. J. Bray, Technical Supervisor, Alcoa Technical Center, Alcoa Center, PA.

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Table 3. Dimensions and Mass of SiC Whisker Reinforced Alumina Composite Specimens

Specimen	Dimensions (cm)	Mass (g)
HP158	7.01 x 0.702 x 0.297	5.5893
HP160	7.01 x 0.700 x 0.297	5.6291
HP171	7.00 x 0.700 x 0.298	5.6039

multiplying the average intercept length by 1.5 [49]. A SEM micrograph of a fracture surface of the SiC whisker reinforced alumina composite is given in figure 2.

3.2. Room Temperature Elasticity Measurement

The sonic resonance technique described by Forster [50] was used to determine the elastic moduli and internal friction. Figure 3 shows schematically the experimental arrangement used. The modulus equipment measures the resonant frequencies of the specimen. Cotton strings suspended the prismatic specimen horizontally from a driver and a phonograph pickup (Figure 4).

An electronic signal produced by a frequency generator (3325A Synthesizer/Function Generator made by Hewlett-Packard) was amplified and converted into mechanical vibrations via a high power

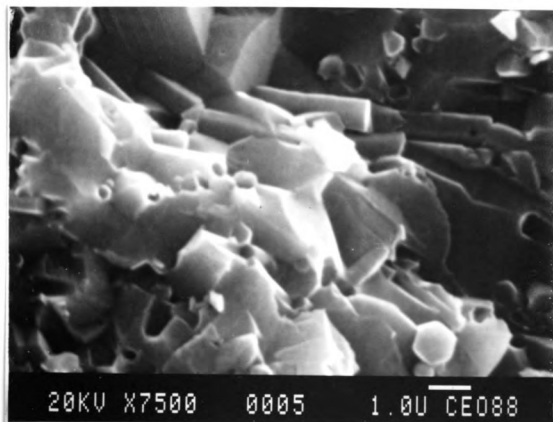


Figure 2. A SEM micrograph showing a fracture surface of the SiC whisker reinforced alumina composite specimen.

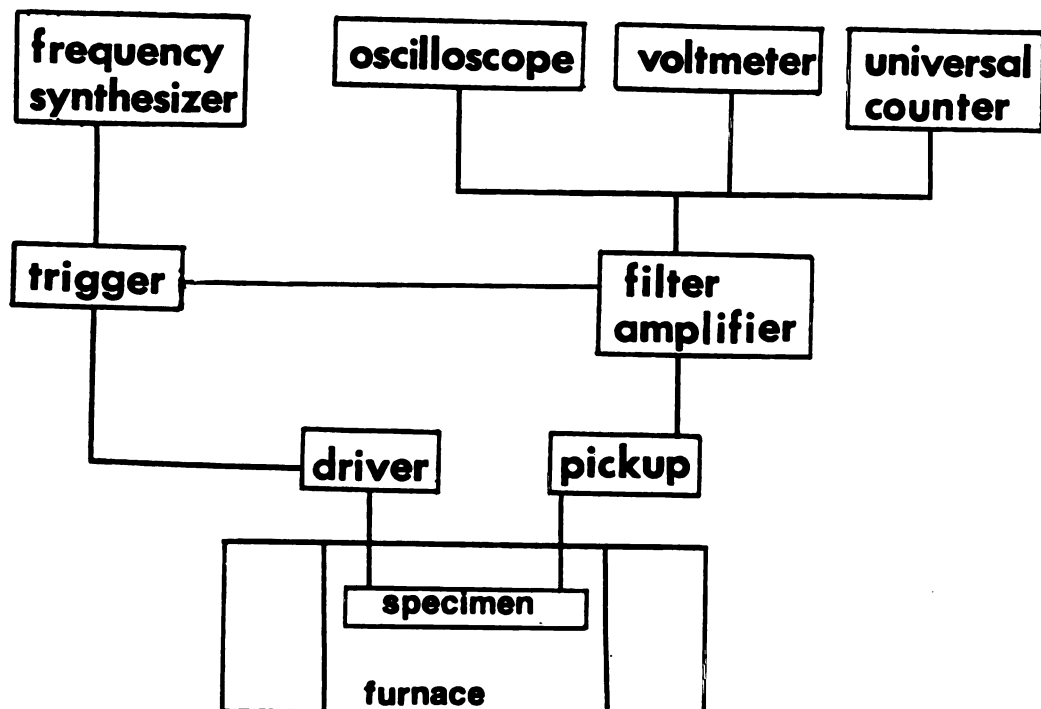


Figure 3. Schematic of the sonic resonance apparatus for determining the elastic moduli and the apparatus for determining internal friction via the log decrement method (free decay of the natural vibration modes).

Figure 4.

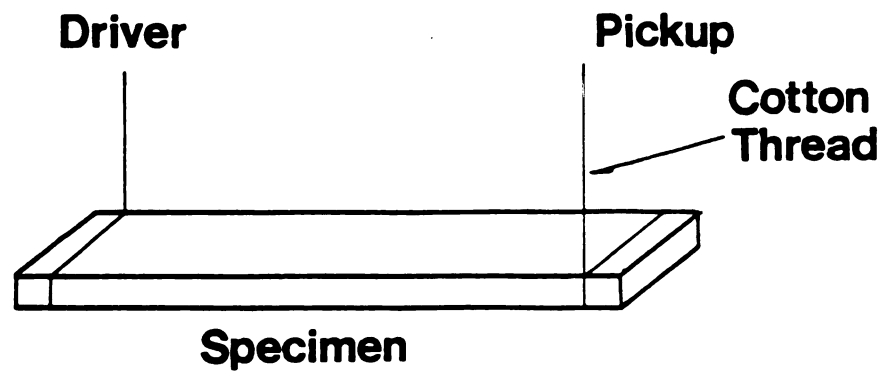


Figure 4. Method of specimen suspension for the sonic resonance measurement

Table 4.

Property
Porosity
H ₂ O Abs.
% of theoretical density
Strength (MOR Bars)
M.O.E.
Hardness
Fracture toughness

* The data supplied in Celsius

** TOKAMA

*** ARCO

Table 4. Mechanical Properties of SiC Whisker Reinforced Alumina Composite Specimens*

Property	HP158**	HP160***	HP171***
Porosity (%)	.72	.26	.43
H ₂ O Abs. (%)	.19	.07	.11
% of theor. -density	98.3	99.3	98.8
Strength (Kpsi) (MOR Bars)	58.79	67.39	51.25
M.O.E. (Mpsi)	56.74	57.11	57.09
Hardness (GPa)	19.81	19.93	20.66
Fracture toughness (MPa m ^{0.5})	6.2	5.9	5.8

* The data in this table except hardness and fracture toughness was supplied by Alcoa. All specimens were hot pressed at 1800 degrees Celsius and include 20 volume percent SiC whiskers.

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piezoelectric driver transducer (model number 62-1 made by Astatic Corp., Conneaut, Ohio). The signal then passed through the specimen, and converted from a mechanical to an electronic signal by the pick up transducer. This signal was amplified, filtered (4302 Dual 24DB/Octave Filter-Amplifier made by Ithaco, Ithaca, N.Y.), and passed through a voltmeter (8050A Digital Multimeter made by Fluke, Everett, WA), and oscilloscope (V-100A 100MHz Oscilloscope made by Hitachi, Japan).

To calculate the elasticity, one must know the type and the mode of the vibration of the specimen [51, 52]. A prismatic bar can be excited in a variety of vibrational modes, including the fundamental flexural frequency. Thus if one identifies a resonant frequency, one must determine which particular mechanical vibrational mode is being excited before a determination of elastic modulus can be made. The vibrational mode can be identified by locating the position of the vibrational nodes and antinodes along the specimen in which a standing-wave vibration forms. The positions of the nodal and antinodal lines on a prismatic bar-shaped specimen vibrating at a mechanical resonance may be determined by mechanically probing the bar [51].

In this study, the nodal and antinodal positions were probed using a sewing needle, balanced at various locations along the length of the resonating bar. When the needle is on a nodal line, the amplitude of resonant frequency changes very little. If the needle is positioned away from a nodal line, then the needle will dampen the

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mechanical vibrations in the specimen reducing the vibrational amplitude measured by the pick-up transducer. Anderson [51] indicates the position of the nodes as a fraction of the length of the specimen (Table 5).

Calibration of the continuous excitation of the present experimental system can be verified using NIST standards. Table 6 shows the results of the verification tests using NIST Standard Reference Material 718, polycrystalline alumina reference bar #1. The results show no significant difference between the resonances and error of less than 0.04 percent with respect to the reference bar.

A "working reference" bar of unshocked alumina was used to check the calibration of the equipments on a weekly basis. Such calibration is advisable since a transducer might be damaged during use. The transducer was replaced whenever the flexural resonance frequency measured by the "working reference" bar was changed.

The resonant frequency was taken as the average of five or more readings. This experiment was done at room temperature in air.

The resonance frequency data were converted into moduli values by using the equations of Pickett [53] as modified by Hasselman [54] (Appendix A).

$$G = P (2L*f/N)^2 * R \quad (2)$$

where N - an integer (unity for the fundamental mode)

f - torsional resonance frequency

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Table 5. Position of Nodes Expressed as a Fraction of the Specimen Length

Mode of Vibration\ Type of Vibration	Flexural	Torsional
Fundamental	0.224 0.776	0.500
First overtone	0.132 0.500 0.868	0.250 0.750

Table 6. Calibration Results of Present Dynamic Modulus Measurement System

	Present System	NIST	error
Fundamental Flexural Freq. (Hz)	2001.30	2001.43	0.0065 %
Fundamental Torsional Freq.(Hz)	11069.01	11073.05	0.036 %

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L - specimen length

P - mass density

R - a shape factor.

For prismatic specimens of rectangular cross section, R can be approximated by an equation as follows;

$$R = \frac{(1 + (b/a)^2) \cdot (1 + (0.0085 N^2 b^2 / L^2))}{[4 - 2.521(a/b)((1 - 1.991)/(\exp(b/a) + 1))] - 0.06(Nb/L)^{1.5} (b/a - 1)^2} \quad (3)$$

where a - thickness of specimen

b - width of specimen.

Young's modulus, E, for a rectangular specimen can be calculated from the following equation

$$E = \frac{0.94642 L^4 f^2 P T}{D^2} \quad (4a)$$

$$= \frac{0.94642 L^4 f^2 m T}{D^3 b} \quad (4b)$$

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where f - fundamental flexural resonant frequency

L - length of the specimen

P - mass density of the material

T - a shape factor

D - cross sectional dimension in the direction of vibration.

b - specimen width

m - specimen mass

The shape factor T for the prismatic specimen is the following function of the specimen dimensions and Poisson's ratio

$$T = 1 + 6.585(1 + 0.0752\nu + 0.8109\nu^2) (D/L)^2 - 0.868(D/L)^4 - \frac{8.34(1 + 0.2023\nu + 2.173\nu^2) (D/L)^4}{1 + 6.338(1 + 0.14081\nu + 1.536\nu^2) (D/L)^2} \quad (5)$$

where ν - Poisson's ratio.

3.3. Room Temperature Internal Friction Measurement

Internal friction (Q^{-1}) may be determined using Forster's method [50]. The free decay method is more effective with the specimen having smaller values of internal friction such as dense ceramics. For the free decay method, two additional items of apparatus (a

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trigger* and a counter**) are required. Figure 3 shows the "internal friction apparatus" for the free decay method. With the specimen driven at its fundamental flexural resonant frequency, the driving signal is turned off by changing the mode from drive to free decay. The number of cycles, N, between a fixed amplitude, A, and a smaller fixed amplitude, B, is counted (figure 5). The measured internal friction, Q_m^{-1} , is given by

$$Q_m^{-1} = \frac{\ln(A/B)}{\pi N} \quad (6)$$

where Q_m^{-1} - measured internal friction

A - amplitude of driving signal

B - preset amplitude

N - number of cycles required for a driving signal to decay from driving amplitude to preset amplitude.

The measured internal friction, Q_m^{-1} , is composed of the internal friction of the specimen, Q_s^{-1} , and the internal friction of the apparatus, Q_a^{-1} . The internal friction of the apparatus includes the effects of the specimen suspension position.

 * Constructed by the Biochemistry Electronics Shop, Michigan State University

** Model 5314A Universal Counter made by Hewlett-Packard

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Figure

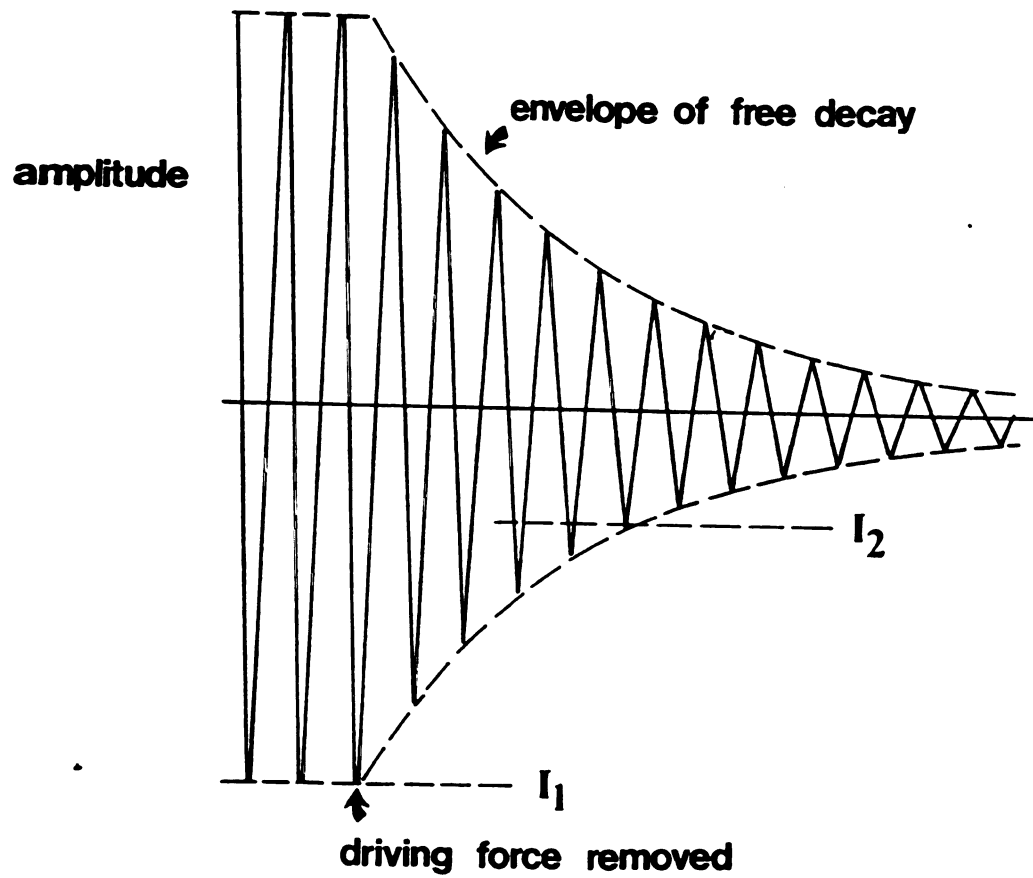


Figure 5. Free decay of natural vibration of an anelastic solid, which is the basis for the internal friction measurements.

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Wachtman and Tefft [55] formulated an equation relating Q_a^{-1} and Q_s^{-1} to the measured internal friction, Q_m^{-1} ,

$$Q_m^{-1} = \frac{(Q_s^{-1} + kQ_a^{-1}(Y/Y')^2)}{(1 + k(Y/Y')^2)} \quad (7)$$

where Q_s^{-1} - the internal friction of the specimen

k - an empirical constant

Q_a^{-1} - the internal friction of the apparatus

Y - the vertical displacement of the suspension point from equilibrium

Y' - the vertical displacement of the end of the specimen.

The Y/Y' term is given by Rayleigh [56] for the transverse fundamental mode of vibration as

$$Y/Y' = (1.018 (\cosh 4.730 x/L + \cos 4.730 x/L) - (\sinh 4.730 x/L + \sin 4.730 x/L)) / 2.036 \quad (8)$$

where L - the length of the specimen

x - the distance to the suspension point from the end of the specimen.

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More than three pairs of suspension positions were marked on each specimen. Figure 4 illustrates the suspension of the specimen. The horizontal distance, x , from the end of the specimen was measured using metric vernier calipers. The specimen was suspended by cotton string and internal friction was measured for the each pair of suspension positions. The measured Q_m^{-1} were put into the non-linear least-squares best fit program to obtain values for Q_a^{-1} , k and Q_s^{-1} (Appendix A). The average of three successive determinations of the internal friction of the specimen was taken as the value of the internal friction of the specimen.

Figure 6 illustrates the typical measured internal friction, Q_m^{-1} , as a function of the suspension position. Each of these determinations of Q_s^{-1} were made in air, neglecting the damping effect of air. The effect of air damping increases the measured internal friction by a small, additive constant [55].

3.4. Thermal Shock Fatigue Tests

3.4.1. SiC Whisker Reinforced Alumina Composites and Polycrystalline Alumina Subjected to Water Quench

For the SiC whisker reinforced alumina composite specimens and polycrystalline alumina specimens, thermal shock fatigue tests were performed using the apparatus shown in Figure 7. The apparatus was designed to perform one thermal shock per hour. During a typical thermal shock cycle, the specimen was suspended in the middle of the

Q_m^{-1} ($\times 10^{-5}$)

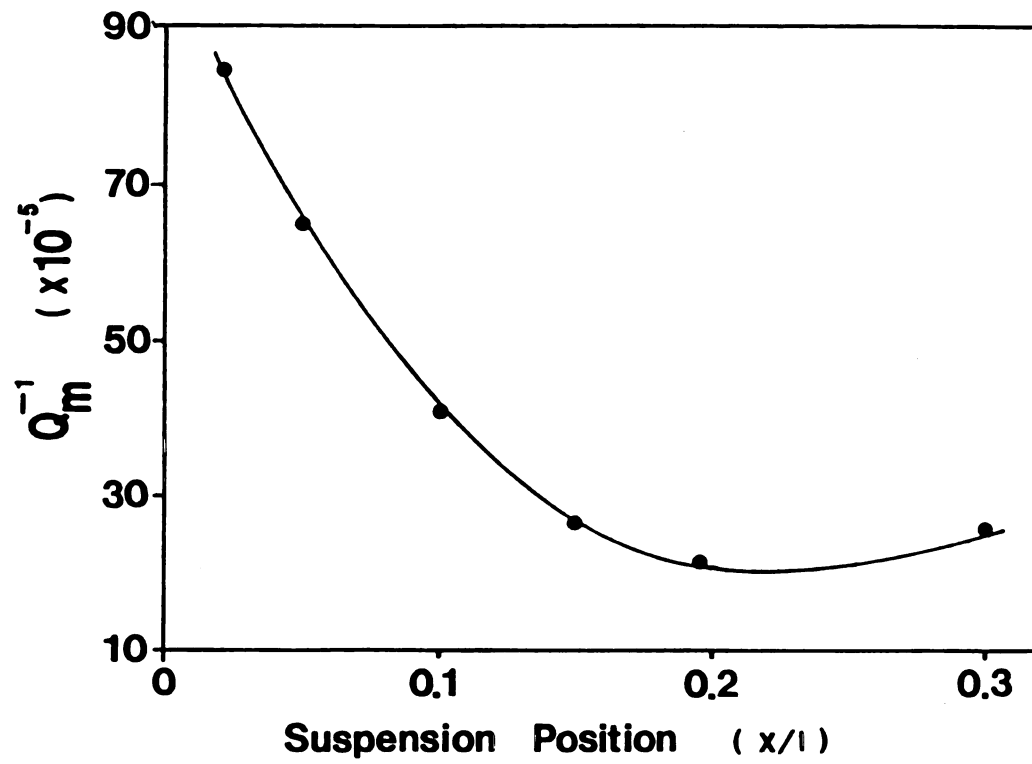


Figure 6. Typical measured internal friction as a function of suspension position.

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Figure

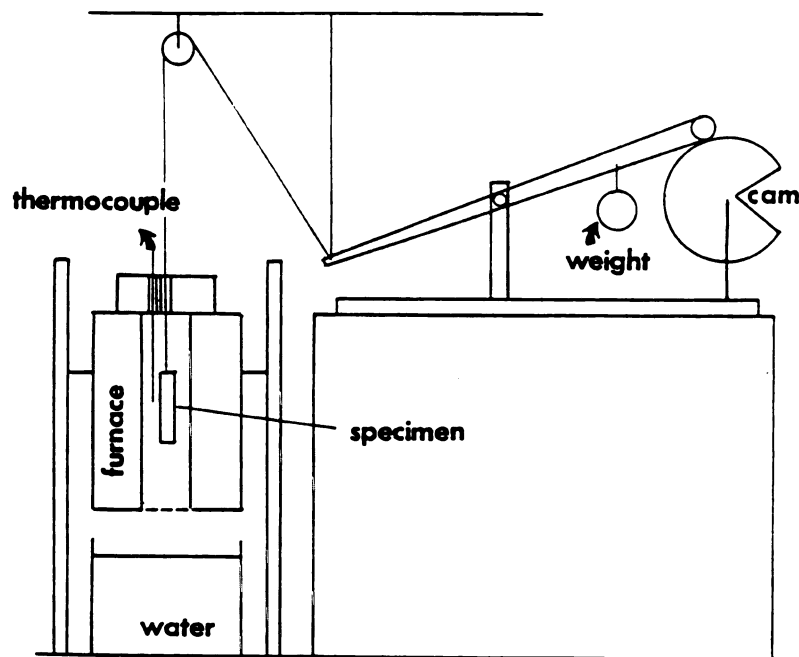


Figure 7. Schematic diagram of the thermal shock test apparatus including furnace and quenching bath.

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furnace for approximately 45 minutes. After thermal shock, the specimen remained in the water bath for 15 minutes.

The furnace used in this study was an electric resistance furnace. The heated chamber of the furnace was 13.5 inches long and 3 inches in diameter. The power input to the furnace was controlled by a variable transformer (2PF136 type Powerstat, The Superior Electric Co., Bristol, Conn). The variable transformer was adjusted to a predetermined power level and the furnace was allowed to approach thermal equilibrium before operating the thermal fatigue equipment. Up to two and one-half hours was required for thermal equilibrium to be reached. Once thermal equilibrium was reached, the change in thermocouple output was ± 1 millivolt during the entire thermal shock which corresponds to an uncertainty of ± 2 degrees Celsius error. Temperature measurement was made with a J type thermocouple with its hot junction located to the side and near the middle of the specimen.

The plastic container for the water bath was rectangular with dimensions 24cm x 50cm x 75cm. Approximately 8 liters of distilled water were used for the water bath. The temperature of the water bath was measured by a mercury-in-glass thermometer with a temperature range of -10 degrees Celsius to 260 degrees Celsius. The thermometer markings were at 1 degree Celsius intervals, so temperature measurements were accurate to within ± 0.1 degree Celsius.

After attaining the preselected temperature in the middle of the furnace, the thermal shock fatigue tests were performed. Rapid thermal quenching was done one time per hour. The typical time interval for

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the specimen free fall between the furnace and the water bath was less than one second. All specimens were removed from the water bath after having the desired number of thermal shocks. Specimens were dried with a paper towel and kept 10 hours at room temperature before performing the elasticity and internal friction measurements.

The thermal shock damage was examined by measuring the changes in the elastic modulus and internal friction.

3.4.2. Polycrystalline Alumina Subjected to Silicone Oil Quench and Liquid Nitrogen Quench

For the polycrystalline alumina specimens, thermal shock fatigue tests into silicone oil bath and liquid nitrogen bath were also performed using a horizontal furnace designed and assembled at Michigan State University. Details of the construction of the furnace were described elsewhere [6].

The liquid nitrogen was contained in a dewar with inner diameter 9 cm and depth 20 cm. The silicone oil was contained in a 7000 cc beaker.

Specimens were placed horizontally on a fused silica boat and heated in a horizontal electric furnace for 20 minute at a preselected temperature, after which they were quenched into the quenching bath (silicone oil or liquid nitrogen) along the length direction. At a preselected number of thermal shock cycles, the liquid nitrogen shocked specimen was placed in a desicator for one hour prior to elasticity measurements. The silicone oil quenched specimen was

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cleaned by a paper towel and held in laboratory air about 30 minutes before performing the thermal shock damage measurement.

3.5. Temperature Dependent Material Properties Measurements

3.5.1. Elevated Temperature Elastic Moduli and Internal Friction

Elevated temperature data were obtained by suspending the specimen horizontally by platinum wire (Ted Pella, Inc., Tustin, CA) in the hot zone of a electric resistance furnace. The heated chamber of the vertical furnace was 14 inches long and 4 inches in diameter. The temperature was controlled by a digital temperature controller (CN5000 Omega, Engineering Inc., Stamford, CT). For SiC whisker reinforced alumina composites and polycrystalline alumina, the fundamental flexural resonance frequency was determined over the range from room temperature to 900 °C and 600 °C, respectively, with the temperature being raised by 50 °C increments.

The resonance frequency f_t at some temperature ΔT above room temperature was converted to relative elastic modulus E_t/E_0 using the relationship [51]

$$\frac{E_t}{E_0} = \frac{f_t^2}{f_0^2 (1 + \alpha_{th} \Delta T)} \quad (9)$$

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where E_t , E_o = high temperature and room temperature elastic modulus

f_o = room temperature resonance frequency

α_{th} = linear thermal expansion coefficient.

The true internal friction value of the specimen was not measured at elevated temperature. Only one suspension point was used for elevated temperature internal friction measurements. Thus, these values were corrected to the specimen room temperature corrected internal friction value, Q_s^{-1} , by the equation 7

$$Q_{st}^{-1} = Q_s^{-1} \frac{Q_{mt}^{-1}}{Q_o^{-1}} \quad (10)$$

where Q_{st}^{-1} = the measured specimen corrected internal friction at elevated temperature

Q_{mt}^{-1} = the measured internal friction at elevated temperature

Q_o^{-1} = the measured internal friction with platinum wires in air at room temperature.

The assumption inherent in this equation is that the observed changes in internal friction as a function of temperature are due to changes in the internal friction of the specimen.

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3.5.2. Measurement of Thermal Expansion

Thermal expansion is required for the calculation of maximum surface thermal stress during thermal shock. Coefficients of thermal expansion for SiC whisker reinforced alumina composites between room temperature and 500 degrees C were obtained by using 943 Thermomechanical Analyser (TMA) (Du Pont 9900, Du Pont Company, Wilmington, DE) at Composite Center, Michigan State University. The dimensions of sample were 3 mm x 3 mm x 4 mm. To measure the thermal expansion the expansion mode probe was used. The standard weight on the TMA weight tray was 5 grams. The probe and tray themselves were calibrated by using an alumina standard in air. The heating rate for thermal expansion measurements was 10 degrees C per minute from room temperature to a maximum temperature of 500 degrees C.

The temperature dependence of the thermal expansion coefficient for solids is typically approximated by a second order polynomial in temperature. The thermal expansion data on heating between 20 degrees C and 500 degrees C (figures 8a and 8b) for SiC whisker reinforced alumina composites were fitted via least squares best fit to a second order polynomial. The resulting equation was

$$\alpha_{th} = (5.13 + 5.7 \times 10^{-3} T + 4.5 \times 10^{-6} T^2) \times 10^{-6} \quad (1/C) \quad (11)$$

The average coefficient of thermal expansion was about $7.11 \times 10^{-6}/C$ between 20 degrees C and 500 degrees C. This value was similar to the

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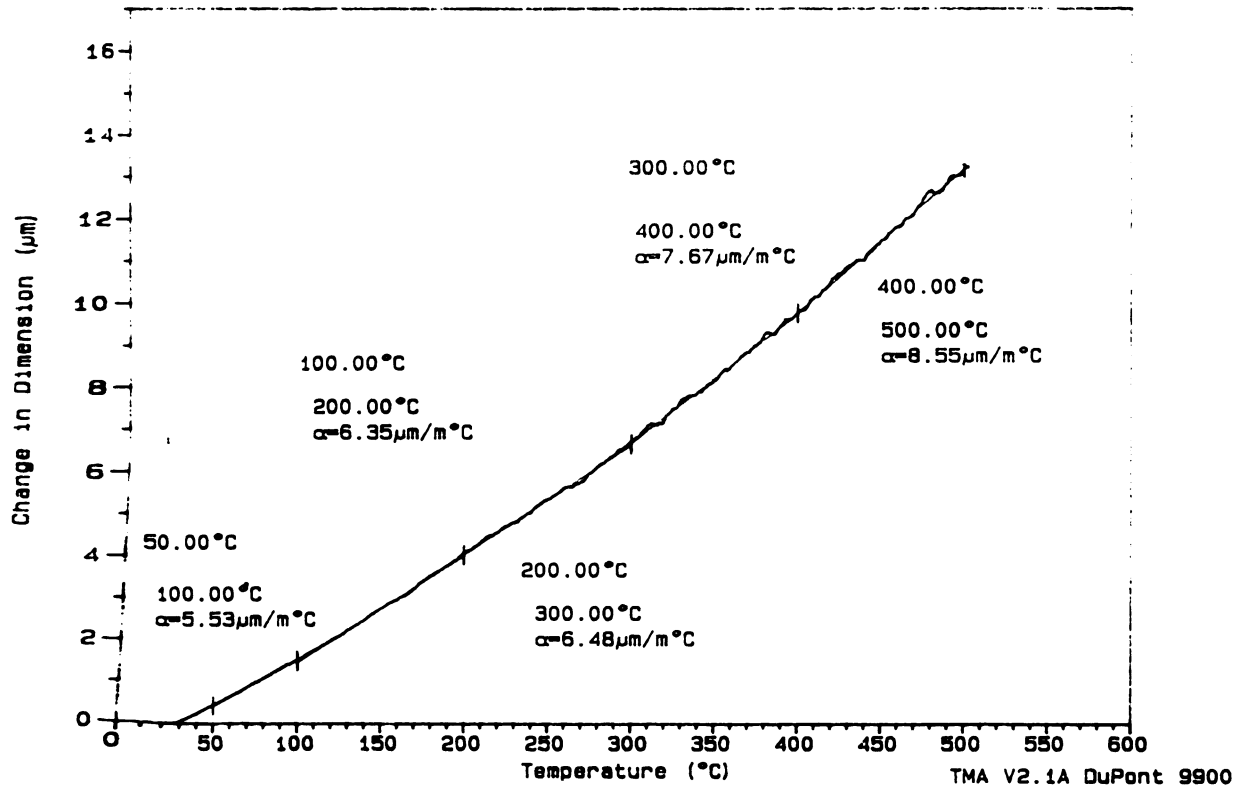


Figure 8a. TMA results of SiC whisker reinforced alumina composite.

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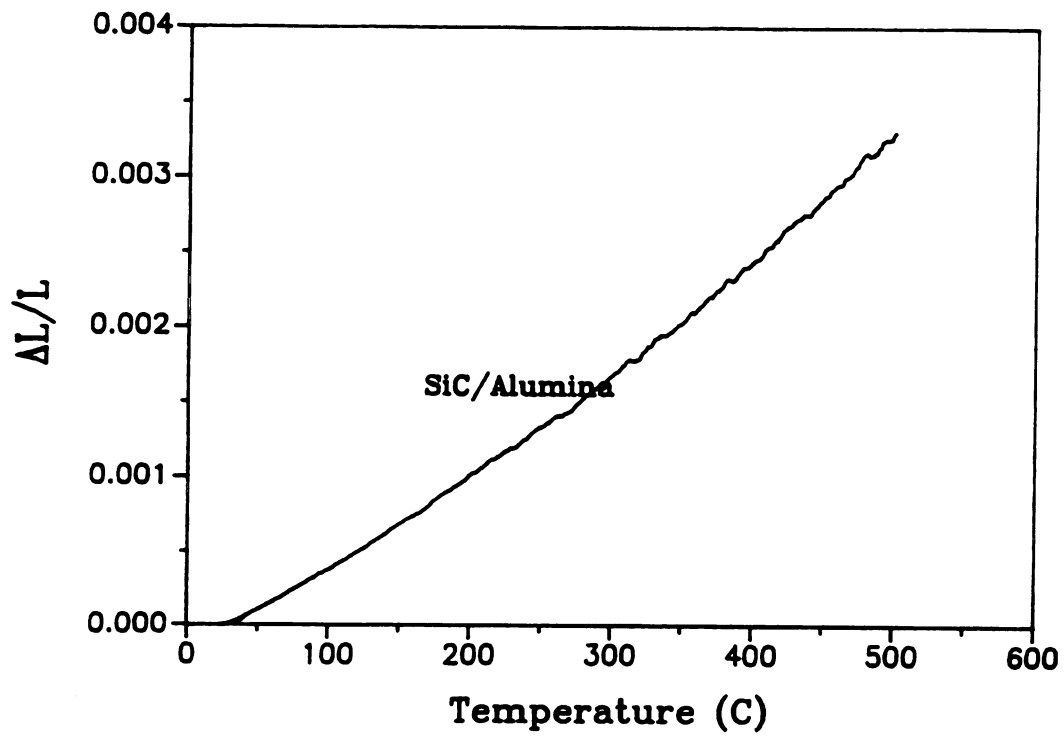


Figure 8b. Thermal expansion of SiC whisker reinforced alumina as a function of temperature.

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For polycrystalline alumina, thermal expansion data are available from literature [58].

3.6..Hardness and Fracture Toughness measurement

The hardness and fracture toughness of the polycrystalline alumina and SiC whisker reinforced alumina composites were determined by the Vickers indentation method.

The indentation was produced with 4.9 to 196 N loads at room temperature on polished sample surfaces, using a Vickers diamond indenter (Semimacro Hardness Tester Series No. 5987, Buehler Ltd., Lake Bluff, IL). A loading time of 10 seconds was used for all indentations with a loading speed of $70 \mu\text{m}/\text{sec}$. The hardness was determined by dividing the load by the contact area using [59]

$$H = \frac{0.47P}{a^2} \quad (12)$$

where H = hardness

P = the peak indentation load

$2a$ = the pyramid diagonal of the indentation impression.

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Fracture toughness was determined from an equation described by Lawn et al. [60]

$$K_C = A(E/H)^{1/2} (P/C^{3/2}) \quad (13)$$

where K_C = fracture toughness

E = Young's modulus

C = radial crack length measured from the center of the indentation

A = a calibration constant, 0.017 ± 0.001 [60].

3.7. Transient Surface Temperature Measurements

To evaluate the heat transfer coefficient of polycrystalline alumina for water quench, silicone oil quench and liquid nitrogen quench, the surface temperature changes were measured by using a thin foil "Cement-on" K type thermocouple (CO2-K, Omega Engineering, Stamford, CT) (figure 9). This thermocouple can withstand temperatures up to 600 degrees C. The sensors are made from 0.0127 mm foil with 0.0508 mm thick leads. The response time of this thin foil thermocouple is 2 to 5 milliseconds experimentally by quenching the thermocouple itself into a room temperature water bath (figure 10). A thermocouple was bonded with Omega CC high temperature cement (Omega Engineering, Stamford, CT) on the surface of the specimen (6.98 cm x 1.12 cm x 0.1 cm). Using a small glass rod, the cement powder (3

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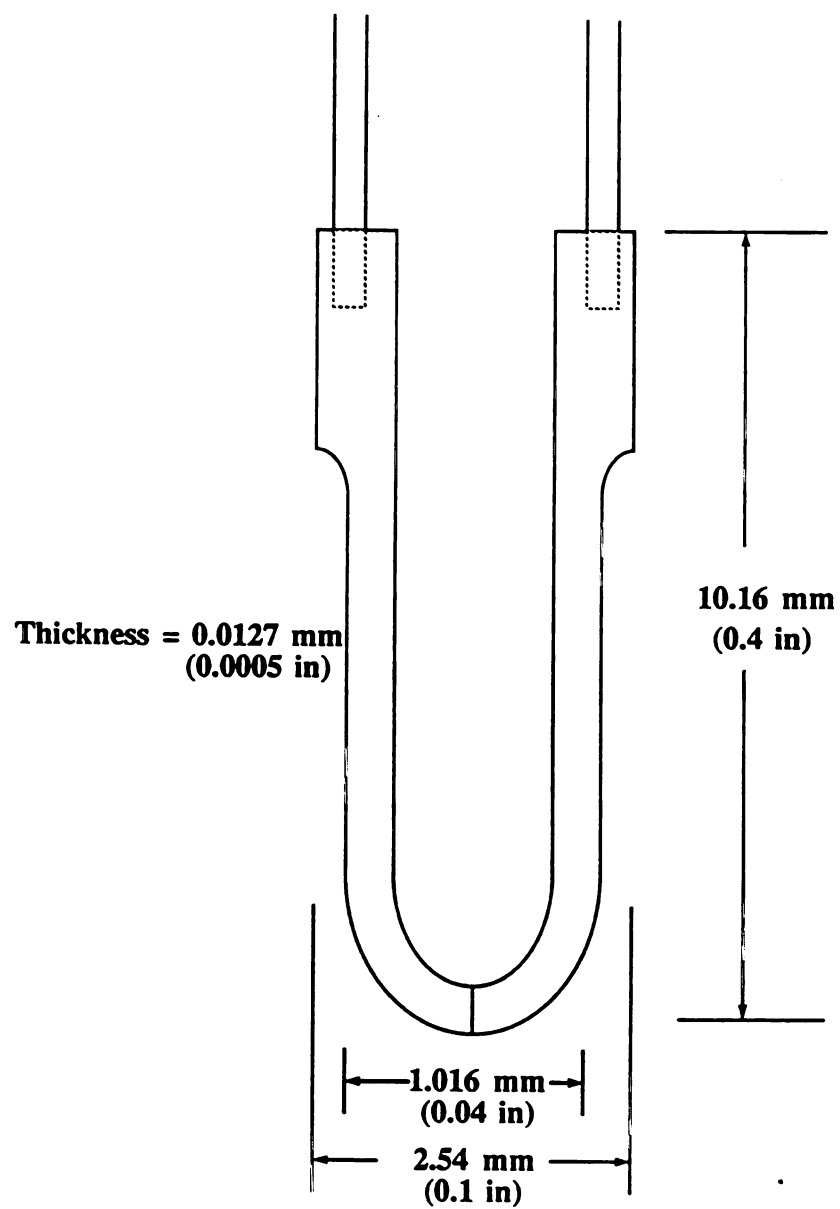


Figure 9. Schematic of a thin film "Cement-on" K type thermocouple.

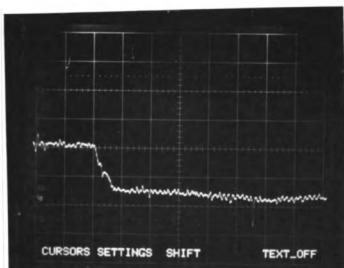


Figure 10. The experimentally obtained thermocouple voltage versus cooling time for quenching the thermocouple itself into a room temperature water bath, showing the thermal response of the thermocouple without perturbations from an attached substrate. Each division on x axis represents 3.125 msec, each division on y axis represents 5 mV.

parts by weight) was mixed with liquid binder (1 part by weight) on a slide glass for about 30 seconds.

When using these cements, it is important that the surfaces to be bonded are clean. The specimen surface was cleaned using acetone. A stream of acetone was applied to the specimen using a plastic "squeeze bottle". The acetone was then allowed to evaporate.

The cement mixture was applied by a small glass rod to a cleaned specimen surface. Immediately after the cement mixture was applied to the specimen, the tip of thermocouple was applied by pressing it with tweezers. Pressure was applied via the tweezers for about 3 to 5 minutes. Following that, a microscopic glass was placed over the thermocouple and a mass of about 200 grams was placed on the microscopic glass to apply pressure to the cemented thermocouple assembly. (In this procedure, the cement was sufficiently set by the time that the microscope slide was applied that it did not stick to the cement). The cemented thermocouple (figure 11) was allowed to dry for about 20 to 30 minutes, after which the slide glass was removed and a coating of glue not more than 0.5 mm thick was applied to the thermocouple leads in order to fix the thermocouple leads onto the specimen. After applying the cement to the thermocouple leads and waiting 3 to 5 minutes, the glass slide plus the mass was again placed on the thermocouple assembly. The glass slide and mass remained on top of the thermocouple assembly during the entire curing time of the cement (18 to 24 hours, as recommended by Omega, the manufacturer). After curing, the K type thermocouple lead wire was bonded to the lead

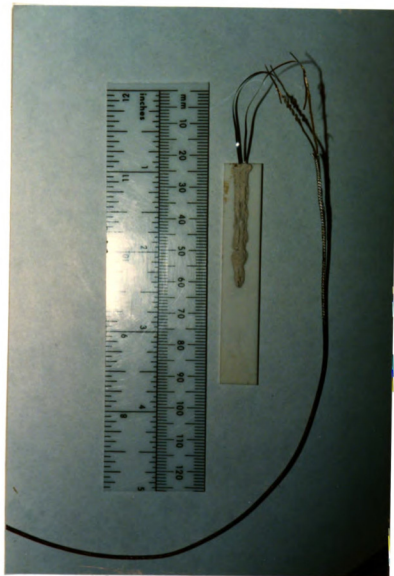


Figure 11. Photograph of a thin foil thermocouple bonded to a prismatic polycrystalline alumina bar specimen.

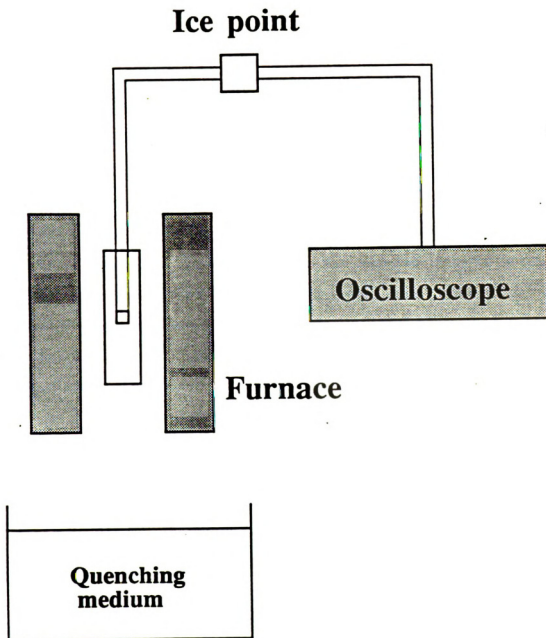


Figure 12. Schematic of experimental setup for surface heat transfer coefficient measurement

ribbon by spot welding (Rocky Mountain/Orthodontics 660)*. The other side of thermocouple lead wire was connected to an ice point reference junction (Model MCJ, Omega Engineering). Leads from the ice point were connected to a digital storage oscilloscope (Model PM 3365, Philips), which recorded the voltage signal produced by thermocouple during a quenching process (figure 12). The voltage signal was recorded by using the Tectronix C-5c oscilloscope camera.

The thermocouple attached specimen was quenched into the silicone oil bath or liquid nitrogen bath about 1 second after the specimen was removed from the horizontal furnace. The other specimen was immersed into water bath immediately from the vertical furnace. Three different transient temperature measurements for water quench, silicone oil quench and liquid nitrogen quench were performed (figures 13 a-i).

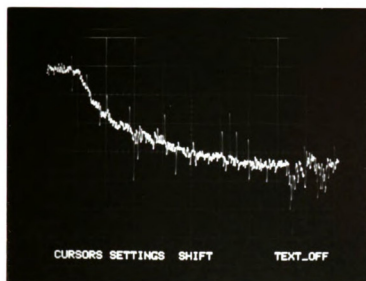
3.8. Estimation of Heat Transfer Coefficient

From the transient surface temperature measurements, the surface heat transfer coefficient was estimated using a parameter estimation method [61, 62]. If we assume the specimen is a thermally lumped⁺

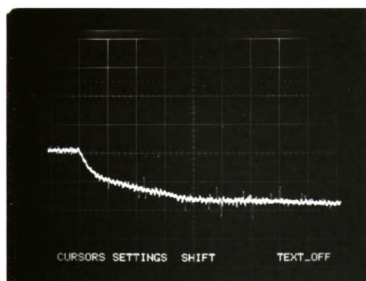
* Department of Biochemistry, Michigan State University.

+ Thermally lumped bodies are bodies in which the thermal conductivity is very large or the characteristic length scale (volume/surface area) is small, such that the temperature of the solid is spatially uniform at any instant during the transient process. In this study, the characteristic length scale of the alumina specimen is small.

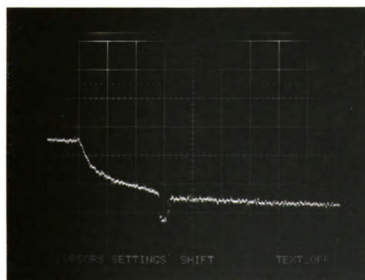
Figure 13. The experimentally obtained thermocouple voltage versus cooling time for alumina quenched into water at ΔT - (a) 250 C, (b) 300 C and (c) 350 C; each division on x axis represents 6.25 msec, each division on y axis represents 5 mV.



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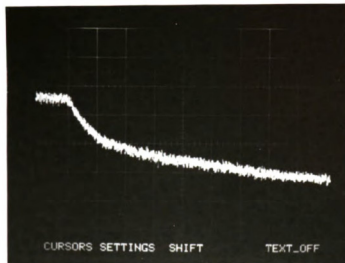
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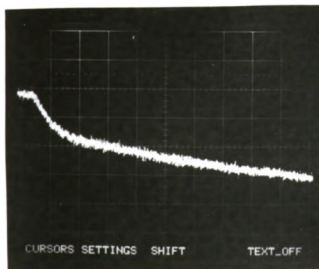
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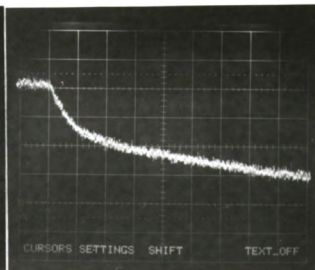
Figure 10.



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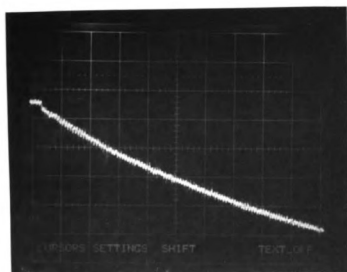


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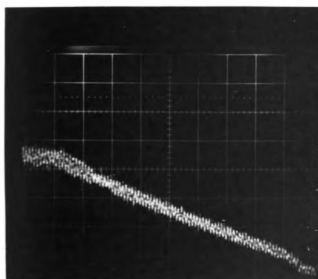
Figure 13. The experimentally obtained thermocouple voltage versus cooling time for alumina quenched into oil at $\Delta T =$ (d) 480 C, (e) 510 C and (f) 560 C; each division on x axis represents 200 msec, each division on y axis represents 5 mV.



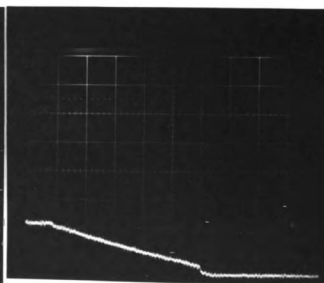
Figure



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Figure 13. The experimentally obtained thermocouple voltage versus cooling time for alumina quenched into **liquid nitrogen** at $\Delta T =$ (g) 696 C, (h) 515 C and (i) 305 C; each division on x axis represents 2 sec, each division on y axis represents 5 mV.

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body then temperature is a function of time only. The differential equation for a lumped body which is suddenly immersed into a quenching fluid at a temperature T_∞ is [61, 62]

$$hA(T_\infty - T) = \rho c_p V \cdot dT/dt \quad (14)$$

where, h = heat transfer coefficient

ρ = density of the specimen

c_p = specific heat of the specimen

t = time

V, A = volume or heated surface area of the specimen

T = true specimen temperature.

Several estimation procedures are available for lumped bodies. Among them [61, 62], we used a regression method. Polynomials of various degrees can be used. The polynomial form in the regression of the temperature-time data is

$$T = \beta_1 + \beta_2(t/\Delta t) + \dots + \beta_p(t/\Delta t)^{p-1} + \epsilon \quad (15a)$$

$$\hat{T} = \beta_1 + \beta_2(t/\Delta t) + \dots + \beta_p(t/\Delta t)^{p-1} \quad (15b)$$

where $\hat{T}$ = estimated temperature

Δt = time interval between readings of the thermocouple voltage

ϵ = residue of the polynomial.

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In this study, fourth order polynomials ($p = 5$) were used to obtain $\hat{T}$ from the time-temperature data. The derivative of estimated temperature, $d\hat{T}/dt$, was obtained from equation 15, such that

$$d\hat{T}/dt = (\beta_2 + 2\beta_3(t/\Delta t) + 3\beta_4(t/\Delta t)^2 + 4\beta_5(t/\Delta t)^3) / \Delta t \quad (15)$$

Using equations 14, 15, and 16 we obtain the estimated heat transfer coefficient $\hat{h}$ [61, 62], such as

$$\hat{h} = \frac{\rho c_p V}{A(T_\infty - \hat{T})} \cdot \frac{d\hat{T}}{dt} \quad (17)$$

In addition to $\hat{T}$ and $d\hat{T}/dt$, the temperature dependence of the other parameters in equation 17 should be considered. For a given material, an expression for $c_p(T)$, the specific heat as a function of temperature may be obtained by regression on available c_p versus temperature data. From the definition of mass density, the product ρV is the specimen mass, which is temperature independent. The change in the surface area, A , with temperature will be proportional to 2α , where α is the linear thermal expansion coefficient. Since A is a rather weak function of temperature compared to the other temperature dependent terms in equation 17, A was considered to be constant in this study.

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4. RESULTS AND DISCUSSION

4.1. Cyclic Thermal Shock of SiC Whisker Reinforced Alumina Composites

4.1.1. Elastic Modulus and Internal Friction Measurements on Unshocked Specimens

The room temperature fundamental flexural resonant frequency, Young's modulus and internal friction of the three as-received SiC whisker reinforced alumina composite specimens and a high temperature modulus measured specimen are given in table 7.

Figure 14 illustrates the Young's modulus as a function of temperature for SiC whisker reinforced alumina composite HP171. The modulus decreased continuously with increasing temperature, which is typical of polycrystalline ceramics [63-66].

The modulus changed linearly up to 900 °C, which was the maximum test temperature for the present study. The modulus versus temperature data was fit via least-squares to a linear function of temperature. The modulus-temperature data was fit (via a least-squares procedure) to the empirical equation,

$$E = E_0 - cT \quad (18)$$

where E_0 - the zero temperature Young's modulus

T - the temperature in °C

and c - the modulus-temperature slope.

Table 7. Fundamental Flexural Resonance Frequency, Room Temperature Modulus and Internal Friction of SiC Whisker reinforced Alumina Composite Specimens

Specimen	Resonant Frequency (Hz)	E (Gpa)	$Q_s^{-1} (10^{-8})$
HP158	6387.67	404.56	13.2
HP160	6406.17	404.84	14.2
HP171	6431.00	406.14	18.6
HP160-RA	6392.50	405.45	14.8

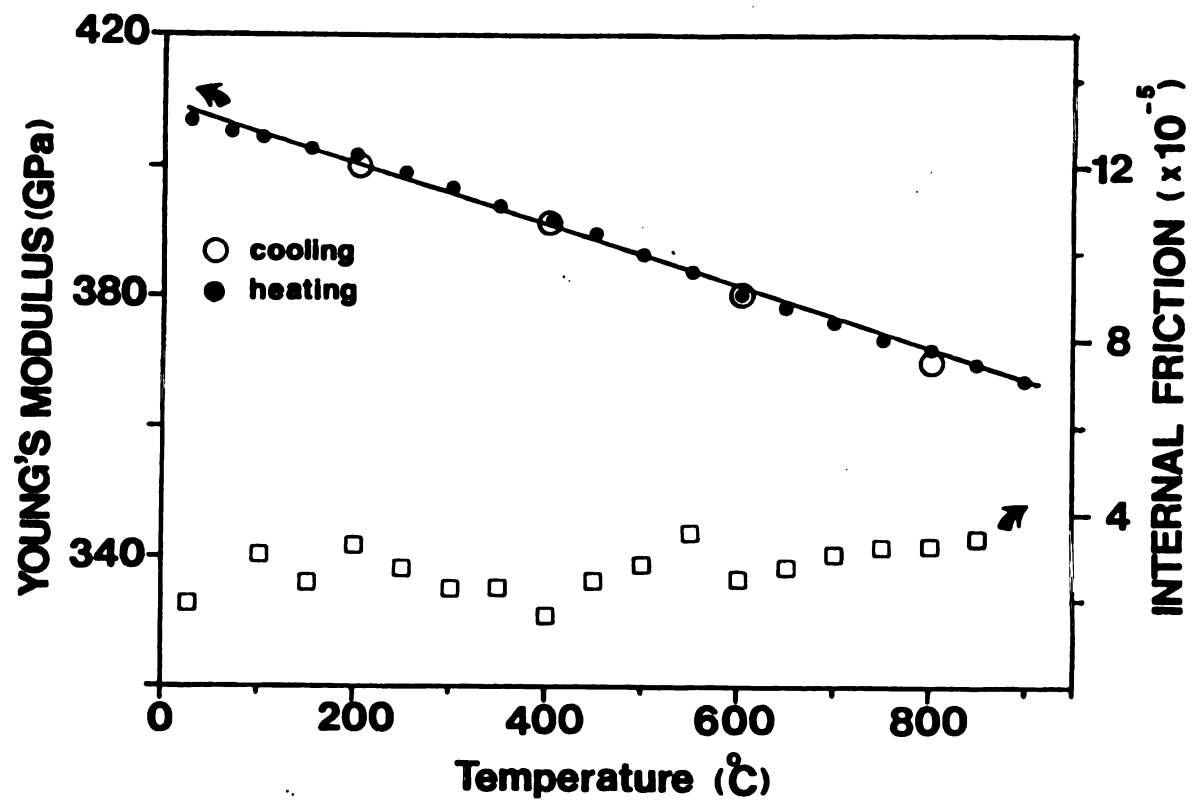


Figure 14. Young's modulus and internal friction of the alumina/SiC whisker composite specimen HP171 as a function of temperature prior to thermal shock damage.

The value of c obtained from the least-square procedure was $0.047 \text{ GPa}/^{\circ}\text{C}$. The correlation coefficient for the least-squares fit of the 19 Young's modulus-temperature data pairs was 0.998. The modulus decreased linearly at a rate of $1.1 \text{ percent}/100^{\circ}\text{C}$, which agrees favorably with data for other polycrystalline ceramics [63-65]. In particular, a Young's modulus versus temperature slope of $1.2 \text{ percent}/100^{\circ}\text{C}$ has been determined for a polycrystalline monolithic alumina [63].

The internal friction was measured as a function of temperature with the modulus measurements (figure 14). The internal friction varied by approximately ± 30 percent about a mean value of 2.7×10^{-5} . Before heating the specimen HP171 during the elevated temperature measurements, the room temperature internal friction was 18.6×10^{-5} . After the elevated temperature measurements were completed the room temperature internal friction of specimen HP171 was 11.1×10^{-5} .

Small changes in the internal friction upon annealing could result from the relaxation of residual stresses in the specimen, or in part from diffusive crack healing of cracks induced during specimen preparation. The anneal temperature of 900°C (1173°K) is about one-half the melting temperature of the alumina matrix, thus diffusion related stress relaxation and crack healing should be possible during the anneal [63].

4.1.2. Functional Form of Elasticity and Internal Friction as a Function of the Cumulative Number of Cycles

Figure 15 and figure 16 show the room temperature Young's modulus and internal friction, respectively, of SiC whisker reinforced alumina plotted as a function of the cumulative number of thermal shock cycles. As expected, the internal friction increases while the Young's modulus decreases [5, 6, 8-10, 66, 67].

The internal friction of the SiC whisker reinforced alumina composite specimen HP160 increased by up to 120 percent while the Young's modulus decreased by a maximum of about 0.07 percent (Table 8). Apparently, internal friction measurements are more sensitive to the microcrack damage than are modulus measurements [5, 6, 8-10].

In the present study, no discontinuous change in elastic modulus or internal friction was observed as a function of increasing thermal shock damage (figures 15 and 16). Bradt and co-workers have shown by both a theoretical (statistical) analysis and by experimental work that thermal shock induced strength degradation does not necessarily shows discontinuities with increasing thermal shock damage levels [2, 3].* According to Bradt et al. if a thermal shock study includes a

* Bradt et al. thermally shocked 4 mm diameter cylindrical specimens of polycrystalline alumina, glass-bonded mullite, and borosilicate glass [2, 3]. In order to obtain statistically significant results, sets of 100 nominally identical specimens for each material were shocked at each ΔT condition. Of the 100 specimens shocked at each ΔT , 50 were fractured in a water bath and 50 were fractured in inert conditions (see Appendix B).

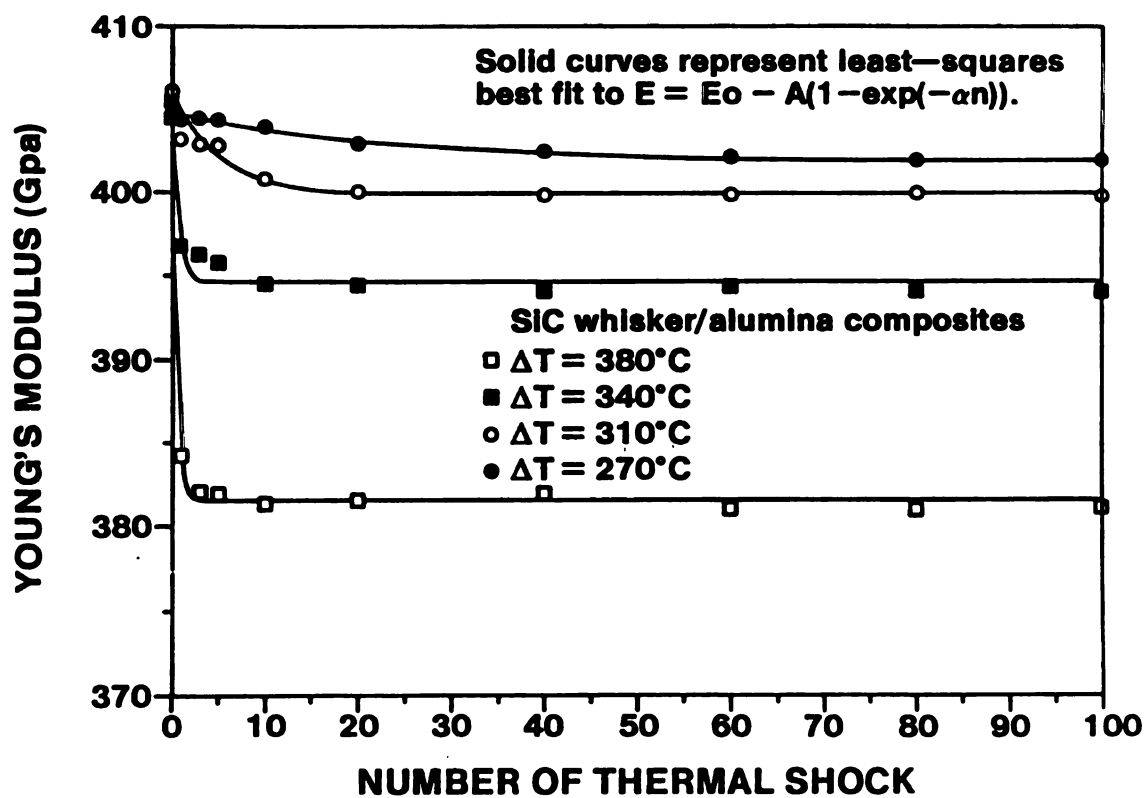


Figure 15. Young's modulus of the alumina/SiC whisker composites HP160 ($\Delta T = 270$ degrees Celcius), HP171 ($\Delta T = 310$ degrees C), HP160-RA ($\Delta T = 340$ degrees C) and HP158 ($\Delta T = 380$ degrees C) as a function of the cumulative number of thermal shock cycles.

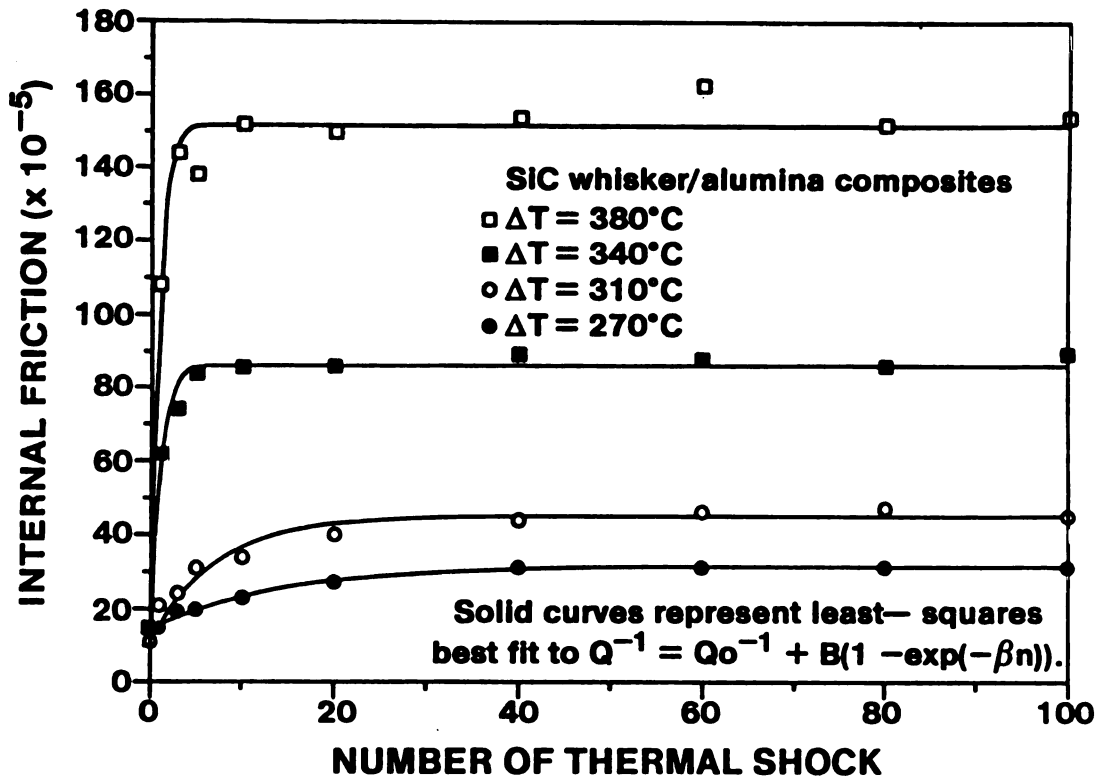


Figure 16. Internal friction of the alumina/SiC whisker composites HP160 ($\Delta T = 270$ degrees C), HP171 ($\Delta T = 310$ degrees C), HP160-RA ($\Delta T = 340$ degrees C) and HP158 ($\Delta T = 380$ degrees C) as a function of the cumulative number of thermal shock cycles.

Table 8. Relative Changes in Young's Modulus and Internal Friction for the Thermal Shocked Specimen (SiC Whisker Reinforced Alumina Composites)

Specimen	ΔT (°C)	$\frac{\Delta E_{\max}}{E_o} \times 100$	$\frac{\Delta Q_{\max}^{-1}}{Q_o^{-1}} \times 100$
HP160	270	0.07	120
HP171	310	1.72	318
HP160-RA	340	2.85	504
HP158	380	5.83	1049

sufficient number of nominally identical specimens to characterize the crack distribution statistics, then the "typical" discontinuous strength drop at a critical quench temperature ΔT_c (figure 17a) is replaced by smooth decreases in strength over some temperature interval (from ΔT_1 to ΔT_2 in figure 17b) [2, 3]. Bradt et al. determined the retained strength for groups of 50 glass and ceramic specimens at each of several thermal shock damage levels to ensure statistical significance for the retained strength testing. (Since brittle fracture is inherently a stochastic process, a substantial number of specimens must be tested to overcome the inherent scatter in fracture data.)

In the present study, the elastic modulus and internal friction measurements are sensitive to the entire spectrum of cracks in each individual specimen through the damage parameter ϵ , equation 22. Instead of using a large number of specimens to clarify the crack statistics for a stochastic process like fracture, the crack statistics are reflected by measurements of internal friction and modulus for individual specimens in the present study.

4.1.3. Saturation Behavior of Thermal Shock Damage

Empirical equations for Young's modulus, E , and for internal friction, Q^{-1} , as a function of the number of thermal shock cycles can be written as

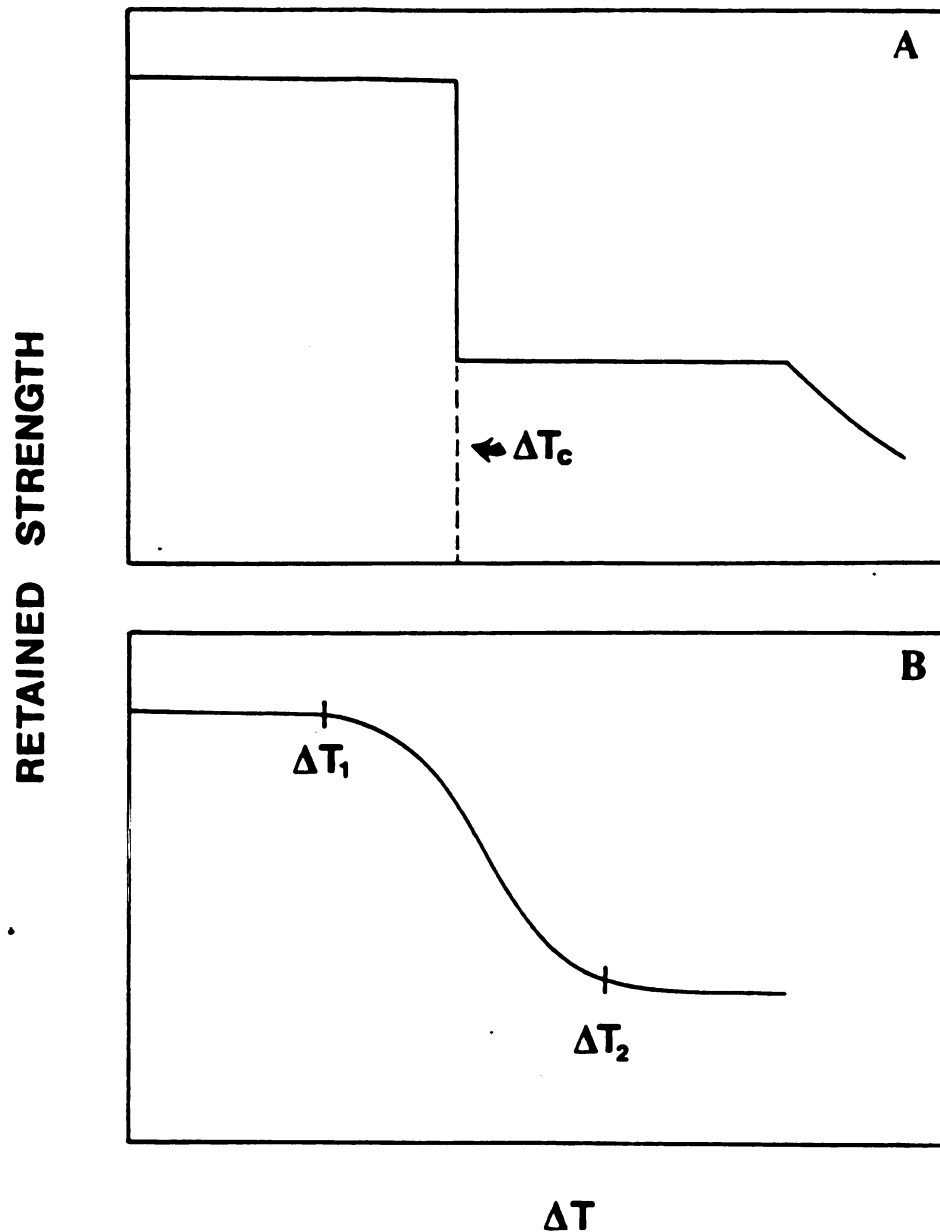


Figure 17. Schematic of retained strength single-quench test protocol (a) after Kingery [88] and (b) after Bradt et al. [2, 3].

$$E = E_0 - A(1 - \exp(-\alpha n)) \quad (19)$$

$$Q^{-1} = Q_0^{-1} + B(1 - \exp(-\beta n)) \quad (20)$$

where E - the effective Young's modulus in GPa

E_0 - Young's modulus non-microcracked material

Q^{-1} - the effective internal friction

Q_0^{-1} - internal friction for the non-microcracked material

n - cumulative number of thermal shock cycles

A, B - damage saturation parameters determined by non-linear regression

α, β - rate constants.

The results of the non-linear regression analysis are shown in table 9. For a fixed value of ΔT , the constant A in equation 19 represents a "damage saturation" or steady state level of damage for a large number of thermal shock cycles, n . Likewise, the constant B in equation 20 represents the large n value (or asymptotic value) of the internal friction. The thermal fatigue damage, as measured by both the internal friction and Young's modulus shows a saturation level, and this saturation damage level increases with increasing ΔT .

The constant " α " in equation 19 is a "rate of decrement" of the Young's modulus as a function of thermal fatigue treatment. At a cumulative number of cycles given by $n_{oe} = 1/\alpha$, the relative decrement in Young's modulus is $(1 - e^{-1})$ (Table 10). Similarly, $n_{oi} = 1/\beta$ is the

Table 9. For the SiC Whisker Reinforced Alumina Composites, Results of the Nonlinear Regression Analysis for the Damage Saturation Parameters A, and B and the Rate Constants α , and β (see equations, 19 and 20). The Unshocked Values of Internal Friction Q_0 , and Young's Modulus, E_0 Shear Modulus, G_0 , are included for reference.

Property	HP160	HP171	HP160-RA	HP158
ΔT (°C)	270	310	340	380
Q_0^{-1} ($\times 10^{-5}$)	14.18	11.10	14.80	13.40
E_0 (Gpa)	404.84	406.10	405.45	404.60
A (Gpa)	3.00	6.21	10.87	23.17
B ($\times 10^{-5}$)	20.38	33.65	71.42	138.31
α	0.042	0.220	1.51	2.11
β	0.061	0.150	0.93	1.11
cor. coeff. (A, α)	0.993	0.956	0.989	0.991
cor. coeff. (B, β)	0.996	0.978	0.976	0.991

Table 10. Thermal Fatigue Constants for the SiC Whisker Reinforced Alumina Composites

Property	HP160	HP171	HP160-RA	HP158
ΔT ($^{\circ}\text{C}$)	270	310	340	380
$(A-E_o)/E_o$	0.9924	0.9847	0.9731	0.9427
$(B-Q_o^{-1})/Q_o^{-1}$	0.437	2.072	3.826	9.33
n_{oe}	24.3	4.83	0.662	0.50
n_{oi}	16.3	7.25	1.08	0.89

fatigue constant for the thermal shock induced changes in internal friction. The values of both n_{oe} and n_{oi} decrease rapidly as ΔT increases (Table 10). Thus, N_{sat} , the number of thermal shock cycles required to reach the saturation damage level is a sensitive function of ΔT .*

4.1.4. Comparison of Saturation Behavior of Thermal Shock Damage in the Various Studies

An empirical equation similar to equations 19 and 20 was provided by Herakovich and Hyer [69] in their cyclic thermal loading study (121 degrees C to -156 degrees C) of fiber reinforced graphite-epoxy composites. Two types of specimen geometry were used, a flat laminate and a tube shape. The specimens were placed on a mechanically driven slide tray which alternated between two air chambers maintained at the maximum and minimum test temperatures. Each thermal cycle took approximately 15 minutes. Herakovich and Hyer [69] counted the cumulative number of cracks developed during thermal cycling by using an optical microscope. An empirical function expressing the average crack density as a function of cumulative number of thermal cycles is [69]

* Equations 19 and 20 have the same mathematical form as capacitor charging and discharging equations for a resistive-capacitive (RC) electrical circuit. The values of n_{oe} and n_{oi} are analogous to the RC time constants, in that after about 3 time constants have "elapsed", the process is essentially at steady state.

$$\rho = C_1(1 - \exp(-\lambda N)) \quad (21)$$

where ρ = crack density (number of cracks per unit specimen length)

C_1 = positive empirical constant called "saturation value"

(C_1 has the same units as ρ in equation 21.)

λ = positive empirical constant called "rate constant"

N = number of thermal loading cycles.

A least squares best fit of the experimental data (9 to 14 data points) determined the values of C_1 , 0.62-0.81 (cracks/mm) and the values of λ , 0.00019 - 0.098 [69] (Table 11). The empirical equations 19 and 20 for accumulative damage is not exactly same as that of Herakovich and Hyer [69] but both equations show saturation behavior.

Damage saturation behavior has been observed during the mechanical fatigue of polymer composites. Liu and Nairn [75] carried out fatigue experiments on $[0_2/90_4]_s$ Fiberite 934 epoxy/T 300 graphite fiber laminates. The load was set to cycle between a maximum stress and a small positive stress ($\sigma_{\max} \approx \Delta\sigma$). The cycling rate was 5 Hz. The microcrack density was observed by examining the sample edges using an optical microscope. The experimental results showed that the linear crack density did not increase after it reached 0.25/mm (figure 18a). However, Liu and Nairn did not fit their data to an empirical equation. We fit Liu and Nairn's data to empirical equation 21. A least squares best fit of the experimental data determined the values of C_1 , 0.204 (cracks/mm) and the values of λ , 0.000196 (Table 11).

Table 11. Comparison of the Damage Saturation Parameters and the Rate Constant for Various Studies

Materials & reference	ΔT (C)	Quench medium	Property* measured	Saturation** parameter	Rate constant	Maximum number of thermal shock cycles	$A\alpha/E_o$
Alumina/SiC this study	270	water	E	0.0074	0.042	100	0.0003
	310	water	E	0.0153	0.220	100	0.0034
	340	water	E	0.0268	1.51	100	0.0405
	380	water	E	0.0572	2.11	100	0.1207
TiB ₂ [71]	200	water	E	0.0014	0.065	100	0.00009
	250	water	E	0.0040	0.012	100	0.00005
	300	water	E	0.0361	0.164	100	0.0059
	400	water	E	0.0397	0.267	100	0.0106
	500	water	E	0.0577	0.245	100	0.0141
Macor [12]	200	water	E	0.0086	0.004	100	0.0003
	300	water	E	0.0222	3.60	100	0.0799
	350	water	E	0.0253	2.39	100	0.0605
	400	water	E	0.0442	0.475	100	0.0210
	450	water	E	0.0629	1.16	100	0.0730
	500	water	E	0.0668	0.964	100	0.0644
SiC/AS [11]	300	water	E	0.0020	0.097	100	0.0002
	370	water	E	0.0041	0.114	100	0.0005
	450	water	E	0.0072	0.193	100	0.0014
Alumina this study	230	water	E	0.0010	0.030	100	0.00003
	250	water	E	0.0086	0.087	100	0.0007

Table 11. (Continued)

Materials & reference	ΔT (C)	Quench medium	Property* measured	Saturation** parameter	Rate constant	Maximum number of thermal shock cycles	$A\alpha/E_o$
Alumina this study	270	water	E	0.0147	0.499	100	0.0073
	290	water	E	0.0171	0.616	80	0.0105
	310	water	E	0.0574	1.005	20	0.0577
	400	oil	E	0.0001	0.242	40	0.00002
	500	oil	E	0.0011	0.028	40	0.00004
	575	oil	E	0.134	0.08	40	0.0107
	935	liquid nitrogen	E	0.0007	0.169	40	0.0001
	1050	liquid nitrogen	E	0.0011	0.232	40	0.0003
MgO (refractory) [70]	1180	air	σ	0.249	1.107	30	0.276 ⁺
Al ₂ O ₃ -SiO ₂ (refractory) [70]	1180	air	σ	0.575	0.272	15	0.156 ⁺
MgO-Cr ₂ O ₃ (refractory) [70]	1180	air	σ	0.835	0.737	30	0.615 ⁺
60 % Al ₂ O ₃ (refractory) [72]	825	air	E	0.54	0.550	10	0.297
Al ₂ O ₃ -Cr ₂ O ₃ (refractory) [72]	825	air	E	0.76	0.781	10	0.593
70 % Al ₂ O ₃ (refractory) [72]	825	air	E	0.26	0.566	36	0.147
90 % Al ₂ O ₃ (refractory) [72]	825	air	E	0.41	0.642	36	0.263
70 % Al ₂ O ₃ (burned Phosphate bonded, refractory) [72]	825	air	E	0.64	0.308	36	0.197

Table 11. (Continued)

Materials & reference	ΔT (C)	Quench medium	Property* measured	Saturation** parameter	Rate constant	Maximum number of thermal shock cycles	$A\alpha/E_0$
80 % Al_2O_3 (refractory) [73]	1175	N.A.++	E	0.41	1.04	4	0.426
99 % Al_2O_3 (refractory) [73]	1175	N.A.	E	0.49	1.08	4	0.529
40 % Al_2O_3 (refractory) [73]	1175	N.A.	E	0.81	1.54	4	1.247
20 % Cr_2O_3 (refractory) [73]	1175	N.A.	E	0.67	1.39	4	0.931
96 % MgO (refractory) [73]	1175	N.A.	E	0.64	1.69	4	1.082
Si_3N_4 [74]	730	water	H	0.362	2.233	8	0.81^+
	730	air	H	0.145	1.222	8	0.18^+
graphite- epoxy [69]	278	air	ρ	$0.62-0.81_1$ (mm^{-1})	$0.00019-$ 0.098	500	N.A.
graphite- epoxy [75]	mechanical fatigue ($\Delta\sigma = 188.4$ MPa)		ρ	0.204 (mm^{-1})	0.000196	56000	N.A.

* E = Young's modulus, σ = modulus of rupture, H = hardness and ρ = crack density

** All the values except ρ are normalized values. For example, the modulus decrement values A (figure 26) were normalized by the undamaged modulus E_0 .

+ These values represent normalized damage times rate constant.

++ N.A. = not available

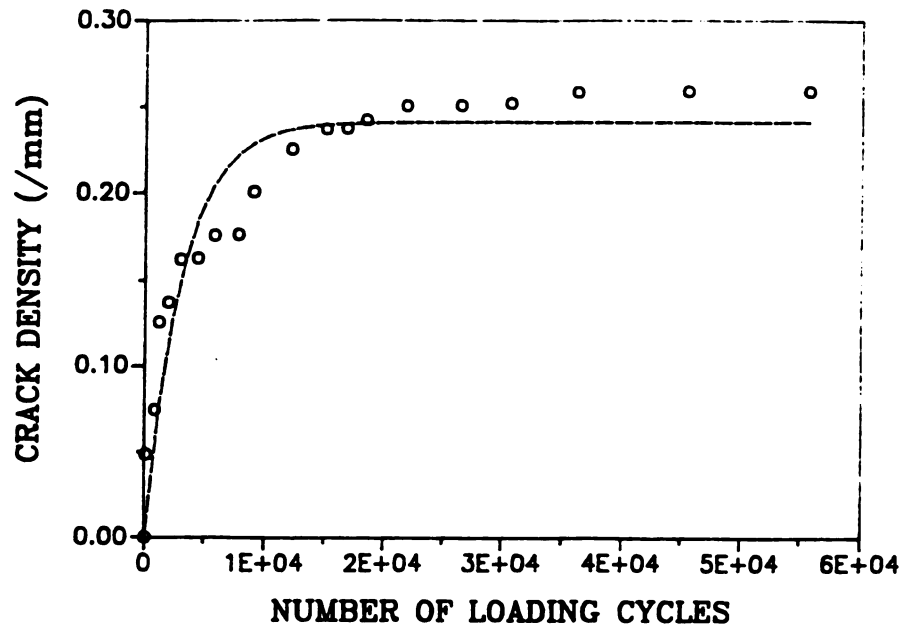


Figure 18a. Linear crack density as a function of mechanical loading cycles for $[0_2/90_4]_s$ Fiberite 934/T300 composites. The curves represent least-squares best fit to equation 21. (After [75])

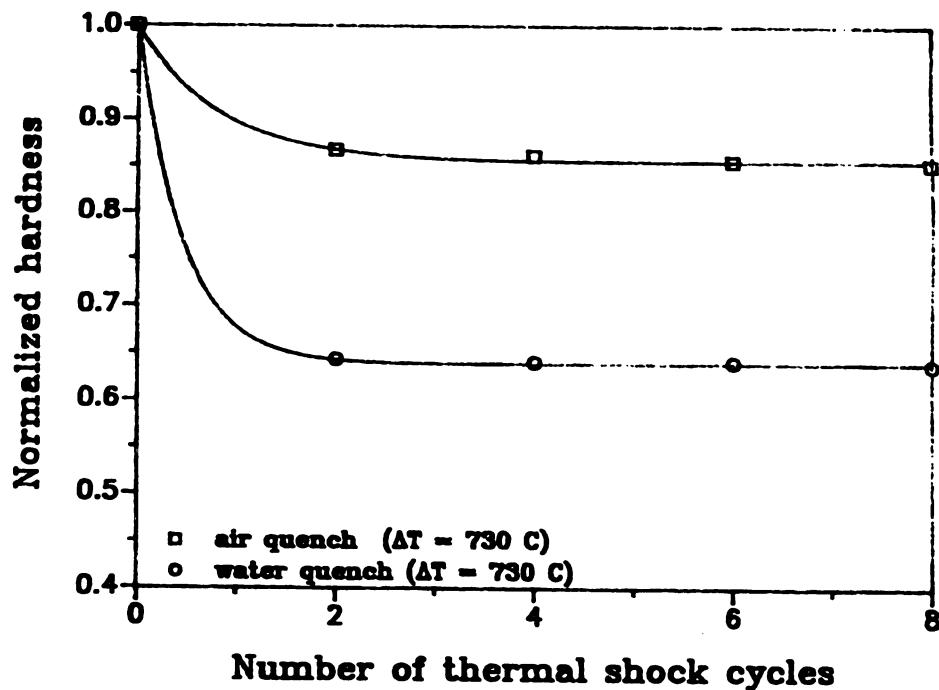


Figure 18b. Microhardness of Si_3N_4 as a function of the cumulative number of thermal shock cycles ($\Delta T = 730 \text{ C}$). The curves represent least-squares best fit to equation 22. (after [74])

Surface degradation of Si_3N_4 was observed in a study of repeated quenching [74]. Polished silicon nitride specimens were held at 750 C for 30 minutes. The specimens were then quenched into room temperature water or into air for up to 8 cycles. The surface degradation, which was attributed to a combination of oxidation and microcracking, was assessed by measuring a microhardness. The surface degradation in terms of normalized hardness (figure 18b) saturated in a manner analogous to the modulus saturation observed in this study (figures 15 and 16). We fit Sheu and Scrutton's hardness data to empirical equation

$$P/P_0 = 1 - D/P_0 (1 - \exp(-\delta N)) \quad (22)$$

where P_0 , P - undamaged and damaged specimen's material parameter

D , δ - positive constants similar to A and α

N - number of thermal shock cycles.

The results of the non-linear regression analysis of the hardness data are shown in table 11. The correlation coefficients were 0.99.

Damage saturation behavior has been observed during the thermal cycling of refractories. Ainsworth and Herron [70] studied thermal shock damage of refractories, such as MgO , $\text{MgO-Cr}_2\text{O}_3$, and $\text{Al}_2\text{O}_3\text{-SiO}_2$. Up to 30 thermal shock cycles between room temperature and 1204 C were performed with air cooling. An electrically heated furnace was heated to 1204 C and the test specimens were placed inside. After 10

minutes, the specimens were removed from the furnace and cooled in room temperature air for 10 minutes. The modulus of rupture (MOR) was measured before and after the thermal cycling. The damage was evaluated by modulus of rupture changes. The damage in terms of normalized MOR (figure 18c) saturated in a manner analogous to the modulus saturation observed in this study (figures 15 and 16). However, Ainsworth and Herron did not fit their thermal shock data to an empirical equation. We fit Ainsworth and Herron's MOR data to empirical equation 22. The results of the non-linear regression analysis of the MOR data are shown in table 11. The correlation coefficients were 0.92-0.98.

Semler and Hawisher [72] expressed the thermal shock damage of various high alumina refractories in terms of modulus. Various specimen sizes tested, including 0.228 m straight brick ($0.228 \times 0.114 \times 0.064$ or 0.076 m), splits ($0.228 \times 0.114 \times 0.025$ or 0.038 m), soaps (0.228×0.061 or 0.076×0.057 m), quarters ($0.228 \times 0.057 \times 0.032$ m) and bars ($0.228 \times 0.025 \times 0.025$ m)*. The specimens were heated by using a 1.5 m long line burner. The hot face region was exposed to the burner flame during the heating cycles and to moving air during the cooling cycles. Forced air cooling was achieved with the burner flame off and with an air blower on. Each thermal shock cycle was 15 minutes heating and 15 minutes cooling. A hot face temperature of

 * The second term in the parenthesis represents the hot face to cold face thickness.

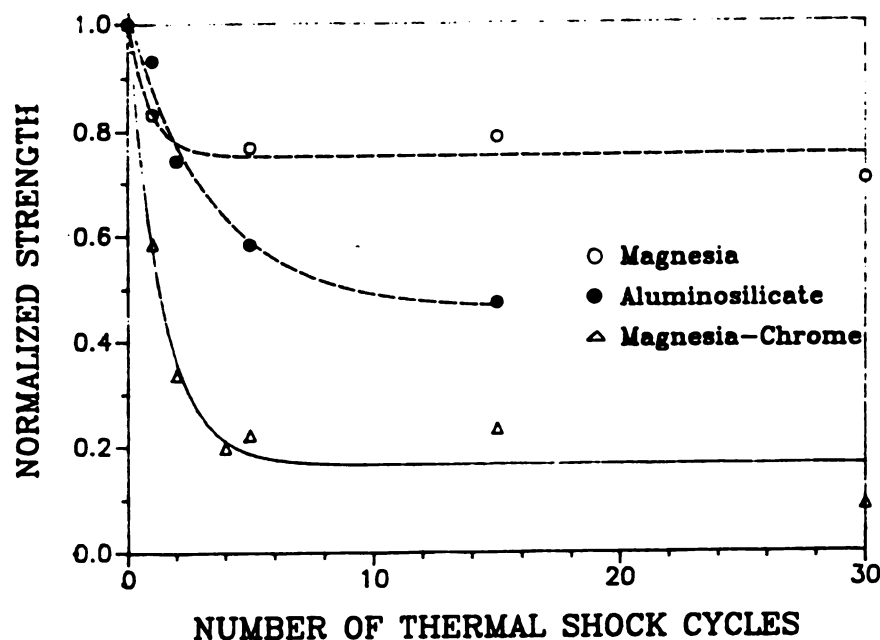


Figure 18c. Modulus of rupture of refractories (MgO , $\text{MgO-Cr}_2\text{O}_3$ and Al_2O_3) as a function of the cumulative number of thermal shock cycles (static air shock, $\Delta T = 1204^\circ\text{C}$). The curves represent least-squares best fit to equation 22. (after [70])

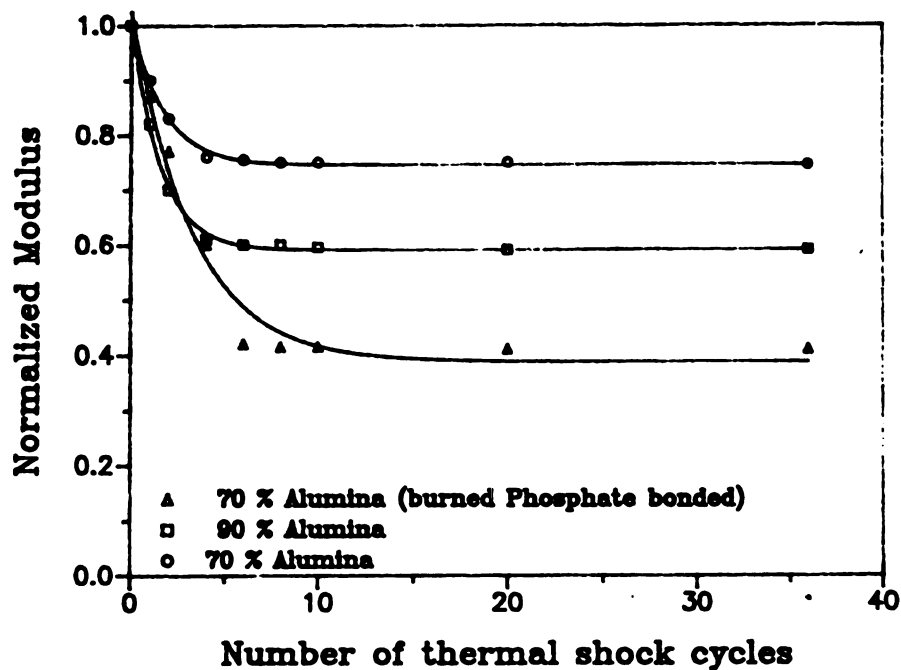


Figure 18d. Modulus of alumina refractories as a function of the cumulative number of thermal shock cycles (forced air shock, $\Delta T = 825^\circ\text{C}$). The curves represent least-squares best fit to equation 19. (after [72])

1000 C, as measured by a thermocouple, was achieved during heat-up while the temperature dropped to 150-200 C during cool-down. Semler and Hawisher's data [72] shows saturation behavior in the plot of elastic modulus versus the number of thermal shock cycles (figures 18d and 18e). However, Semler and Hawisher did not fit their thermal shock data to an empirical equation. Again, we fit Semler and Hawisher's modulus data to our empirical equation 19. The results of the non-linear regression analysis of the modulus data are shown in table 11. Note that figures 18c and 18d (Semler and Hawisher's modulus data for thermal shocked refractories [72]) shows trends very similar to the modulus data measured for thermally shocked polycrystalline alumina (figure 23) and thermally shocked SiC whisker reinforced alumina (figure 15).

When a surface is cooled, the thermal stresses are depend on the ratio (Biot modulus) of products of surface heat transfer coefficient and the sample dimension to the thermal conductivity of the material (equation 66). As Biot modulus increases, the thermal shock severity increases (Section 4.4.4. and Appendix E). The heat transfer coefficient for forced air quench can be as large as a $1/4$ - $1/40$ of the heat transfer coefficient for water quench [79], depending on the flow rate of the forced air. However due to the high ΔT and larger thickness for the air shocked refractories, the severity of the forced air shock for the refractories can be comparable to the water shock of the smaller specimens included in this study.

Lemmens [73] showed the effect of successive thermal shock

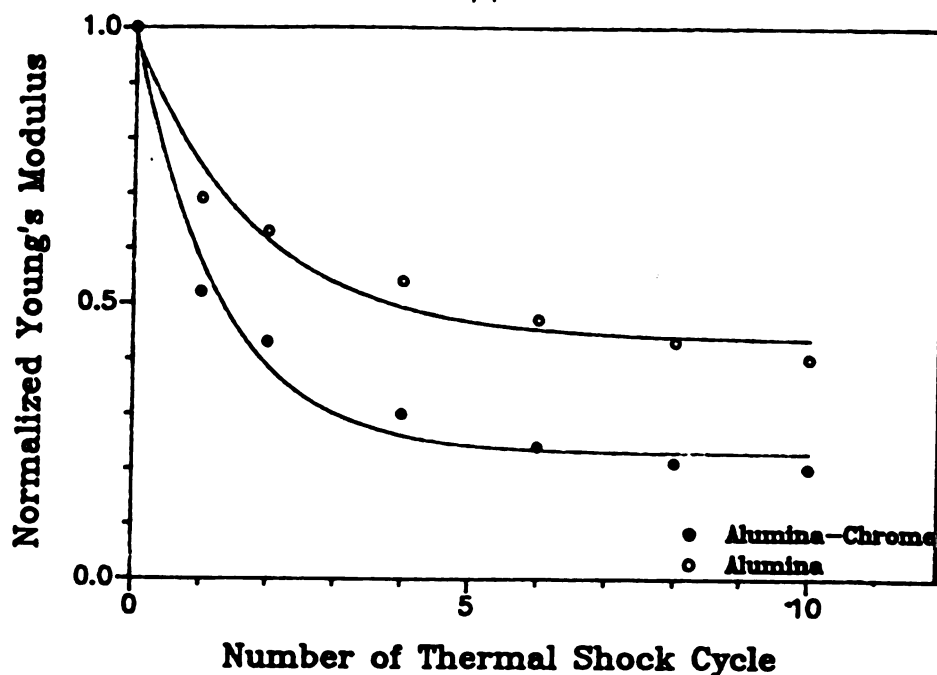


Figure 18e. Modulus of refractories (60 % alumina and $\text{Al}_2\text{O}_3\text{-Cr}_2\text{O}_3$) as a function of the cumulative number of thermal shock cycles (forced air shock, $\Delta T = 825^\circ\text{C}$). The curves represent least-squares best fit to equation 19. (after [72])

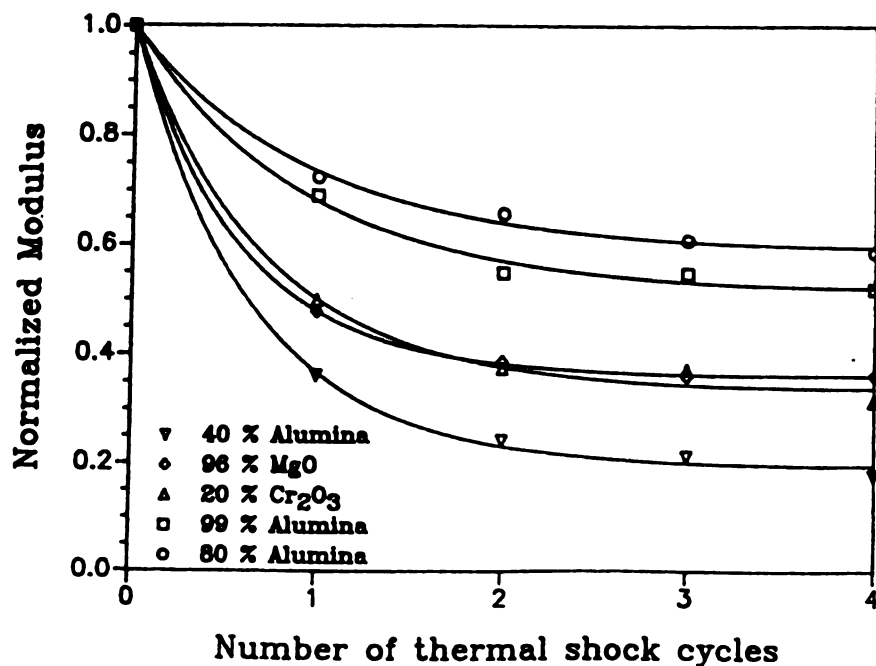


Figure 18f. Modulus of refractories as a function of the cumulative number of thermal shock cycles ($\Delta T = 1175^\circ\text{C}$). The curves represent least-squares best fit to equation 19. (after [73])

cycling (1200 C to 25 C) on modulus of refractory materials (80 % Al_2O_3 , 99 % Al_2O_3 , 40 % Al_2O_3 , 20 % Cr_2O_3 and 96 % MgO). Lemmens did not describe the details of the thermal shock experiments. Specimens were thermally shocked for up to only 4 cycles. The thermal shock damage was expressed the thermal shock damage in terms of a resonant frequency change. For a fixed geometry and density, the square of normalized resonance frequency should be proportional to the normalized modulus (equations 2 and 4). Thus we can convert the normalized resonance frequency data into the normalized modulus. We fit Lemmens' converted modulus data to our empirical equation 19. The results of the non-linear regression analysis of the modulus data are shown in table 11. Again figures 18f shows trends very similar to the modulus data measured for thermally shocked polycrystalline alumina (figure 23) and thermally shocked SiC whisker reinforced alumina (figure 15).

The damage saturation constants and rate constants for cyclic thermal shock study for various ceramics that were obtained in this study were compared to the cyclic thermal shock results of other researchers' (Table 11) [69, 70-75]. The constants summarized in Table 11 were determined for a variety of quenching media. Furthermore the constants obtained from the cyclic mechanical fatigue of a polymer based composite were included for comparison.

In order to compare our regression equation (equation 19) to Heracovich and Hyer's regression equation (equation 21), we modified equation 19 as follows.

The observed decrease in the elastic modulus with increasing number of thermal cycles (equation 19) can be expressed in terms of crack density parameter. The reduction in Young's modulus as a function of microcrack density parameter may be expressed as [76]

$$E = E_0 (1 - f(\nu_0)\epsilon) \quad (23)$$

$$\text{where } f(\nu_0) = 16(10 - 3\nu_0)(1 - \nu_0) / 45(2 - \nu_0) \quad (24)$$

ν_0 - Poisson's ratio for the noncracked material

E - the effective Young's modulus

E_0 - the effective or original Young's modulus

ϵ - crack density parameter.

For randomly oriented circular cracks of mean radius $\langle a \rangle$, the crack density parameter ϵ is defined as the product, $N\langle a \rangle^3$, where N is microcrack number density (number of microcracks per unit volume). The effective modulus varies linearly with the crack density parameter for microcracking models due to Salganik [76] and Budiansky and O'Connell [77]. The critical value of the damage, ϵ_{crit} , at which E vanishes is $1/f(\nu)$.

A similar linear relationship between modulus and the linear crack number density, ρ was obtained by Ogin et al. [78] in a mechanical fatigue study of glass fiber reinforced plastic composites. Ogin et al. [78] expressed the modulus reduction as a function of crack density in the form of

$$E = E_0(1 - c\rho) \quad (25)$$

where c is constant. A c value of 0.054 mm/cracks was determined from the experimental results [78]. Note that the ρ refers only to those cracks that are visible in an optical microscope. In contrast ϵ represents the total cracks including microcracks. Nevertheless equation 25 fit Ogin et al's experimental data [78] well.

If we combine equations 19 and 23, then we can express the accumulative damage in terms of crack density parameter. The resulting equation is

$$N = C_2(1 - \exp(-\alpha N)) \quad (26)$$

where $C_2 = A/[E_0 * f(\nu_0) * \langle a \rangle^3]$

Equations 21 and 26 are of the same mathematical form. Note that the dimension of C_1 in equation 21 is [cracks/unit length] whereas C_2 in equation 26 is [cracks/unit volume]. It is difficult to compare values of C_1 and C_2 directly due to the dimensional differences. However, values of α are generally larger than the values of λ (Table 11). The low values of λ might result from the difference in brittleness between polymers and ceramics. Actually in the thermal cycling study of graphite epoxy composites the most brittle system had the highest λ value for a given ΔT condition [67]. Herakovich and

Hyer [67] argued that "In the more brittle system, the increase in crack length was more sudden."

The cyclic loading data from other researchers [70, 72-75] were taken directly from their published plots of measured property. The constants were calculated by a nonlinear regression fit of equations 19, 21 and 22 to their data. Although the quenching media, materials and ΔT are different, the integrated saturation parameter and rate constants data (figure 19) show that as rate constant increases, saturation parameter also increases. A comparison of the $A\alpha/E_0$ constants for this study and the calculated $A\alpha/E_0$ from refractories showed that the $A\alpha/E_0$ for the refractories are much higher (Table 11, figure 19) than those for dense ceramics.

Although the quenching media and ΔT were different among the dense ceramics and the refractories, A/E_0 for the refractories is much higher than that for the dense ceramics. Refractories usually have a relatively high volume fraction porosity, which in turn provides a high density of Griffith flaws. When stressed, pores act to initiate fracture locally. As pore volume increases, the number of pores or possible fracture initiation sites increases. For a given severity of thermal shock, more cracks are propagated in a porous samples than in a nonporous one. Pores also may act as crack arresters. When the crack front arrives at a pore, the crack may be forced to change its path or stop. The large number of pores serve as crack initiation site as well as crack arrest sites. For polycrystalline alumina, as thermal shock severity increases, the values of A/E_0 increase.

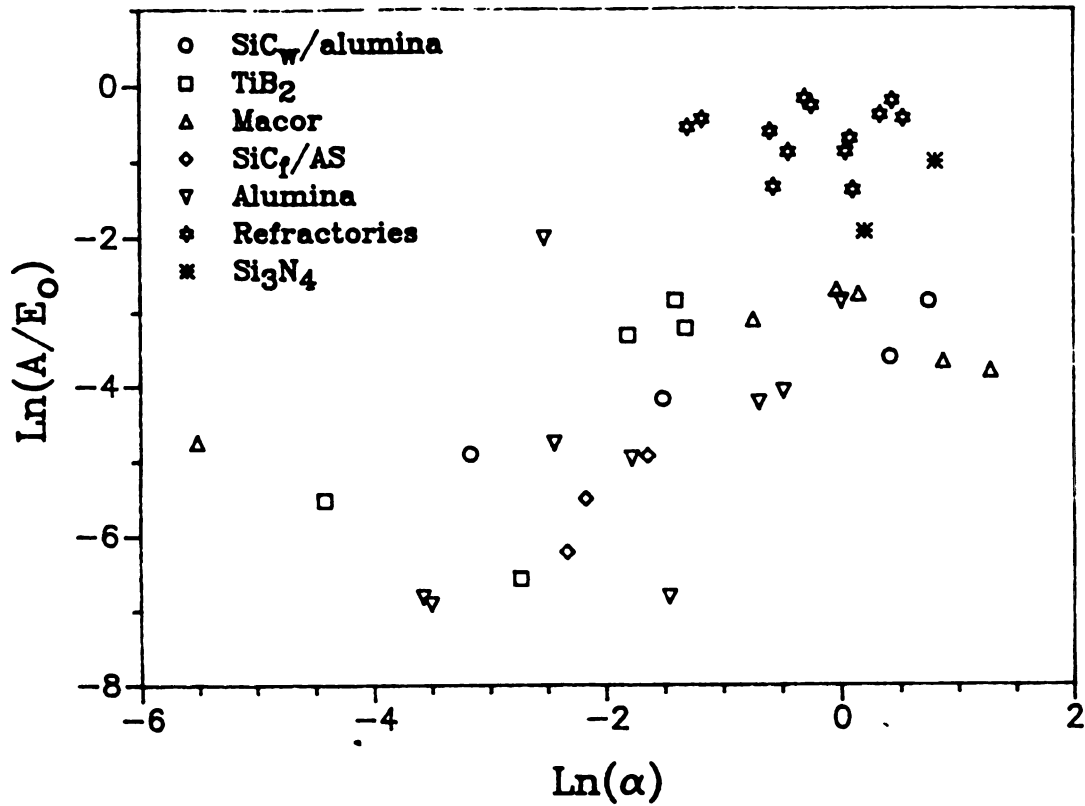


Figure 19a. Logarithm of the normalized saturation damage versus logarithm of the rate constants

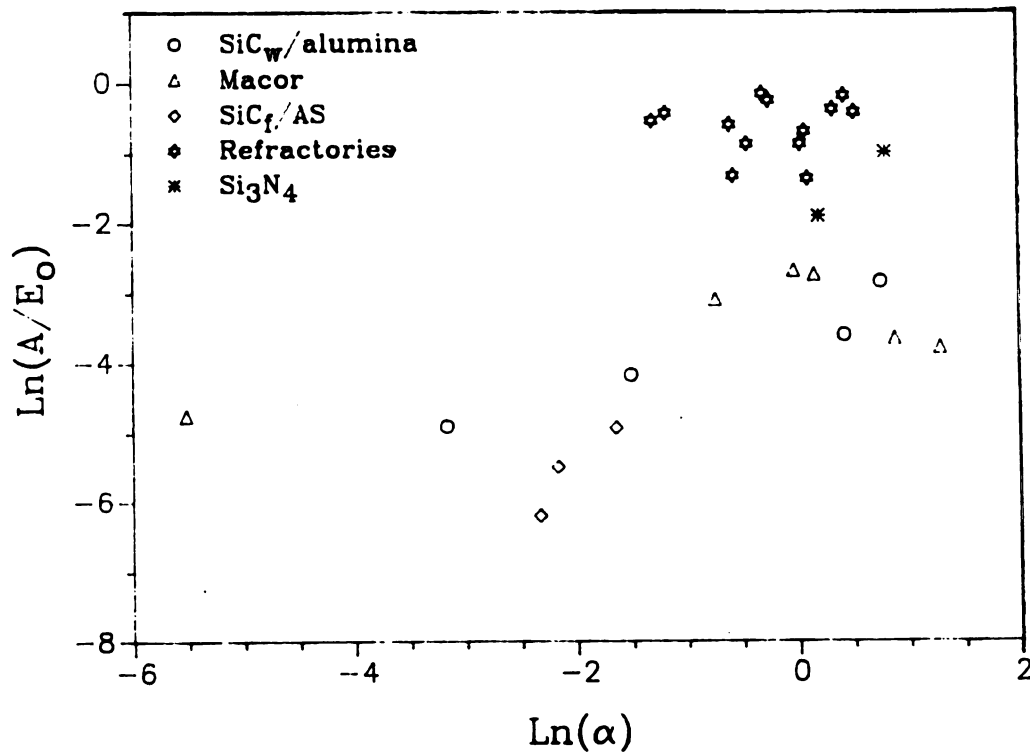


Figure 19b. Logarithm of the normalized saturation damage versus logarithm of the rate constants (without Al_2O_3 and TiB_2)

However due to the lack of crack arresters (pores or reinforcement), at high values of ΔT , dense ceramic specimens tend to fracture without saturation behavior. Thus for porous ceramics and composites, the crack arresters allow a proliferation of small cracks to propagate and stop, rather than having a few catastrophic cracks grow and fracture specimen. Such behavior (that is, the propagation of a myraid of small cracks rather than a few catastrophic cracks) for porous refractories ceramics was recognized and modeled in 1969 by Hasselman [116].

A key result of this study is that the thermal shock induced damage saturated after a number N_{sat} of thermal shock cycles. Furthermore, N_{sat} was found to be a sensitive function of ΔT , the thermal shock severity (figures 15, 23 and Table 10). The observed steady state value of Young's modulus and MOR for the 5th through the 36th thermal cycles for the refractory specimens [70, 72-73] is assumed to indicate crack arrest in the specimens. Some of the factors that may give rise to such a saturation effect will be discussed.

During cooling, transient tensile stresses develop on the exterior specimen surfaces, with compressive stresses in the interior bulk of the specimen. In essence, the quickly-cooled outer layer of the specimen attempts to "shrink" over the still hot interior bulk of the specimen.* Crack arrest during thermal shock can occur when a

* The rate at which the "cooling signal" propagates through the specimen will be a function of the thermal diffusivity of the specimen material [80].

thermal shock induced crack grows from the surface region of high tensile stresses into a region with rapidly decreasing tensile stresses and eventually into a compressive stress region [79]. Furthermore especially for water shock experiments included in this study the stress pulse is very transient (tens of milliseconds). This temporal limitation on the stress pulse likely contributes to the limited crack growth [115].

In addition to the stress distribution through the specimen thickness, crack arrest may result from repeated crack deflections from the inclusions [32, 33] such as reinforcing whiskers, fibers, particulates, or pores. As an example, for a semicircular crack of area ζ and radius r in a whisker (particulate) reinforced composite, in which (ideally) the angular orientation of the whiskers (particulates) is random and that the whiskers (particulates) are homogeneously distributed in the matrix, the number of whiskers (particulates) ω intersected by the crack is then

$$\omega = \Gamma \zeta \quad (27)$$

where Γ is the areal whisker (particulate) number density in the composite. For the semicircular crack of area ζ ($\pi r^2/2$), the change in crack area per unit crack advance dr would be

$$d\zeta = \pi r dr \quad (28)$$

Thus, for constant Γ , $d\omega = \pi \Gamma r dr$. (29)

Therefore, the differential number of whiskers (or particulates) intersected by the semicircular moving crack increases linearly with whisker (particulate) number density, Γ , the crack radius r , and the differential crack length increment dr .^{*} Crack deflection and pinning by the whiskers (particulates), in turn depends on the whisker (particulate) spacing, aspect ratio, and penetrability [32, 33]. The key point here is that the rate at which an advancing semicircular crack encounters whiskers (particulates) increases with crack length, for each incremental unit of crack advance.

Irreversible processes such as friction upon loading and loading of whiskers bridging the crack may contribute to fatigue and eventual damage saturation [81, 82]. For unidirectionally oriented fibers with a macrocrack running normal to the fiber direction, Marshall and Evans [81, 82] have discussed matrix-fiber frictional losses during loading and unloading the crack. For the SiC whisker reinforced specimens and Macor included in this study, whisker (particulate) pull out is likely to be a less important energy dissipative mechanism than in the unidirectional fiber case. For example, in a micrograph of a surface of the SiC whisker reinforced alumina specimens fractured in bend

^{*} In the case of a through crack advancing through a plate, $d\omega$ would be independent of crack length. While thermal shock cracks are likely either elliptical or slot shaped cracks, it is assumed that the growing semicircular crack is a plausible first order model of damage induced by thermal shock damage. The model of a through crack advancing in a plate would of course be more appropriate for fracture tests such as the double cantilever beam.

(figure 2), only those whiskers oriented nearly normal to the macrocrack pulled out, while whiskers not normal to the plane of the macrocrack tended to fracture rather than pull out. Whiskers nearly parallel to the macrocrack plane tended to remain embedded in one of the two macrocrack surfaces.

For a 20% SiC whisker reinforced alumina composite, Tiegs and Becher [57] found that flexural strength did not decrease for an initial thermal shock with temperature differences up to 900 °C. However Tiegs and Becher observed a large decrease in flexural strength for an initial thermal shock of alumina. For ten thermal shock cycles of SiC whisker reinforced alumina composites at $\Delta T = 500$ °C, only a slight decrease in flexural strength was observed, indicating that after the first few thermal shocks, a steady state damage level was obtained. The resistance to crack growth was explained as "a result of interaction between the SiC whiskers and microcracks in the matrix [57]."

The damage saturation observed for the thermal fatigue experiments included in this study may likely be explained in terms of the transient thermomechanical stress distribution in the specimen and by crack-crack arrester interactions. For refractory materials studied by other researchers [70, 72, 73], crack extensions due to cyclic thermal shock are likely arrested by the pre-existing pores during the repetitive cycling in the cyclic thermal shock test. Unless higher thermal stresses are provided, crack growth may not proceed.

4.1.5. Elasticity and Internal Friction Changes Expressed in terms of the Crack Damage Parameter

The observed decrease in the elastic modulus of the SiC whisker reinforced alumina specimens with increased thermal cycling can be modeled quantitatively on the basis of microcrack damage accumulation with increased thermal shock damage. The reduction in Young's modulus as a function of microcrack damage may be expressed as [76]

$$E = E_0 (1 - f(\nu_0)\epsilon) \quad (23)$$

$$\text{where } f(\nu_0) = 16(10 - 3\nu_0)(1 - \nu_0) / 45(2 - \nu_0) \quad (24)$$

ν_0 - Poisson's ratio for the noncracked material

E - the effective Young's modulus

E_0 - original Young's modulus

ϵ - crack damage parameter.

The calculated value of Poisson's ratio, ν , was 0.22 as determined from the elasticity data in this study. For the SiC whisker reinforced alumina composite specimens, the calculated value of $f(\nu_0)$ in equation 24 was 1.775. In general the value of $f(\nu_0)$ in equation 24 is a very weak function of Poisson's ratio. In fact, $f(\nu_0)$ changes by no more than a few percent when Poisson's ratio ranges from 0.4 to 0.1, which includes the observed values of Poisson's ratio for most ceramic materials [83].

From equations 19 and 23, one can express

$$E_0(1-f(\nu_0)\epsilon) = E_0 - A(1-\exp(-\alpha n)) \quad (30)$$

From equations 20 and 30, we obtain

$$\Delta Q^{-1} = B(1-[1-(E_0/A)f(\nu_0)\epsilon]^{\beta/\alpha}) - C_1[1-(1-C_2\epsilon)^{C_3}] \quad (31)$$

The entire data set of internal friction as a function of thermal shock damage for the four SiC whisker reinforced alumina matrix composites was fit to the empirical relation given by equation 31 via non-linear regression analysis. The constants C_1 , C_2 , and C_3 were 11.25×10^{-5} , 390, and 0.98, respectively.

For sufficiently small $C_2\epsilon$, the term $(1-C_2\epsilon)^{C_3}$ in equation 31 can be written as $(1-C_2C_3\epsilon)$ using the binomial expansion theorem. Thus, equation 31 becomes

$$\Delta Q^{-1} \approx C_1C_2C_3\epsilon - D\epsilon \quad (32)$$

The least-squares best fit of equation 32 gives a value of 4.25×10^{-2} for constant D. In equation 31, the product $C_1C_2C_3$ was 4.30×10^{-2} , which indicates that the linear approximation (equation 32) to the power-law form (equation 31) is in fact a good approximation (figure 20).

The correlation coefficients for equation 31 and 32 were 0.998 and 0.996, respectively. Thus both equations fit the data equally well. However, as a semi-empirical description of the internal

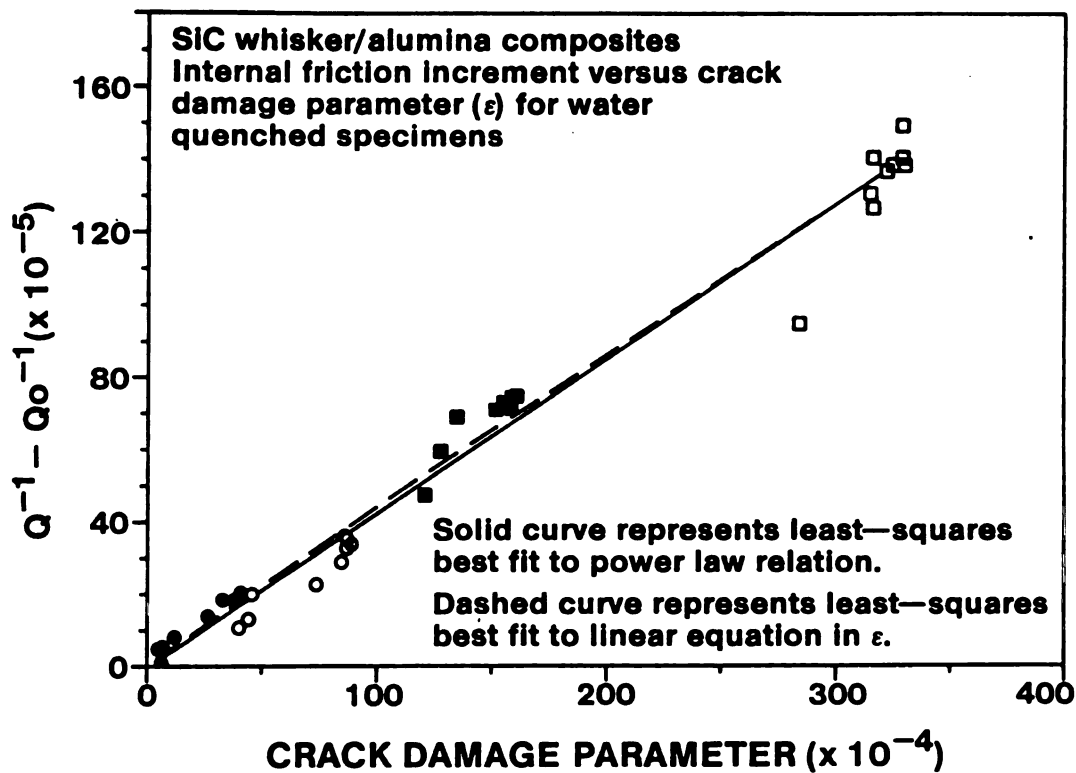


Figure 20. ΔQ^{-1} as a function of ϵ for the alumina/SiC whisker composite specimens. Symbols indicate the ΔT value for a given measurement on thermally fatigued specimens. Symbols are the same in figures 15 and 16. Note that measurements with similar ΔT values are grouped together along the curve.

friction-crack damage data, a simple linear equation with one adjustable parameter (equation 32) is certainly preferable to a power law relation with three adjustable parameters (equation 31). In figure 20 the four ΔQ^{-1} versus ϵ curves in figures 15 and 16 are effectively "collapsed" onto a single, linear ΔQ^{-1} versus ϵ , the crack damage parameter. As the crack damage increases, ϵ increases and in turn the observed ΔQ^{-1} increases linearly with ϵ . Note that measurements with similar ΔT values are grouped together along the curve. Symbols indicating ΔT values are the same as in figures 15 and 16.

4.1.6. Development of a Model for the Internal Friction Increase due to Microcrack Damage

The decrement in elastic moduli with increasing microcrack damage is treated theoretically by Salganik [76] and Budiansky and O'Connell [77] in terms of crack damage parameter ϵ . However, no similar theory is available for the corresponding increase in internal friction that accompanies the accumulation of thermal shock damage.

In order to develop a simple model for the internal friction increase due to microcrack damage, we shall begin by assuming that the microcracks' contribution to internal friction scales as the total integrated area of microcracks per unit volume of microcracked material. This assumption is consistent with the hypothesis that internal friction arises from the tribological interaction (rubbing) of asperities on opposing crack faces [8, 66, 84, 85]. For example,

we can consider the internal friction due to microcracks as arising from ζ , an average areal number density of asperities on opposing crack faces. We shall show that the assumption that internal friction scales as the integrated microcrack area leads to a functional form (equation 39) which is consistent with the observed changes in internal friction as a function of increased microcrack damage.

If the internal friction change due to microcracks is proportional to the integrated microcrack area, then in order to proceed one must establish a relationship between the third moment (ϵ) and the second moment (area) for a given distribution of crack sizes.

To determine the relationship between the third moment and the second moment (area) of a given distribution, we can use the moment generating functions (MGF) for the distribution* [86, 87], where the MGF, $\psi(t)$ is defined in terms of the expectation value of e^{tx} for the probability density function $f(x)$

$$\psi(t) = E [e^{tx}] = \int_{-\infty}^{\infty} e^{tx} f(x) dx \quad (33)$$

In particular, let us consider the gamma probability density function (pdf), which for the parameters k and λ is given by

* If the moment generating function is not available for a given distribution, we can evaluate the j th moment directly by $E[x^j]$. Alternatively, the moments can be evaluated from the characteristic function for the distribution, which is the Fourier transform of the pdf. A characteristic function may be defined for any continuous or discrete pdf [86, 87].

$$f(x) = [\lambda e^{-\lambda x} (\lambda x)^{k-1}] / (k-1)! \quad \text{for } x > 0 \quad (34a)$$

$$\text{and } f(x) = 0 \quad \text{for } x < 0 \quad (34b)$$

To find the j th moment of the pdf from $\Psi(t)$, we find the j th derivative of $\Psi(t)$ evaluated at $t = 0$. Thus, for the second moment and third moment can be evaluated as

$$\Psi''(t) = k(k+1)\lambda^k (\lambda-t)^{-k-2} \quad (35)$$

$$\text{and } \Psi'''(t) = k(k+1)(k+2)\lambda^k (\lambda-t)^{-k-3} \quad (36)$$

The ratio of the third moment, m_3 ($= -\Psi'''(0)$), to the second moment, m_2 ($= -\Psi''(0)$), is given by

$$m_3/m_2 = (k + 2)/\lambda \quad (37)$$

where k and λ are constants for a given gamma pdf. Thus for a given gamma distribution, the quantity $(k + 2)/\lambda$ is a constant that depends on the "shape parameters" k and λ for the distribution.*

* We can use the $\Psi(t)$ to evaluate the first moment (the mean) of the gamma pdf, which is k/λ . The variance of the gamma pdf is k/λ . Thus the "shape" of the gamma distribution (that is, the mean and the "spread" or variance of the distribution) can be expressed in terms of the constants k and λ (equation 28) that define the pdf.

Thus, if the crack size distribution may be approximated by a gamma distribution, then

$$\langle a^3 \rangle = [(k + 2)/\lambda] \langle a^2 \rangle = b \langle a^2 \rangle \quad (38)$$

where b is a constant for a given gamma distribution. Multiplying through equation 38 by the crack number density N gives

$$N \langle a^3 \rangle = b N \langle a^2 \rangle \quad (39)$$

Therefore, for the gamma distribution, if the internal friction increases linearly with the average crack area per unit volume (as assumed here), then the change in internal friction will increase linearly with the measured crack damage parameter, ϵ , as shown in the experimental data in figure 20.

Relationships similar to equation 39 can be derived for both the Rayleigh and the lognormal distributions (Appendix C).

4.1.7. The Dependence of the Saturation Values for Elastic Modulus on the Temperature Difference ΔT

For the cumulative number of thermal shock cycles, n , sufficiently high (that is, for $n \gg n_{oe}$), the factor $(1 - \exp(-\alpha n))$ approaches unity and equation 19 can be rewritten as

$$A/E_0 = 1 - E_{sat}/E_0 \quad (40)$$

where E_{sat} is the saturation (steady state) value of modulus as a function of thermal shock treatment (figure 15). The parameter A in equation 10 thus measures the difference in modulus between the undamaged state, E_0 , and the damage saturation value E_{sat} . Similarly from equation 20, B measures the increase in internal friction due to microcracking, namely

$$B/Q_0^{-1} = 1 + Q_{sat}^{-1}/Q_0^{-1} \quad (41)$$

If we plot the logarithm of the normalized (nondimensional) saturation damage values (A/E_0) and (B/Q_0^{-1}) versus the logarithm of normalized $\Delta T/T_r$ (in $^{\circ}K$), we obtain a straight line (figure 21), which implies a power law relationship between the normalized saturation values and $\Delta T/T_r$, namely

$$A/E_0 = j_1(\Delta T/T_r)^{p^1} \quad (42)$$

$$B/Q_0^{-1} = j_2(\Delta T/T_r)^{p^2} \quad (43)$$

where T_r , a reference temperature, was taken as 293 $^{\circ}K$ (room temperature) and ΔT was expressed in terms of $^{\circ}K$. A least-squares best fit yielded values of $j_1 = 0.0127$ and $j_2 = 2.447$ for the prefactors in equations 42 and 43. The exponents p^1 and p^2 are respectively 5.976 and 5.701.

For a given ΔT , and for a fixed reference temperature T_r , the

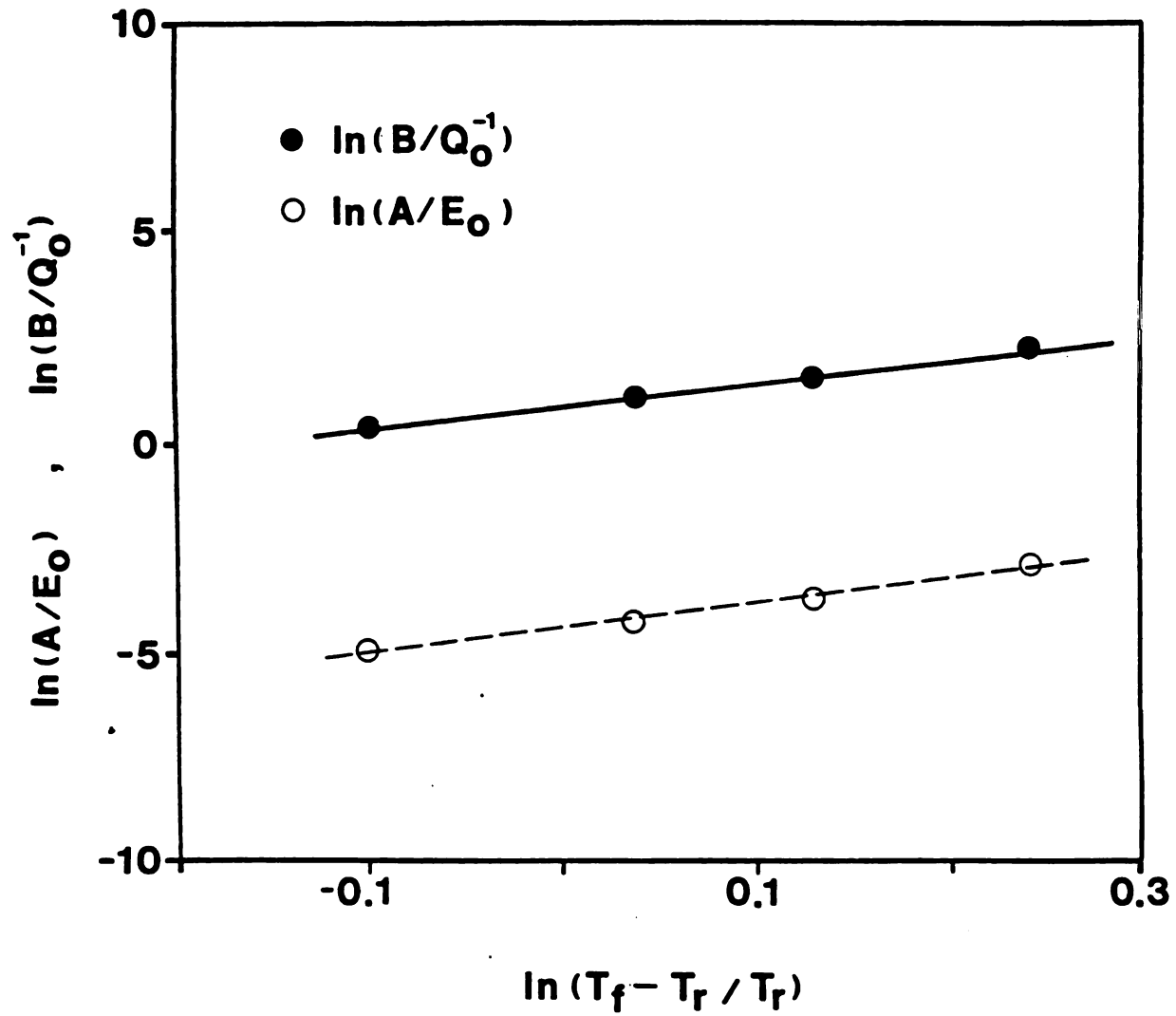


Figure 21. Logarithm of the normalized saturation damage parameters (A/E_0) and (B/Q_0^{-1}) versus logarithm of the normalized temperature for SiC whisker reinforced alumina.

ratio of the two normalized saturation parameters $(B/Q_0^{-1})/(A/E_0)$ is approximately equal to j_2/j_1 , which is a ratio of two constants that are not functions of either ΔT or T . The ratio j_2/j_1 (which is approximately 200) hence could be called a "sensitivity ratio" since it represents, for a given level of thermal shock damage, the relative sensitivity (or relative change) in internal friction compared to the changes observed for Young's modulus.

Equations 42 and 43 specify the ΔT dependence for the saturation values of damage from this thermal shock study. Both the similarity of the slopes of the curves in figure 21 and the least square fitting constants p^1 and p^2 indicate that the power p for both the internal friction and the elastic modulus is essentially the same.

4.1.8. Cumulative Damage Expressed in Terms of Fatigue Type Equation

The saturation damage parameter (A/E_0) can be expressed directly in terms of the crack damage parameter, if we use equation 23 (Salganik's expression) for modulus decrement

$$E_{sat} = E_0 [1 - f(\nu_0) \epsilon_{sat}] \quad (44)$$

where E_{sat} - saturation damage value of modulus (as in equation 40)

ϵ_{sat} - crack damage parameter for the saturation damage state

(see equation 23 for ϵ)

$f(\nu_0)$ - a function the nonmicrocracked Poisson's ratio ν_0 (see equation 24).

Combining equations 44 and 40 gives

$$A/E_0 = 1 - [1 - f(\nu_0)\epsilon_{sat}] - f(\nu_0)\epsilon_{sat} \quad (45)$$

For the particular case of circular cracks, ϵ_{sat} is given by $N\langle a^3 \rangle$.^{*} A similar expression in ϵ_{sat} can be derived for the internal friction parameter (B/Q_0^{-1}) . The damage saturation parameters can thus be written explicitly in terms of the crack number density and crack size.

Using equation 45, equation 42 may be written as

$$\epsilon_{sat} = j_3(\Delta T/T_r)^{p_1} \quad (46)$$

where j_3 - a constant given by $j_2/f(\nu_0)$.

If the final mean crack size at saturation, $\langle a \rangle_{sat}$, is much greater than the size of the initial flaws in the thermally shocked specimens (where initial refers to the as-received or unshocked

* The parameter ϵ_{sat} could also be expressed in a variety of noncircular geometries (including slot-shaped cracks), following the relationships developed for the crack damage parameter, ϵ , developed by Budiansky and O'Connell [77].

state), then the total crack growth due to thermal shock is measured by ϵ_{sat} . As discussed in section 4.1.6., the ratios of the third moment of crack size distribution $\langle a^3 \rangle_{\text{sat}}$ to the second moment $\langle a^2 \rangle_{\text{sat}}$ is given by

$$N \langle a^3 \rangle_{\text{sat}} = b N \langle a^2 \rangle_{\text{sat}} \quad (47)$$

where b = a constant depending on the shape parameters of the distribution (see equation 39).

Parameter $\langle a^2 \rangle_{\text{sat}}$ measures the area of the thermal shock induced cracks at the saturation damage condition. Using equation 47, equation 46 can be rewritten as

$$\Delta \zeta = j_4 (\Delta T / T_r)^{p_1} \quad (48)$$

where $\Delta \zeta$ = the total integrated crack area for the cracks induced by thermal shock damage

j_4 = a constant.

The power law in ΔT is suggestive of a power law relation in the surface stress σ , since the thermal shock induced stresses tend to scale linearly with the temperature differences [88]. Thus, in terms of stress and crack increments, equations 42 and 43 could be written as

$$\Delta \zeta = M \sigma^p \quad (49)$$

where p , M - constants.

Typically, models of crack fatigue characterize crack growth in terms of increments in crack length (an essentially one-dimensional view) rather than crack area. If L is a characteristic dimension of the crack (say the crack radius in the case of a circular crack), then we could write equation 49 as

$$\Delta L = H \sigma^p \quad (50)$$

where ΔL - the total, integrated crack growth occurring for all the thermal shock induced microcracks

H , p - constants.

The power law function in stress σ could of course be expressed alternatively as a power law function of the stress intensity factor, K . Although the power law relationship in stress (equation 50) is suggestive of a fatigue relationship similar to that known for metals (equation 1), it should be noted that crack extension due to slow crack growth also can yield a power law relation in stress [29].

4.2. Cyclic Thermal Shock into Water Bath of Polycrystalline Alumina

4.2.1. Young's Modulus, Shear Modulus and Internal Friction of Unshocked Specimens

The initial (unshocked) values of Young's modulus, shear modulus, internal friction, and Poisson's ratio for water quenched specimens are listed in table 12 for each of the specimens included in this study. As is typical for polycrystalline ceramics, the Young's modulus and shear modulus of all the unshocked specimens decreased with increasing porosity [89-93]. The unreinforced alumina specimens exhibit a relatively limited range of porosity, which in turn implies limited effects due to porosity. Appendix D includes a discussion of modulus and internal friction dependencies on porosity.

For unshocked specimens of unreinforced alumina, the internal friction of alumina varied from 6.23×10^{-5} to 10.6×10^{-5} , while that of unshocked SiC whisker reinforced alumina ranged from 13.2×10^{-5} to 18.6×10^{-5} . The internal friction values of the unreinforced alumina were thus about 50 percent lower than those of SiC whisker reinforced composites, despite the fact that elastic moduli for the composite were about 20 percent higher than those of the unreinforced alumina. The higher internal friction of the composites might be explained on the basis of the residual stresses [6] and the presence of interfaces

Table 12. Room Temperature Young's Modulus (E), Shear Modulus (G), Poisson's Ratio (ν) and Internal Friction (Q^{-1}) for Unshocked Unreinforced Alumina Specimens (After annealing)

Specimen \ Property	E (GPa)	G (GPa)	ν	Q^{-1} ($\times 10^{-5}$)
A1	333.85	135.68	0.23	10.6
A2	332.96	135.17	0.22	8.1
A3	323.87	132.78	0.23	8.57
A4	332.5	135.32	0.23	8.3
A5	338.53	*	*	6.23
A6	336.12	136.69	0.23	9.91

* These data were not measured.

between the matrix and the whiskers*, but further work needs to be done to clarify such differences in the internal friction for the composite and non-composite specimens.

Figure 22 illustrates Young's modulus as a function of temperature for annealed polycrystalline alumina specimen. The modulus changed linearly up to 600 degrees C. The modulus versus temperature data were fitted via least squares best fit to an linear equation

$$E = E_0 - cT \quad (18)$$

where E_0 = zero degrees C Young's modulus

T = temperature in Celsius

c = modulus-temperature slope.

The least squares procedure yielded a value of c , 0.027 GPa per degrees C. The modulus decreased linearly at a rate of 0.8 percent per 100 degrees C.

4.2.2. Effects of Cyclic Thermal Shock on Elasticity and Internal Friction

Figures 23-25 display the effects of cyclic thermal shock on the

* In polymer composites, internal friction contributions due to matrix-fiber interactions have been observed [85]. Similar effects are likely in ceramic composites.

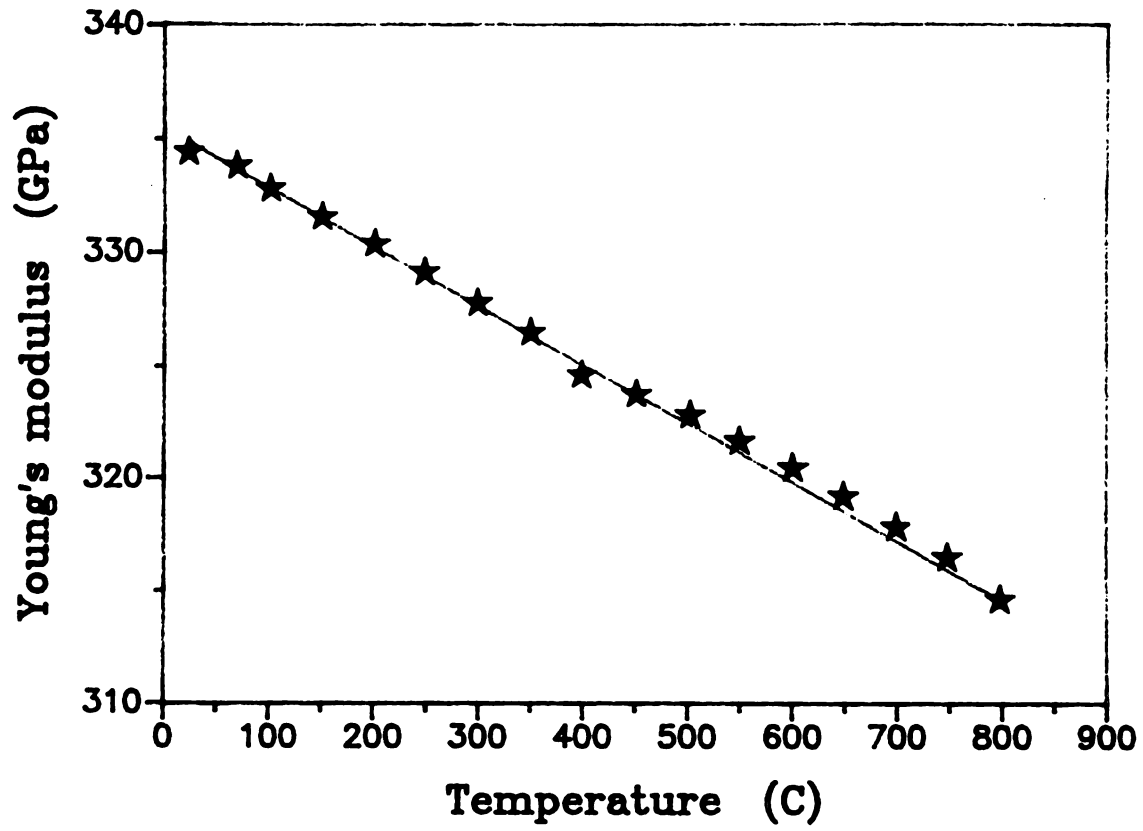


Figure 22. Young's modulus of the polycrystalline alumina specimen as a function of temperature.

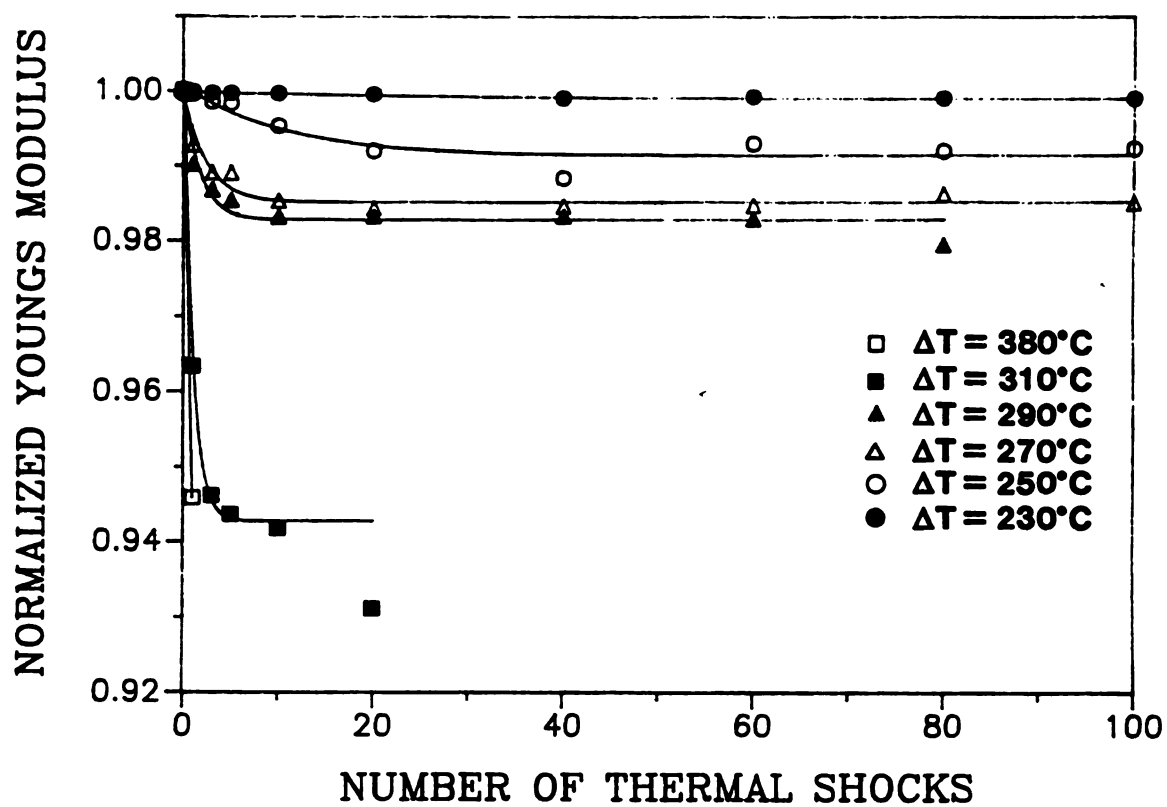


Figure 23. Young's modulus of alumina specimens, A1 ($\Delta T = 230$ degrees C), A2 ($\Delta T = 250$ degrees C), A3 ($\Delta T = 270$ degrees C), A4 ($\Delta T = 290$ degrees C), A5 ($\Delta T = 310$ degrees C), and A6 ($\Delta T = 380$ degrees C) as a function of the cumulative number of thermal shock cycles.

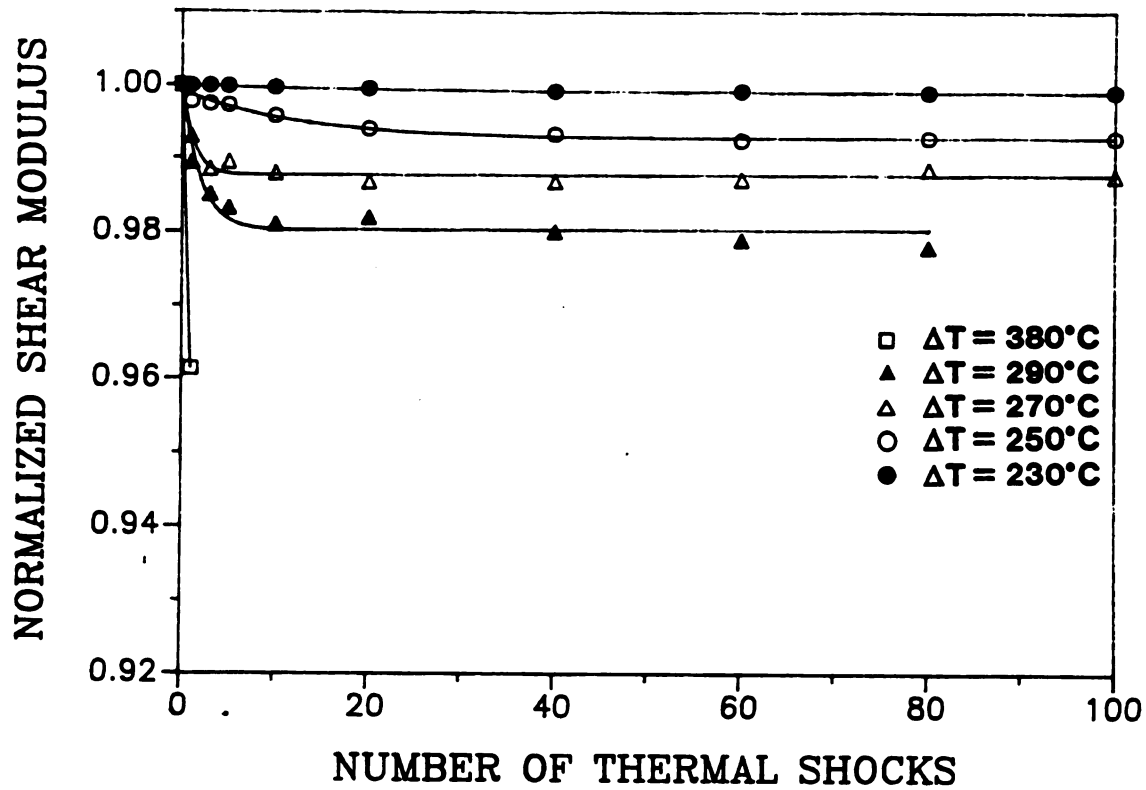


Figure 24. Shear modulus of alumina specimens, A1 ($\Delta T = 230$ degrees C), A2 ($\Delta T = 250$ degrees C), A3 ($\Delta T = 270$ degrees C), A4 ($\Delta T = 290$ degrees C), A5 ($\Delta T = 310$ degrees C), and A6 ($\Delta T = 380$ degrees C) as a function of the cumulative number of thermal shock cycles.

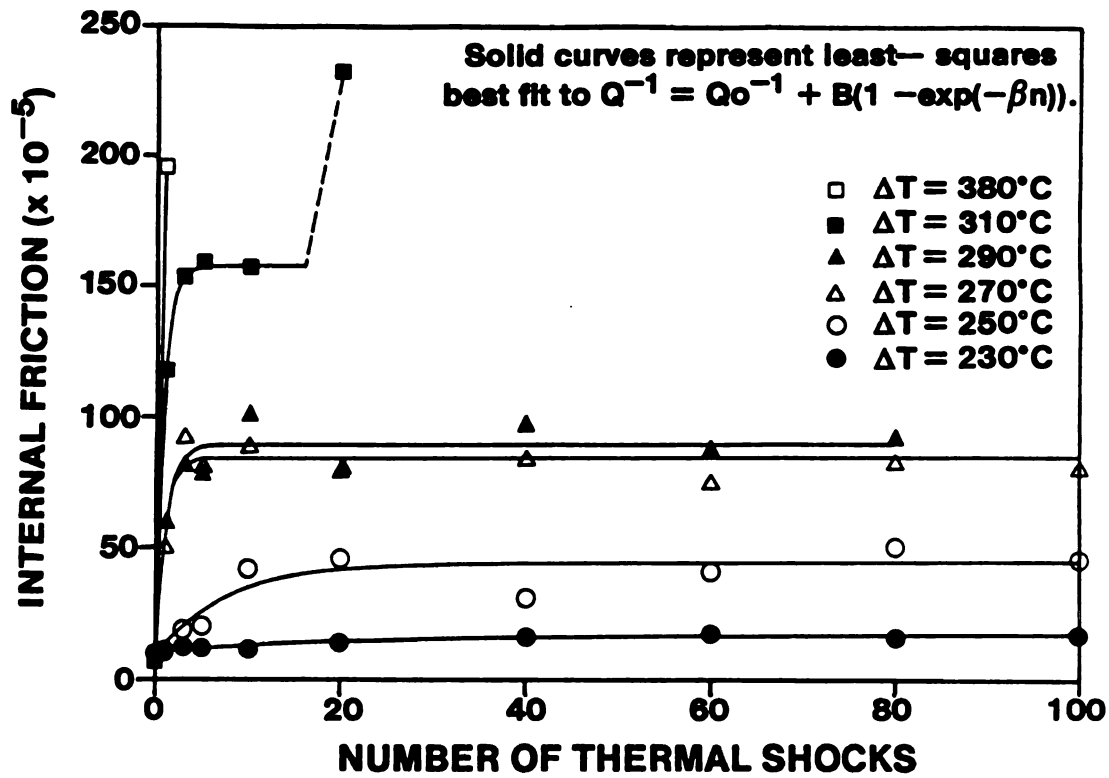


Figure 25. Internal friction of alumina specimens, A1 (ΔT = 230 degrees C), A2 (ΔT = 250 degrees C), A3 (ΔT = 270 degrees C), A4 (ΔT = 290 degrees C), A5 (ΔT = 310 degrees C), and A6 (ΔT = 380 degrees C) as a function of the cumulative number of thermal shock cycles.

Young's modulus, E , shear modulus, G , and internal friction, Q^{-1} for the unreinforced alumina specimens. At low and intermediate values of thermal shock severity, ΔT , the internal friction and modulus of the unreinforced alumina specimens behaved the same as the SiC whisker reinforced alumina (figures 15 and 16). Decreases in both the Young's modulus and the shear modulus always accompanied an increase in internal friction (figures 23-25). As ΔT increases, the thermal shock damage saturation levels also increase, as indicated by changes in modulus and internal friction.

Relative changes in modulus and internal friction are listed in table 13 for each of the six thermally shocked alumina specimens. The magnitude of the internal friction changes were much larger than modulus changes, which agrees with trends in elastic modulus and internal friction observed in other studies [5, 6, 8-10].

For the unreinforced alumina specimens included in this study, no saturation behavior was observed for the two most severe thermal shock conditions ($\Delta T = 310$ degrees Celsius and 380 degrees Celsius). Specimen A5 ($\Delta T = 310$ degrees Celsius) apparently approached saturation in the interval between 5 and 10 thermal shock cycles. However, an abrupt decrease in the Young's modulus and an abrupt increase in internal friction occurred for specimen A5 in the interval between 11 and 20 thermal shock cycles. The abrupt change may result from the linkage of the microcracks to form macrocracks, since a corner of the specimen A5 broke off after 40 cumulative thermal shock cycles. The corner fragment had a mass of 0.083 grams,

Table 13. Relative Changes in Modulus and Internal Friction for the Thermally Shocked Unreinforced Alumina Specimens and SiC Whisker Reinforced Alumina Composites. The subscript "o" refers to the value of the indicated parameter prior to the initial thermal shock, while the subscript "sat" refers to the "saturated" or steady-state value of the parameter that is obtained after repeated thermal cycling (see equations 19, 20 and 51.).

Specimen Label	ΔT (C)	$(E_o - E_{sat})/E_o$	$(G_o - G_{sat})/G_o$	$(Q_{sat}^{-1} - Q_o^{-1})/Q_o^{-1}$
Unreinforced Polycrystalline Alumina				
A1	230	0.0010	0.0009	0.56
A2	250	0.0078	0.0060	6.81
A3	270	0.0143	0.0122	9.00
A4	290	0.0171	0.0157	10.09
A5	310	0.0575	*	24.08
A6	380	**	**	**
SiC Whisker Reinforced Alumina Composites				
HP160	270	0.007	*	1.2
HP171	310	0.0172	*	3.18
HP160-RA	340	0.0285	*	5.04
HP158	380	0.0583	*	10.49

* Shear modulus was not measured for this specimen.

** For $\Delta T = 380$ degrees C, the specimen fractured after third thermal shock, so that saturation values of moduli and internal friction could not be determined.

compared to a total specimen mass of 5.815 grams. For specimen A6 (ΔT = 380 degrees Celsius), thermal shock damage was measurable only after the first and second thermal shock cycle since the specimen broke into two large pieces as a result of the third thermal shock.

For a temperature difference of 290 degrees Celsius, specimen A4 showed damage saturation behavior between 10 and 80 cumulative cycles. However, a 0.145 gram fragment broke off from a corner of the specimen after 81 thermal shock cycles (total mass of A4 was 5.816 grams). In figures 20-22, note the slight changes from the saturation damage level that are evident in the modulus and internal friction measurements at the 80 cumulative cycles. The fracture of a portion of the specimen upon the 81st thermal shock cycle implies that the deviation from the saturation damage level may be related to microcrack link-up and the failure of a portion of the specimen.

In contrast to the unreinforced alumina composites, none of the SiC whisker reinforced alumina specimen exhibited macrofracture (none of the SiC whisker reinforced alumina specimen fractured for temperature differences ΔT up to 380 degrees Celsius). In addition, each of the SiC whisker reinforced alumina specimens showed damage saturation behavior for up to 100 cumulative thermal shock cycles, with no significant deviation from saturation behavior.

4.2.3. Modulus and Internal Friction as a Function of Repeated Thermal Shocks

The same empirical equations developed for the modulus and internal friction changes of thermally shocked SiC whisker reinforced alumina composites can be fit to the thermal shock data for the unreinforced alumina, namely

$$E = E_0 - A (1 - \exp(-\alpha n)) \quad (19)$$

and
$$Q^{-1} = Q_0^{-1} + B (1 - \exp(-\beta n)) \quad (20)$$

where A, B - damage saturation parameters (see figures 26)

α, β - rate of decrement or increment (rate constant)

E_0, Q_0^{-1} - undamaged specimen's Young's modulus or internal friction

E, Q_0 - damaged specimen's Young's modulus or internal friction.

n - cumulative number of thermal shock cycles at a fixed ΔT value.

For the shear modulus, we introduce the relationship

$$G = G_0 - C (1 - \exp(-\gamma n)) \quad (51)$$

where C - damage saturation parameter (see figure 26)

γ - rate of decrement (rate constant)

G, G_0 - damaged or undamaged specimen's shear modulus

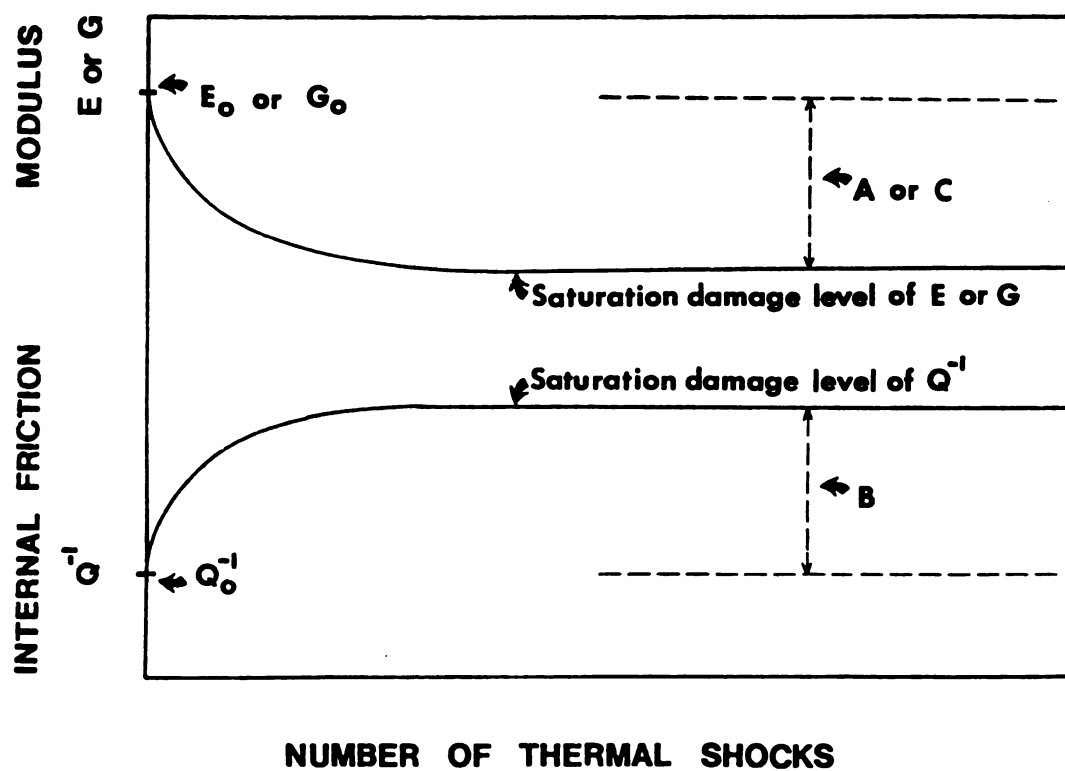


Figure 26. Schematic diagram illustrating thermal shock saturation levels A, B and C and the rate constants α , β and γ . (see equations 19, 20 and 51)

For a fixed value of ΔT , the constants A, B and C represent a damage saturation or steady state level of damage for a large number of thermal shock cycles (figure 26). The constants α , β and γ are related to rate of change of the modulus or internal friction as a function of thermal cycle treatment. We see from equation 19, 20 and 51 that in the limit of $n \rightarrow 0$, $dE/dn = -A\alpha$, $dG/dn = -C\gamma$, and $dQ^{-1}/dn = B\beta$.

Table 14 shows the results of the non-linear regression based on the equations 19, 20, and 51. As ΔT increases, the damage saturation, A, B, and C, and the rate constants, α , β , and γ increases. As discussed previously, equations 19 and 20 were used to describe the thermal fatigue behavior in SiC whisker reinforced alumina composites (Section 4.1.3), but we found the same equations could describe the thermal fatigue behavior of the unreinforced polycrystalline alumina. However, the parameters A, B, α and β for the SiC whisker reinforced alumina were different than the corresponding parameters for the unreinforced alumina. As discussed in section 4.2.7., the damage for given values of ΔT and n is greater for the unreinforced alumina than for the SiC whisker reinforced alumina composites.

4.2.4. Relation between Young's Modulus Saturation Constant A and Shear Modulus Saturation Constant C

Equations 19 and 51, respectively, describe the decrease in Young's modulus and decrease in shear modulus with microcrack damage accumulation. For a sufficiently high number of thermal shock cycles,

Table 14. For the Unreinforced Alumina Specimens, Results of the Nonlinear Regression Analysis for the Damage Saturation Parameters A, B, and C and the Rate Constants α , β and γ (see equations 19, 20 and 51 and figure 26). The Unshocked Values of Internal Friction Q_o , Young's Modulus, E_o , and Shear Modulus, G_o , are included for reference.

Specimen	A1	A2	A3	A4	A5	A6
ΔT (C)	230	250	270	290	310	380
$Q_o^{-1} (x10^{-5})$	10.6	8.1	8.6	8.3	6.2	9.9
E_o (GPa)	333.85	332.96	323.87	332.50	338.53	336.12
G_o (GPa)	135.68	135.17	132.78	135.32	*	136.69
A (GPa)	0.34	2.87	4.77	5.70	19.42	**
B ($x10^{-5}$)	6.0	34.9	75.4	80.68	150.9	**
C (GPa)	0.12	0.95	1.64	2.13	*	**
α	0.030	0.087	0.499	0.616	1.005	**
β	0.050	0.145	0.970	0.927	1.338	**
γ	0.036	0.178	0.836	0.886	*	**
cor. coeff. (α , A)	0.96	0.92	0.97	0.96	0.99	**
cor. coeff. (β , B)	0.96	0.94	0.98	0.97	0.99	**
cor. coeff. (γ , C)	0.99	0.97	0.98	0.95	*	**

* Shear modulus was not measured for this specimen.

** At $\Delta T = 380$ degrees C, no saturation behavior was observed since the specimen broke into two pieces after third thermal shock.

n, the factors $(1-\exp(-\alpha n))$ and $(1-\exp(\gamma n))$ approaches unity and equations 19 and 51 become

$$E_{sat} = E_0 - A, \text{ thus } E_{sat}/E_0 = 1 - A/E_0 \quad (52)$$

$$G_{sat} = G_0 - C, \text{ thus } G_{sat}/G_0 = 1 - C/G_0 \quad (53)$$

where E_{sat} and G_{sat} are the saturation (steady state) values of the moduli as a function of the cumulative number of thermal shock cycles (figures 24 and 25).

Salganik's [76] relation for the decrement in Young's modulus, E , and shear modulus, G , are

$$E = E_0(1 - f(\nu_0)\epsilon) \quad (23)$$

$$G = G_0(1 - g(\nu_0)\epsilon) \quad (54)$$

$$\text{where } f(\nu_0) = 16(10-3\nu_0)(1-\nu_0^2) / 45(2-\nu_0) \quad (24)$$

$$g(\nu_0) = 32(1-\nu_0)(5-\nu_0) / 45(2-\nu_0) \quad (55)$$

At the saturated state, the equations 23 and 54 can be rewritten as

$$E_{sat}/E_0 = 1 - f(\nu_0)\epsilon_{sat} \quad (56)$$

$$G_{sat}/G_0 = 1 - g(\nu_0)\epsilon_{sat} \quad (57)$$

where ϵ_{sat} - crack damage parameter at the saturated microcrack damage state.

Combining equations 52 and 56 gives

$$A/E_o = f(\nu_o) \epsilon_{sat} \quad (45)$$

Likewise, equations 53 and 57 yield

$$C/G_o = g(\nu_o) \epsilon_{sat} \quad (58)$$

Combining equations 45 and 58 gives

$$C = [g(\nu_o)/f(\nu_o)] \times [G_o/E_o] \times A \quad (59)$$

The relation between Young's modulus and shear modulus is given by

$$G_o = E_o / 2(1+\nu_o) \quad (60)$$

Substituting G_o/E_o from equations 60 into equation 59 yields

$$C = k(\nu_o) A \quad (61)$$

$$\text{where } k(\nu_o) = [(5-\nu_o) / (10-3\nu_o)(1+\nu_o)^2] \quad (62)$$

The proportionality constant $k(\nu_0) = 0.34$ for the alumina specimens included in this study (where Poisson's ratio ranged from 0.22 to 0.23). A least squares best fit of the experimental data for C versus A yielded an experimental value of 0.357 for $k(\nu_0)$, with a correlation coefficient of 0.98 (figure 27). Equation 61 which is based on Salganik's theoretical results has no free parameters yet it predicts well the observed linear relation between the elastic modulus damage parameters A and C . If Poisson's ratio, ν_0 and the damage parameter, A are known then the damage parameter, C can be predicted from equations 61 and 62.

4.2.5. ΔQ^{-1} as a function of crack density parameter, ϵ

From Salganik's equation 23 and empirical equations 19 and 20, the internal friction change can be expressed as (Section 4.1.5.)

$$\Delta Q^{-1} = C_1 [1 - (1 - C_2 \epsilon)^{C_3}] \quad (31)$$

where C_1 , C_2 and C_3 are constants. For a sufficiently small $C_2 \epsilon$, the term $(1 - C_2 \epsilon)^{C_3}$ in equation 31 can be written as $(1 - C_1 C_2 \epsilon)$ using the binomial expansion theorem. Then equation 31 can be approximated to linear equation, $\Delta Q^{-1} = D \epsilon$, where D is constant.

A least-square best fit of equation 31 yields 19.44×10^{-5} , 670, and 0.64 for C_1 , C_2 , and C_3 , respectively. In the section 4.1.5., the

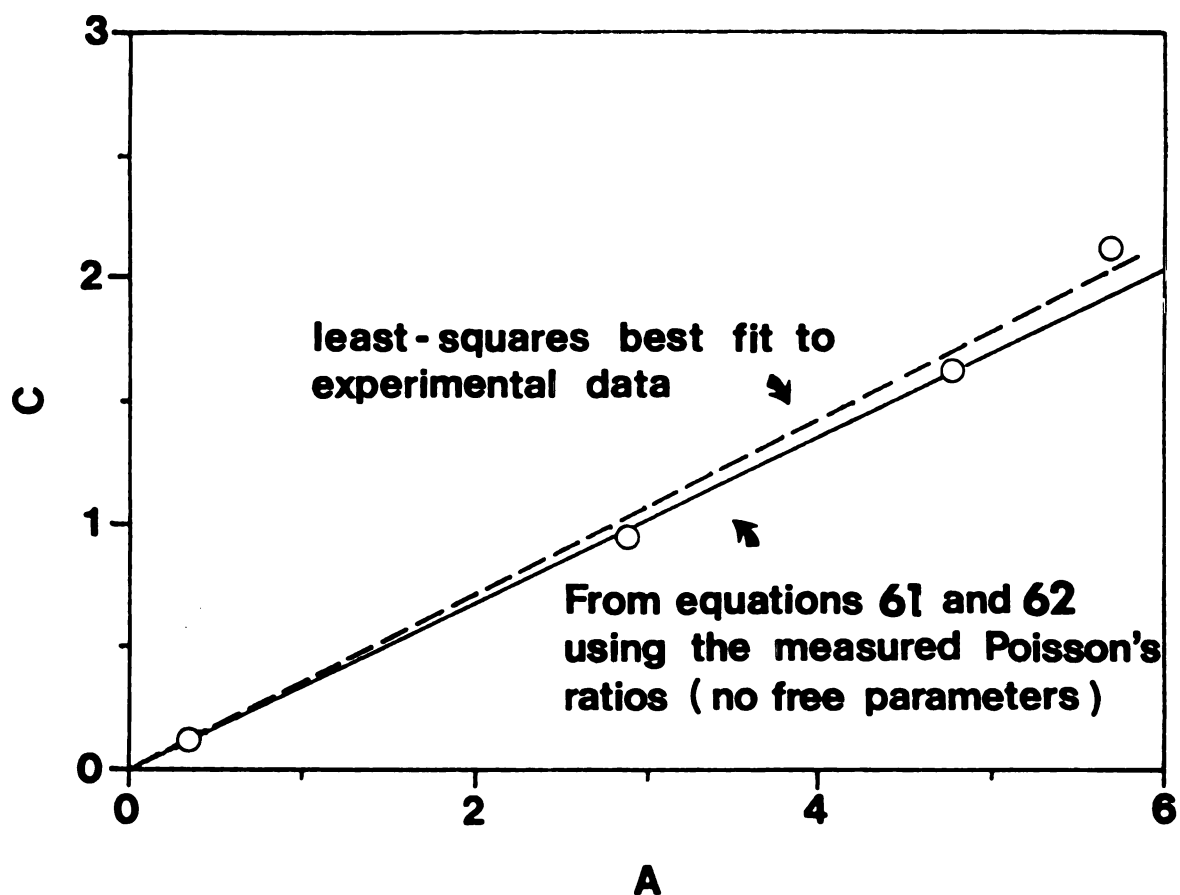


Figure 27. Relation between the measured elastic moduli damage saturation parameters, A and C. The dashed line corresponds to the least squares fit to the experimental data, while the solid curve is the relation predicted from equations 61 and 62 (using the measured values of Poisson's ratio to determine the proportionality constant $k(\nu_0)$).

value C_2 of the SiC whisker reinforced alumina composites was 390, which is relatively small and the linear approximation to the power-law form in fact a good approximation. However, the C_2 value of the polycrystalline alumina is higher than that of SiC whisker reinforced alumina composites. Thus the linear approximation of polycrystalline alumina data to the power-law form is not as good as that for the SiC whisker reinforced alumina composites (figure 28). In figure 28 the five ΔQ^{-1} and ΔE versus ϵ curves in figures 23 and 25 are effectively collapsed onto a single line of ΔQ^{-1} versus ϵ , crack damage parameter.

4.2.6. The Dependence of the Damage Saturation Parameters on the Thermal Shock Temperature Difference

The normalized saturation values, A/E_0 , C/G_0 and B/Q_0^{-1} show a power law relationship with nondimensional temperature difference, $\Delta T/T_r$, (figure 29) namely

$$A/E_0 = j_1(\Delta T/T_r)^{p^1} \quad (42)$$

$$B/Q_0^{-1} = j_2(\Delta T/T_r)^{p^2} \quad (43)$$

$$C/G_0 = j_3(\Delta T/T_r)^{p^3} \quad (63)$$

where T_r , a reference temperature, was taken as 293 degrees K (room temperature). ΔT , the temperature difference between the furnace temperature and the water bath temperature, was expressed in terms of

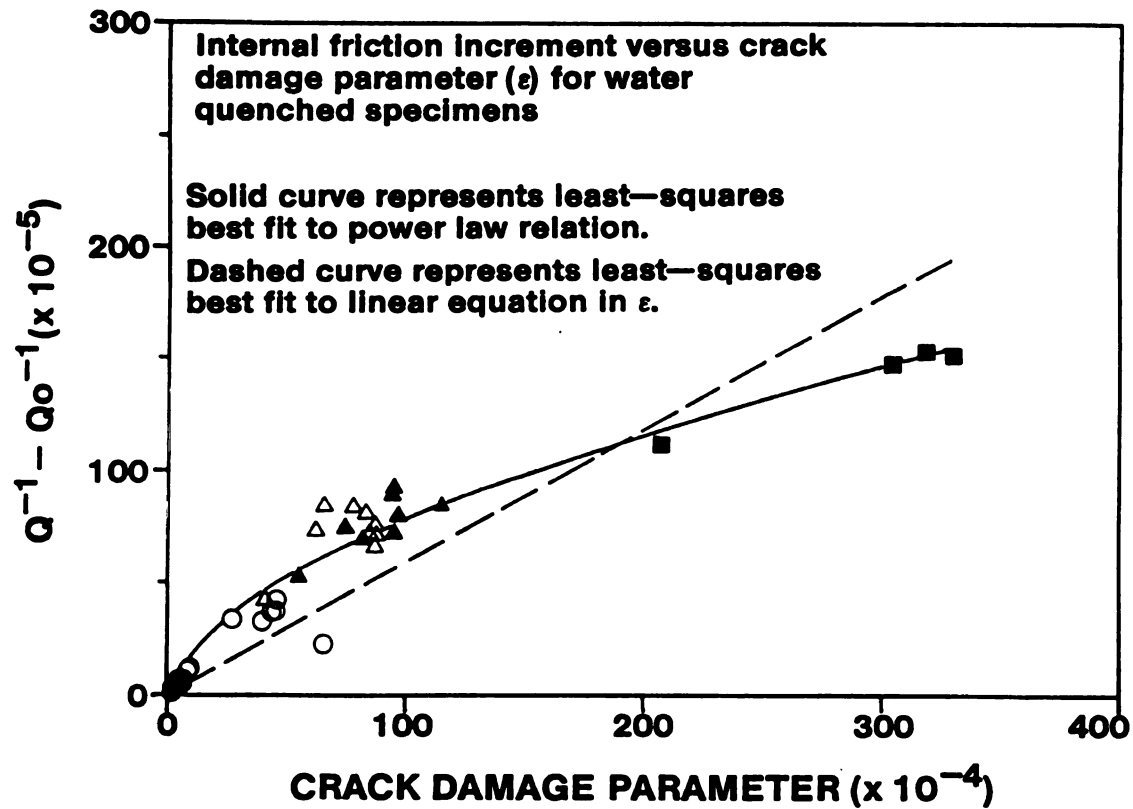


Figure 28. ΔQ^{-1} as a function of ϵ for the alumina specimens. Symbols indicate the ΔT value for a given measurement on thermally fatigued specimens. Symbols are the same in figures 23 and 25. Note that measurements with similar ΔT values are grouped together along the curve.

degrees K. A least-squares best fit yielded $j_1 = 0.030$, $j_2 = 14.79$ and $j_3 = 0.026$ for equations 37, 38 and 58. The exponents p^1 , p^2 and p^3 are respectively 11.8, 11.3 and 12.1.

Figure 29 compares the temperature dependence of the saturation damage levels for the unreinforced alumina (plotted using the open symbols) and the SiC whisker reinforced alumina composites (the closed symbols). In this log-log plot, the slopes of the curves correspond to p^1 , p^2 , and p^3 in equations 42, 43 and 63. Slopes of $\ln(A/E_0)$ and $\ln(B/Q_0^{-1})$ versus $\ln(\Delta T/T_r)$ are very similar for the SiC whisker reinforced alumina composites (5.9 and 5.7, respectively). Similarly, the slopes of $\ln(A/E_0)$, $\ln(B/Q_0^{-1})$, and $\ln(C/G_0)$ are very similar for the unreinforced alumina (11.8, 11.3, and 12.1, respectively). For both the composite and non-composite specimens, the thermal shock damage level as measured by either the elastic modulus or by internal friction, has a very similar functional dependence on the quenching temperature difference.

The exponents p^1 and p^2 of the polycrystalline alumina are almost double those of the SiC whisker reinforced alumina composites. From equation 42 and 43, we see that doubling the exponents p^1 and p^2 squares the value of A and B. Thus the polycrystalline alumina specimens are far more sensitive to thermal shock damage as a function of ΔT than are the SiC whisker reinforced alumina composites. Therefore, the whisker reinforcement is shown to dramatically improve the thermal shock damage resistance of polycrystalline alumina [57].

The relative changes in internal friction are much larger than

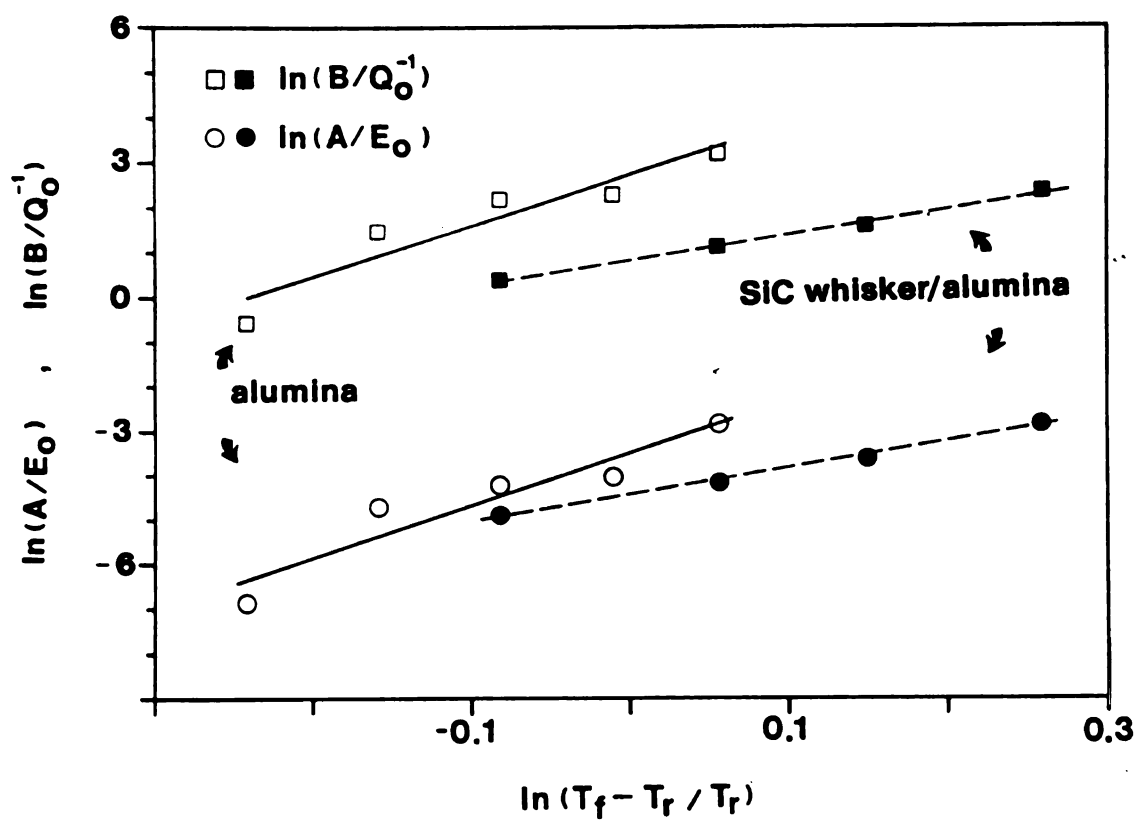


Figure 29. Logarithm of the normalized saturation damage parameters (A/E_0) and (B/Q_0^{-1}) versus logarithm of the normalized temperature.

the corresponding changes in elastic modulus, for a given level of thermal shock damage. For a given ΔT , and for a fixed reference temperature T_r , the ratio of the two normalized saturation parameters $(B/Q_0^{-1})/(A/E_0)$ is approximately equal to j_2/j_1 (see equations 42 and 43). The ratio j_2/j_1 (which is approximately 500) is a "sensitivity ratio" (Section 4.1.7.) since it represents, for a given level of thermal shock damage, the relative sensitivity (or relative change) in internal friction compared to the changes observed for Young's modulus. In comparison, the sensitivity ratio for the SiC whisker reinforced alumina composites was about 200. (Section 4.1.7.)

The sensitivity ratio j_3/j_1 is $(C/G_0)/(A/E_0)$ for a given ΔT represents a relative sensitivity in shear modulus compared to the changes observed for Young's modulus. Thus the saturation damage C can be expressed as

$$C = (G_0/E_0) \times (j_3/j_1) A \quad (64)$$

where for the unreinforced alumina $G_0/E_0 = 1/2(1+\nu_0)$ has the value of 0.41 and $j_3/j_1 = 0.88$. Thus equation 64 becomes $C = 0.36A$, which is essentially identical to the numerical results obtained in a different manner from equation 61 (section 4.2.4.).

In equations 42, 43 and 63, the power law in ΔT can be replaced by a power law in thermal stress, since the thermal shock induced stresses are linearly proportional to the temperature difference. Also the saturation damage parameters can be expressed in terms of

integrated crack growth (Section 4.1.8.). Thus, equations 42, 43 and 63 can be expressed as a power law function of stress. This power law relation in stress shows a fatigue relationship similar to that of metals.

4.2.7. Comparison of the Cyclic Thermal Shocks in Unreinforced Alumina and SiC Whisker Reinforced Alumina Composites

Water quenching of samples produces time varying tensile stresses at surface regions of the specimen. For simple geometry (a flat plate, for example), the maximum stresses can be evaluated using an equation such as [94]

$$\sigma_{\max} = f(N_{bi}) \times [E\alpha_{th}\Delta T/(1-\nu)] \quad (65)$$

where $\sigma_{\max}$ - maximum value of the actual surface stress

E - Young's modulus

α_{th} - the average thermal expansion coefficient

ΔT - temperature difference of thermal shock

ν - Poisson's ratio

$f(N_{bi})$ - function of Biot number (maximum nondimensional thermal stresses at the surface).

The Biot number, N_{bi} , is a nondimensional parameter given by

$$N_{bi} = hz/k \quad (66)$$

where h - surface heat transfer coefficient

z - the half thickness of plate

k - the thermal conductivity of plate.

Mason [94] described $f(N_{bi})$, the maximum nondimensional thermal stresses at the surface of the plate, as

$$f(N_{bi}) = 1/[1.5 + 3.25/N_{bi}] \quad (67)$$

for $0 < N_{bi} < 5$. Since $0 < N_{bi} < 5$ for the unreinforced polycrystalline alumina and SiC whisker reinforced alumina composites (Table 15) we may use equation 67. Thus, we can use equation 65 to compare the magnitudes of the maximum nondimensional thermal surface thermal stresses for the unreinforced alumina and for SiC whisker reinforced alumina composites. To simplify the calculations, we shall assume the same range for the heat transfer coefficient, h , for both the unreinforced alumina and SiC whisker reinforced alumina composites (Table 15). According to equation 65, the maximum thermally induced stresses for SiC whisker reinforced alumina composites were 2 - 3 times larger than those for the unreinforced alumina specimens largely because the alumina specimens used in this research were thinner than the SiC whisker reinforced alumina composites used in this study. (See Appendix E for the maximum thermal stress calculation of polycrystalline alumina and SiC whisker reinforced alumina composites.) The thicker specimens have corresponding higher values

Table 15. Thermal Properties of Polycrystalline Alumina and SiC Whisker Reinforced Alumina Including the Heat Transfer Coefficient, h , the Thermal Conductivity, k , and the Biot Modulus, N_{bi}

	h (cal sec ⁻¹ cm ⁻² C ⁻¹) (for water quenching)	z (cm)	k (cal sec ⁻¹ cm ⁻¹ C ⁻¹)	N_{bi}^*
Polycrystalline Alumina	0.1 - 1.0 [79]	0.05	0.077 [57]	0.07-0.65
SiC Whisker Reinforced Alumina	0.1 - 1.0 [79]	0.15	0.084 [57]**	0.18-1.79

* N_{bi} is calculated from equation 66.

** There was 20 volume percent of SiC whisker in the alumina matrix both in this study and in the SiC_w/alumina specimens studied in reference 57.

Table 16. Comparison of Damage Saturation Values A and B, Rate Constants α and β , and Relative Changes in Young's Modulus and Internal Friction for Unreinforced Alumina and SiC Whisker Reinforced Alumina Composites

ΔT	Material	A (GPa)	B ($\times 10^{-5}$)	α	β	A/E_o	B/Q_o^{-1}
270	alumina/SiC composite	3.00	20.38	0.042	0.061	0.007	1.2
	unreinforced alumina	4.77	75.44	0.499	0.970	0.0143	9.0
310	alumina/SiC composite	6.21	33.65	0.220	0.105	0.0172	3.18
	unreinforced alumina	19.42	150.88	1.005	1.338	0.0575	24.08

of N_{b1} , as shown in equation 66. Nevertheless, the damage (as determined by elasticity and internal friction measurements) in the unreinforced alumina specimens was much larger than the thermal shock induced in the alumina/SiC whisker composites (Table 15). For example, the relative changes in Young's modulus and internal friction for the unreinforced alumina at $\Delta T = 270$ degrees Celsius were 1.4 percent and 900 percent, respectively. In contrast, the Young's modulus and internal friction changes for SiC whisker reinforced alumina composites at the same ΔT were 0.7 percent and 120 percent. The rate constant and the saturation value of SiC whisker reinforced alumina composites are much smaller than those of unreinforced alumina, thus the thermal shock resistance of the SiC whisker reinforced alumina composites is superior to the unreinforced polycrystalline alumina (Table 16).

4.3 Thermal Fatigue Parameters and Their Relation to Material Parameter

Cyclic thermal shock of SiC whisker reinforced alumina composites (Section 4.1.), polycrystalline alumina (Section 4.2.), SiC fiber reinforced aluminosilicate composites [11], Macor machinable glass ceramics [12] and titanium diboride, resulted in monotonically increasing damage as characterized by modulus changes or internal friction changes. The thermal shock damage eventually saturated at a certain thermal shock cycles. The saturation damage level increased as the thermal shock temperature difference increased, according to the empirical relation

$$A/E_0 = j^1 * (\Delta T/T_r)^{p^1} \quad (42)$$

where A is saturated damage level, E_0 is undamaged specimen's Young's modulus, T_r is a reference temperature, and p^1 is empirical constant. p^1 values of approximately 12, 6, 4, 3 and 2.6 for monolithic alumina, SiC whisker reinforced alumina composites, titanium diboride, SiC fiber reinforced aluminosilicate composites and Macor machinable glass ceramics, respectively (figure 30, Table 17). The empirical exponent "p" may be related to the thermal shock resistance of the material (Section 4.2.6.). Larger "p" represents that the material is much more sensitive to the thermal shock damage as a function of ΔT .

Thus these empirical exponent "p" relate to thermal shock resistance parameters presented in the literature. In addition, the

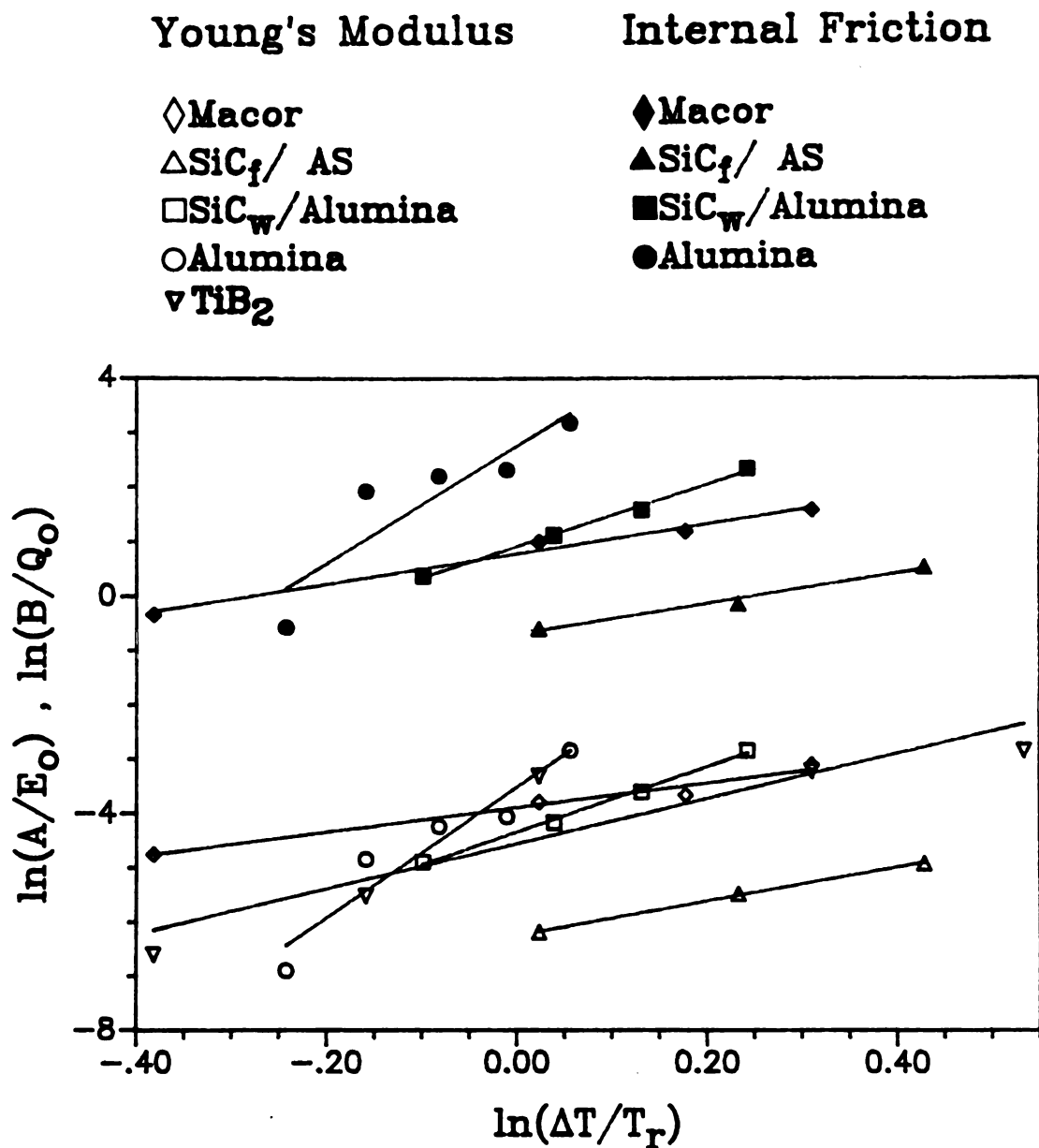


Figure 30. Logarithm of the normalized saturation damage parameters (A/E_0) and (B/Q_0) versus logarithm of the normalized temperature.

Table 17. Linear regression results of $\ln(A/E_o)$ and $\ln(B/Q_o^{-1})$ versus $\ln(\Delta T/T_r)$ (equation 42 and 43).

Material	p^1 (p^2)	intercept	cor. coefficient
Alumina	$p^1 = 11.81 \pm 2.19$ $p^2 = 11.38 \pm 3.23$	-3.54 ± 0.31 2.75 ± 0.42	0.954 0.901
Alumina/SiC _w	$p^1 = 5.97 \pm 0.31$ $p^2 = 5.71 \pm 0.27$	-4.36 ± 0.04 0.90 ± 0.04	0.998 0.998
TiB ₂	$p^1 = 4.15 \pm 1.08$	-4.58 ± 0.36	0.911
AS/SiC _f	$p^1 = 3.14 \pm 0.14$ $p^2 = 2.82 \pm 0.38$	-6.26 ± 0.04 -0.72 ± 0.10	0.999 0.991
Macor	$p^1 = 2.61 \pm 0.27$ $p^2 = 2.72 \pm 0.25$	-3.91 ± 0.07 0.76 ± 0.07	0.986 0.992

thermal fatigue parameters $A\alpha$ and $B\beta$ are linked with the fracture toughness or strength of the material.

4.3.1 Thermal fatigue results and their relation to thermal resistance parameters R''' , R'''' , and R_{st}

As a brittle material undergoes a rapid changes in temperature, sufficient stresses may develop for crack extension. Resistance to cracking is classically termed thermal shock resistance [79]. Thermoelastic analysis involving crack initiation yields thermal shock resistance parameters, R and R' [79] which are not appropriate for thermal fatigue. However stored elastic energy analysis relates the stability of pre-existing cracks to Griffith's energy balance, where the thermal shock resistance parameters include R''' , R'''' and R_{st} , which are

$$R''' = E/\sigma_f^2 (1-\nu) \quad (68)$$

$$R_{st} = K_{IC}(1-\nu^2)^{1/2}/E\alpha_{th} \text{ (in plane strain)} \quad (69)$$

$$\text{and } R'''' = K_{IC}^2(1+\nu)/\sigma_f^2 \text{ (in plane strain)} \quad (70)$$

where, σ_f - fracture strength

ν - Poisson's ratio

E - Young's modulus

α_{th} - thermal expansion coefficient

K_{IC} - critical stress intensity factor

R_{st} measures the resistance of material to thermal shock damage by quasistatic crack growth. R''' (which does not contain a fracture energy term) and R'''' (which does), are measures of the resistance of material to thermal shock damage by kinetic crack growth. The thermal shock resistance parameters R''' , R_{st} , and R'''' , the empirical exponents p_1 and p_2 as well as material properties E , σ_f , α_{th} , ν and K_{IC} are compared in table 18 for the four materials considered in this study.

An approximately hyperbolic relationship between p_1 and R'''' was observed (figure 31 and 32), such that

$$p_1^n R'''' = C = \text{constant} \quad (71)$$

If R'''' is expressed in units of microns, a least square best fit gives $n = 1.46 \pm 0.35$ and $C = 1720 \pm 183 \mu\text{m}$ with correlation coefficient 0.92. p_1 was used to obtain a hyperbolic relationship between thermal fatigue exponent and R'''' . Since p_1 and p_2 are approximately the same for each material tested (table 17), equation 71 would not change significantly if p_2 were used in place of p_1 . Although this preliminary empirical p_1 - R'''' relationship requires further study, it suggests a useful guideline to compare these two parameters, where p_1 and p_2 arise out of recent thermal fatigue studies by the present study and the classical R'''' parameter was

Table 18. Materials properties, thermal shock resistance parameters, and empirical exponent for the five materials included in this thermal fatigue study +

Property++	Alumina	SiC _w /Al ₂ O ₃	SiC _f /AS	Macor	TiB ₂
σ_f (MPa)	350* 320 [57]*	620 [57]*	800 [95]* 700 [96]**	90 * 103 [99]**	308 [97]*
ν	0.23	0.22	N.A.	0.26	0.1 - 0.15 [98]
α_{th} ($\times 10^{-6}$ C)	8.2 [57]	7.35 [57]	2.2 [96]	11 [99]	8.3 - 9.3 [98]
E (GPa)	335	405	135	63	552
K_{IC} (MPa m ^{1/2})	2.75 **** 2.5-5 [100]	6 **** 5.6-8.7 [101]	N.A.	2 **** 1.6-1.8 [102]	3.57**** 3.7-5.7 [98]
$R'''(1-\nu)$	2.73	1.054	0.211	7.78	5.81
$R_{st}/((1-\nu)^{1/2})$	1	2	57	3	0.7
$R''''/(1+\nu)$	61.7 (μm)	93.6 (μm)	450 (μm)	490 (μm)	134 (μm)
p1, p2	11.8, 11.3	6.0, 5.7	3.1, 2.8	2.7, 2.4	4.2
$A/E_0(\Delta T=300)$	0.040	0.014	0.002	0.022	0.036
$B/Q_0(\Delta T=300)$	19.33	2.55	0.55	2.12	N.A.

+ Materials properties and thermal shock resistance parameters are defined in equations 68-70. Empirical exponents are defined in equations 42 and 43.

++ All the material properties without parenthesis were measured in this study. Thermal shock resistance parameters were calculated from the measured materials properties.

* Four point bend test.

** Three point bend test.

*** Tensile strength.

**** K_{IC} was measured by indentation method.

N.A. - not available

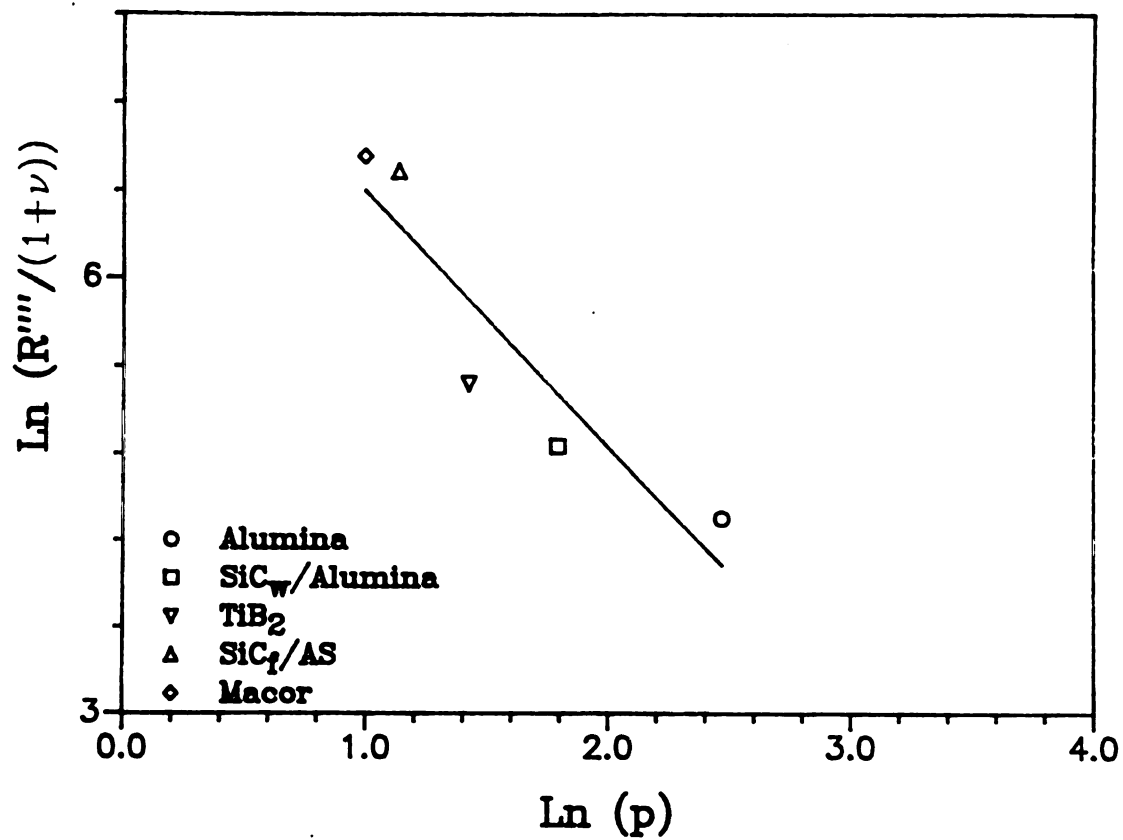


Figure 31. Logarithm of thermal shock damage parameter, $R''''/(1+\nu)$ versus Logarithm of thermal fatigue exponent, p^1 .

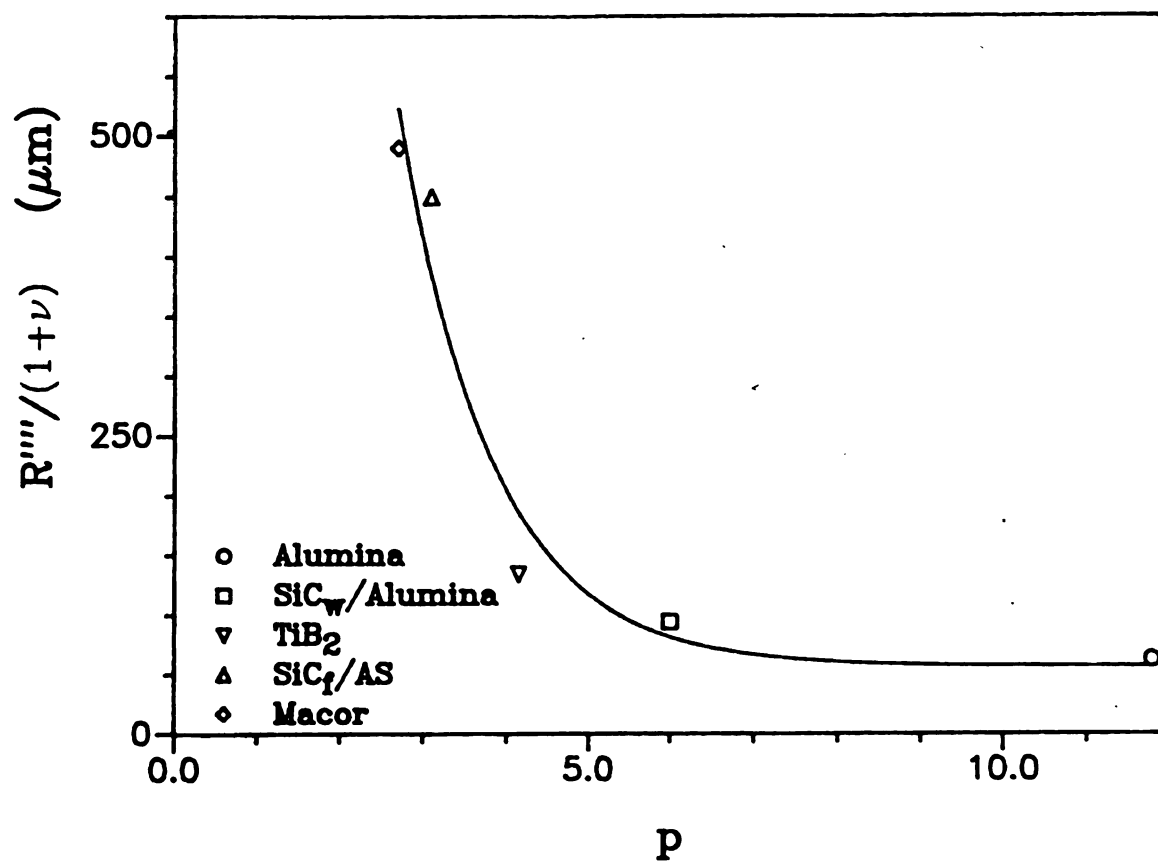


Figure 32. Thermal shock damage parameter, $R''''/(1+\nu)$ versus of thermal fatigue exponent, p^1 .

generated from "single quench and fracture" thermal shock testing protocols for ceramics [79]. In addition, since R'''' is a function of K_{IC} and σ_f , equation 66 indicates a link between the fatigue exponent p_1 and p_2 and material parameters such as fracture toughness.

4.3.2. The relation of A, B, α , and β to material properties

Thermal fatigue studies of SiC whisker reinforced alumina composites (Section 4.1.), unreinforced polycrystalline alumina (Section 4.2.), Macor machinable glass ceramics [12], SiC fiber reinforced aluminosilicate [11] and titanium diboride, found the rate constants α and β , and the damage saturation (A/E_0 and B/Q_0^{-1}) increased as the thermal shock severity increased. Since for small values of n , the cumulative number of thermal shocks, $A\alpha$ (or $B\beta$) is the slope of the E (or Q^{-1}) versus n curve, the product $A\alpha$ is more meaningful than A or α itself (figure 33). Since the normalized thermal shock damage saturation levels A and B are functions of ΔT (equations 42 and 43), and rate constants α and β are also functions of ΔT (Tables 9 and 13), we selected a fixed ΔT of 300 C for the plots of $A\alpha$ and $B\beta$ versus strength and fracture toughness (figure 34a, 34b and 34c).

Table 19 lists α , β , $A\alpha$ and $B\beta$ values at ΔT 300 degrees C for the five material used in this study. When $A\alpha = 0$ or $B\beta = 0$, then from the definition of α , β , A and B , we would not expect damage to be induced by repeated thermal cycling. Thus the intercept values on the

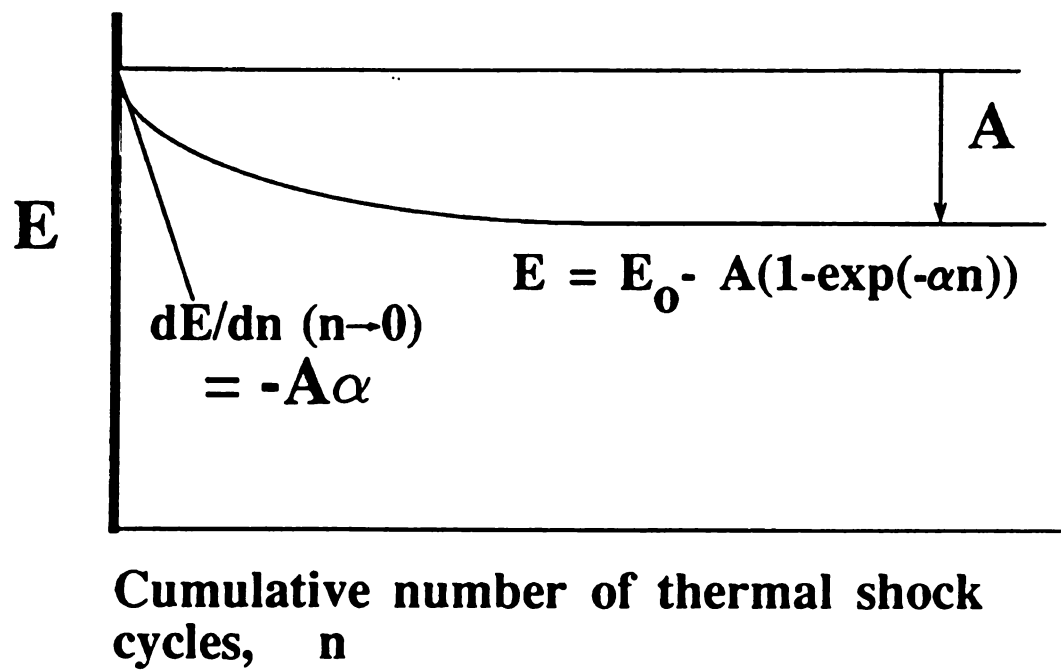


Figure 33. Schematic of products thermal fatigue constants.

Table 19. Rate Constants and Damage Saturation Parameters for the five materials included in this study at ΔT 300 degrees C

Parameters\Material	Alumina [*]	Al ₂ O ₃ /SiC _w ^{**}	LAS/SiC _f	Macor	TiB ₂
α	0.8	0.176	0.097	3.598	0.164
β	1.1	0.128	0.096	2.51	***
A α	4.16	0.948	0.028	5.06	3.37
B β	126.5	3.88	1.344	153.89	***

* These data were averaged from the data at ΔT 290 and 310 C.

** These data were linearly extrapolated from the data at ΔT 270 and 310 C.

*** B and β are not available since the data does not fit to equation 20.

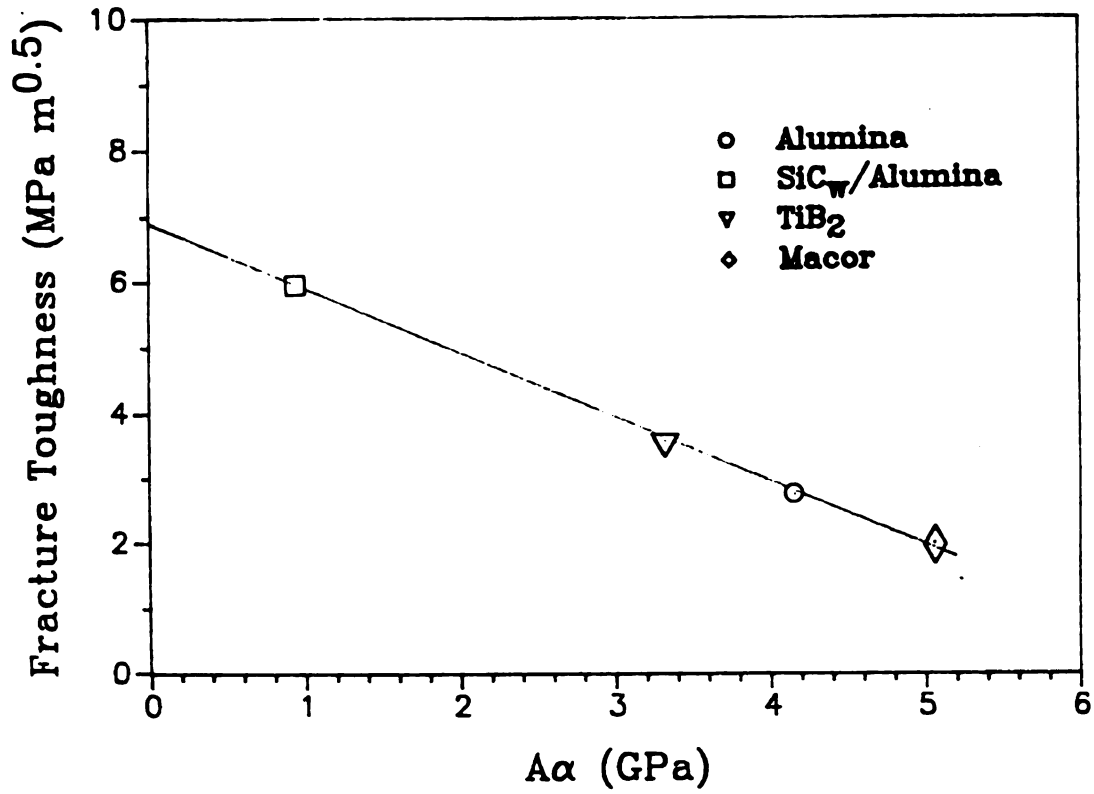


Figure 34a. Fracture toughness versus products thermal fatigue constants $A\alpha$

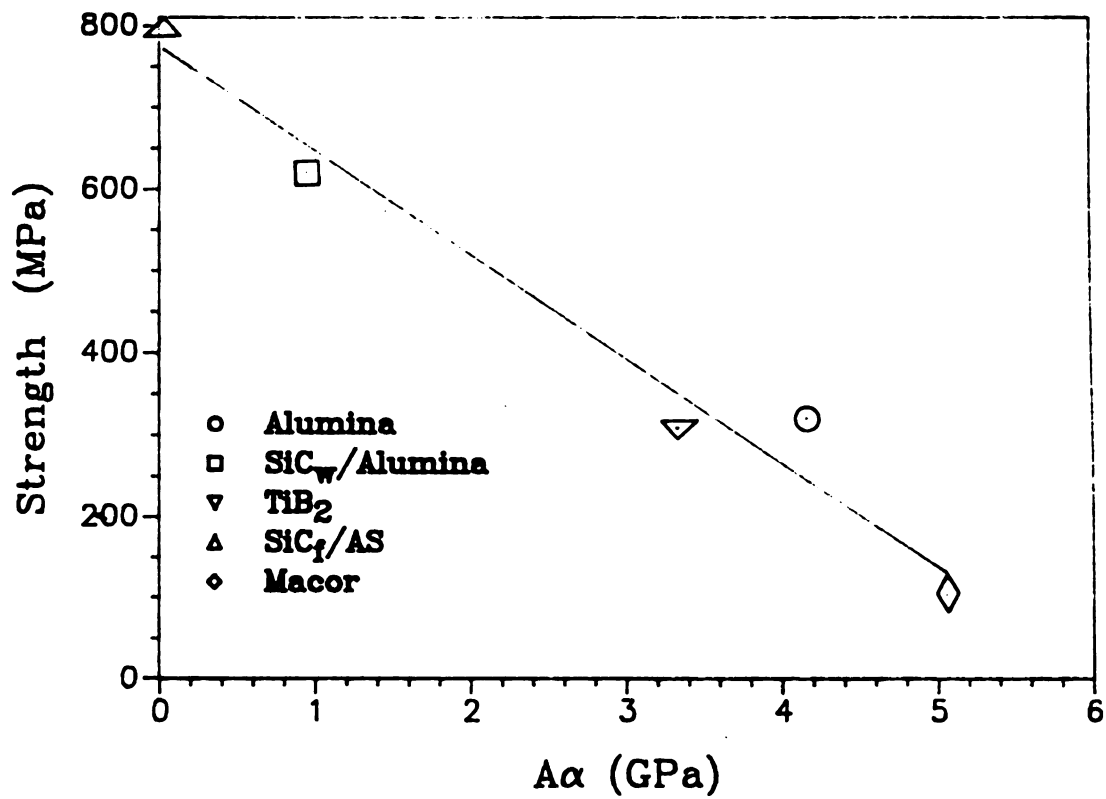


Figure 34b. Strength versus products thermal fatigue constants $A\alpha$

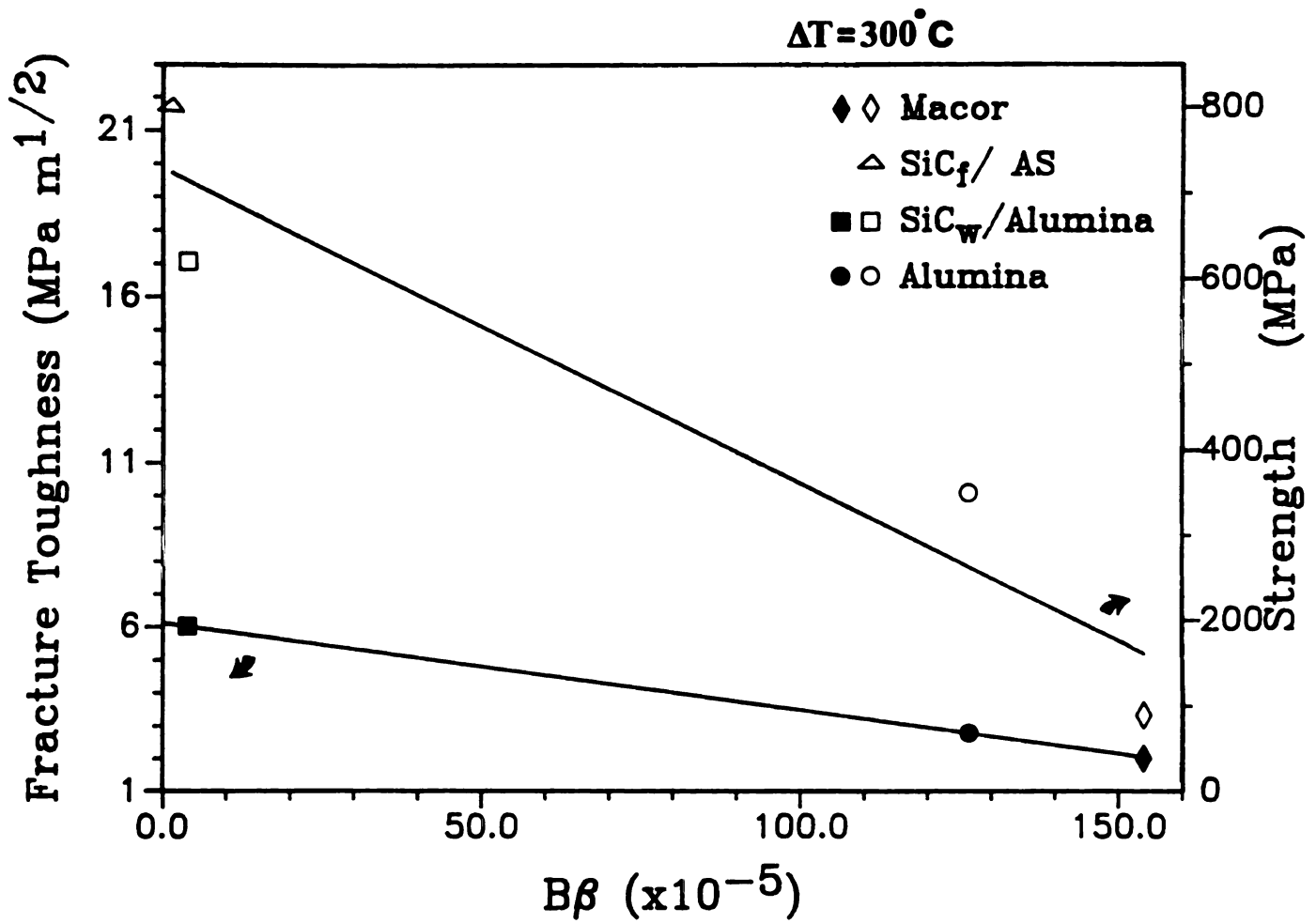


Figure 34c. Fracture toughness and strength versus products thermal fatigue constants $B\beta$.

fracture toughness axes (figures 34a, 34b and 34c) may represent a threshold fracture toughness level, above which the material would not suffer damage upon repeated thermal shock cycling at the corresponding ΔT . (Figures 34a and 34b, as noted above, were constructed from the $\Delta T=300$ C data for quenching into a room temperature water bath.) Thus, in addition to the fatigue exponent p_1 and p_2 (as discussed in the previous section), the thermal fatigue parameters $A\alpha$ and $B\beta$ are functions of the fracture toughness or strength of the material.

4.4. Effect of Quenching Media (Water, Silicone Oil and Liquid Nitrogen) on Cyclic Thermal Shock of Polycrystalline Alumina

Results of Cyclic water quench of polycrystalline alumina were described in the Section 4.2.. In addition to water, various quenching media can be used for the study of thermal shock. Since different quenching media may have different heat transfer characteristics, the transient temperature and stress distribution must be different even if the ΔT is same for each quench. Thus if the thermoelastic stresses are different, the thermal shock damage levels should be different. In this section, we discuss cyclic thermal shock for 20 degrees C silicone oil bath and for a liquid nitrogen bath.

The unshocked values of Young's modulus, shear modulus, internal friction, and Poisson's ratio are listed in table 20 for each of the specimens included for silicone oil quench and liquid nitrogen quench. As is typical for polycrystalline ceramics, the Young's modulus and shear modulus of all the unshocked specimens decreased with increasing porosity [82-86]. The polycrystalline alumina specimens exhibit a relatively limited range of porosity, which in turn implies limited effects due to porosity. Appendix D includes a discussion of modulus and internal friction dependencies on porosity.

4.4.1. Cyclic Thermal Shock of Polycrystalline Alumina Quenched into Liquid Nitrogen Bath

Polycrystalline alumina specimens were thermally shocked up to 40 cycles by quenching into liquid nitrogen bath at four different ΔT .

Table 20. Young's modulus, shear modulus, internal friction and Poisson's ratio of undamaged polycrystalline alumina specimens prior to quenching into either a silicone oil bath or a liquid nitrogen bath

Specimen Label\Properties		E (GPa)	G (GPa)	ν	Q^{-1} ($\times 10^{-5}$)
(Liq. N ₂ quench)	AN1	337.37	138.50	0.22	10.1
	AN2	338.76	139.39	0.22	9.7
	AN3	343.47	140.17	0.23	8.6
	AN4	338.02	139.21	0.21	8.9

(Silicone oil quench)	A01	346.39	142.58	0.21	5.1
	A02	347.42	142.60	0.21	7.4
	A03	350.01	141.63	0.23	7.3
	A04	342.98	142.75	0.20	9.8
	A05	346.65	142.24	0.22	6.9
	A06	361.36	148.10	0.22	4.9
	A07	349.19	142.68	0.22	7.1
	A08	347.41	141.83	0.22	7.5

The cumulative damage in terms of Young's modulus and internal friction versus the cumulative number of thermal shock cycles is shown in figure 35 and 36. Decrement in Young's modulus (for all ΔT values) and increment in internal friction (for two the highest ΔT) is fit by the same empirical equations developed in Section 4.1. and 4.2. for thermal shock of polycrystalline alumina and SiC whisker reinforced alumina composites into water quench bath, namely

$$E = E_0 - A (1 - \exp(-\alpha n)) \quad (19)$$

$$\text{and } Q^{-1} = Q_0^{-1} + B (1 - \exp(-\beta n)) \quad (20)$$

where A, B - damage saturation parameter

α, β - rate of decrement or increment

E, Q^{-1} - specimen's Young's modulus or internal friction as a function of thermal shock cycle at a fixed ΔT

E_0, Q_0^{-1} - undamaged specimen's Young's modulus or internal friction

n - cumulative number of thermal shock cycles at a fixed ΔT .

Table 21 lists the thermal fatigue parameters A, B, α and β obtained via non-linear regression from the liquid nitrogen quench data.

The changes of Young's modulus for $\Delta T = 220$ and 400 degrees C were relatively small, since the differences between the lowest measured Young's modulus and the undamaged Young's modulus, E_0 , were only 0.027 percent of E_0 for $\Delta T = 220$ degrees C and 0.023 percent of

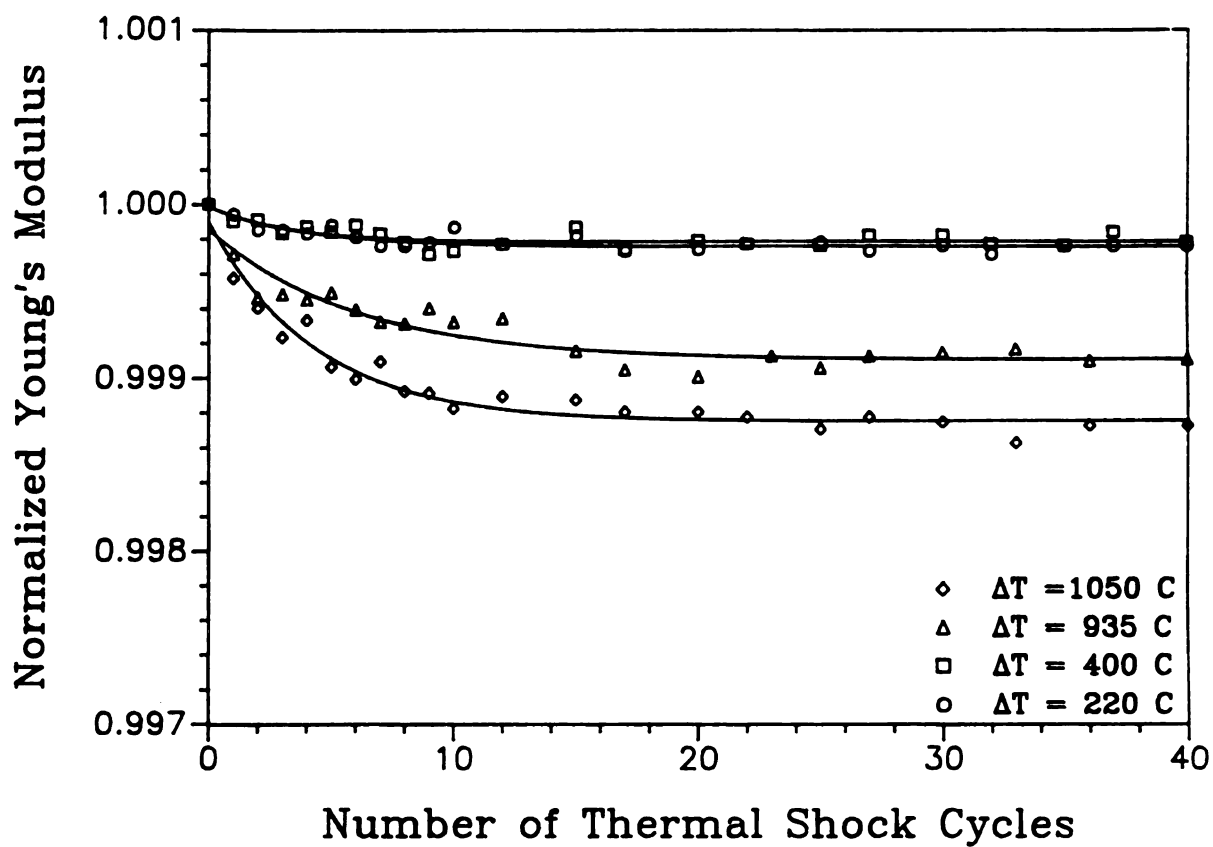


Figure 35. Young's modulus of polycrystalline alumina quenched into liquid nitrogen as a function of cumulative thermal shock cycles.

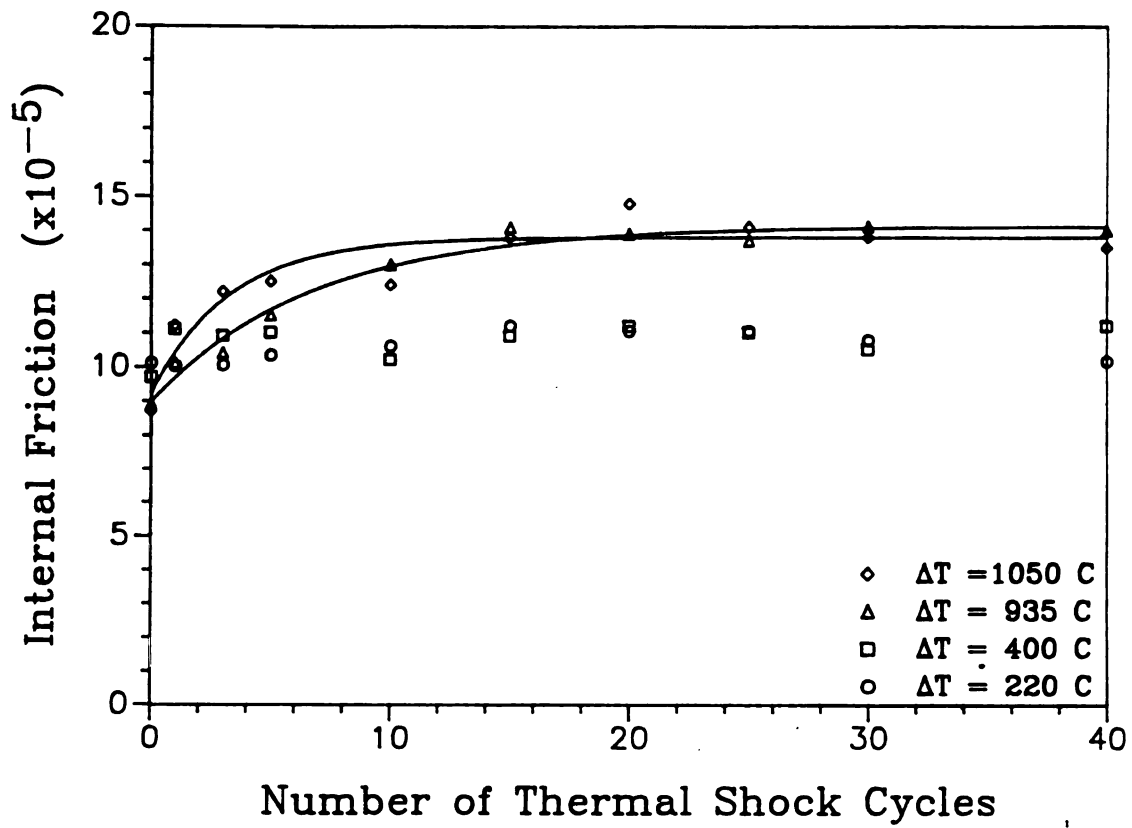


Figure 36. Internal friction of polycrystalline alumina quenched into liquid nitrogen as a function of cumulative thermal shock cycles.

Table 21. Thermal Fatigue Parameters of Polycrystalline Alumina
Quenched into Liquid Nitrogen Bath

Specimen Label	AN1	AN2	AN3	AN4
AT (C)	220	400	935	1050
E_o (GPa)	337.37	338.77	343.37	337.57
A (GPa)	0.076	0.068	0.257	0.388
$A/E_o \times 100$ (%)	0.023	0.020	0.075	0.115
α	0.258	0.313	0.169	0.232
Q_o^{-1} ($\times 10^{-5}$)	10.1	9.7	8.9	9.2
B ($\times 10^{-5}$)	*	*	5.16	4.6
$B/Q_o^{-1} \times 100$ (%)	*	*	58	50
β	*	*	0.147	0.303

*. The internal friction data was not well described by equation 20 (figure 36). The internal friction data fluctuated up and down by about 8 percent around a mean value of approximately 10.5×10^{-5} (figure 36).

E_0 for $\Delta T = 400$ degrees C, respectively. The uncertainty of the Young's modulus measurement* by present method in this study is about 0.02 percent of E_0 . Thus, 0.027 and 0.023 percent changes of E_0 were about the same magnitude of experimental uncertainty. Also, for $\Delta T = 220$ and 400 degrees C, internal friction changes do not fit to equation 20 but vary up and down. However small increase in internal friction was observed after 40 thermal shock cycles. The relatively small decrease in modulus and increase in internal friction for $\Delta T = 220$ and 400 degrees C may possibly be attributed to moisture that freezes on the surface of the quenched specimens when they were from the liquid nitrogen bath.**

For specimens AN3 ($\Delta T = 936$ degrees C) and AN4 ($\Delta T = 1050$ degrees C), a small amount of damage (relatively larger than the amount of uncertainty, see the table 20) was observed. Approximately same amount of damage was observed for the specimen shocked into water bath at a quench temperature difference of $\Delta T = 230$ degrees C (Section 4.2.). The relative changes in Young's modulus and internal friction for the water quenched specimen at $\Delta T = 230$ degrees C were 0.1 percent and 56 percent, respectively. The Young's modulus and internal friction changes for liquid nitrogen quench at $\Delta T = 1050$ degrees C were 0.115 percent and 50 percent.

* The uncertainty of Young's modulus is approximated $2(\Delta f_r / f_r) E$, where Δf_r is uncertainty of resonance frequency [6].

** The development of ice on the specimen surface was inevitable. Thus, the specimen was kept in a desiccator for one hour before the damage assessment to reduce the amount of moisture accumulated on the surface of the specimen.

4.2.2. Cyclic Thermal Shock of Polycrystalline Alumina into 20 degrees C Silicone Oil Bath

Polycrystalline alumina specimens were thermally shocked up to 40 cycles by quenching into 20 degrees C silicone oil bath. Young's modulus and internal friction changes as a function of cumulative number of thermal shock cycles are shown in figure 37 and 38.

For the 20 degrees C silicone oil quench included in this study, no saturation behavior was observed for the two most severe thermal shock conditions ($\Delta T = 578$ and 828 degrees C). For the highest thermal shock severity ($\Delta T = 828$ degrees C), specimen A07 broke into two pieces after first thermal shock cycle. Although specimen A07 fractured before the modulus measurement, the larger piece of the specimen was approximately rectangular in shape, allowing the Young's modulus to be measured approximately. For specimen A06 ($\Delta T = 578$ degrees C), a corner fragment fractured during the second thermal shock cycles, thus second thermal shock damage was not measurable.

Specimens A01 and A02 experiencing the two least severe thermal shock conditions ($\Delta T = 400$ and 500 degrees C) and specimen A06 one of the severe thermal shock conditions ($\Delta T = 575$ degrees C) displayed saturation behavior observed up to 40 thermal shock cycles. The decrement in Young's modulus and increment in internal friction can be described by empirical equations 19 and 20. Thermal fatigue parameters were listed in table 22. However, for specimen A01 ($\Delta T = 400$ C), the damage saturation A was about same as the magnitude of uncertainty.

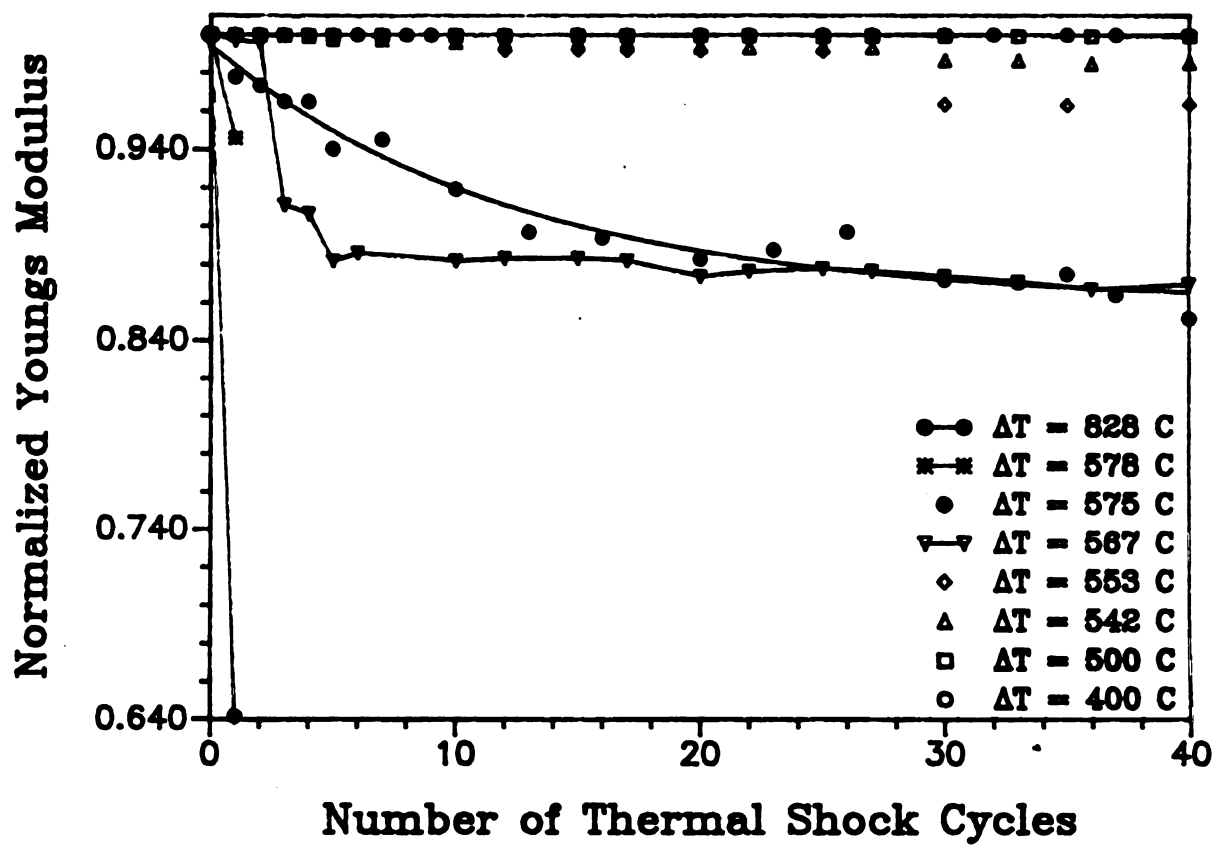


Figure 37a. Young's modulus of polycrystalline alumina quenched into silicone oil as a function of cumulative thermal shock cycles.

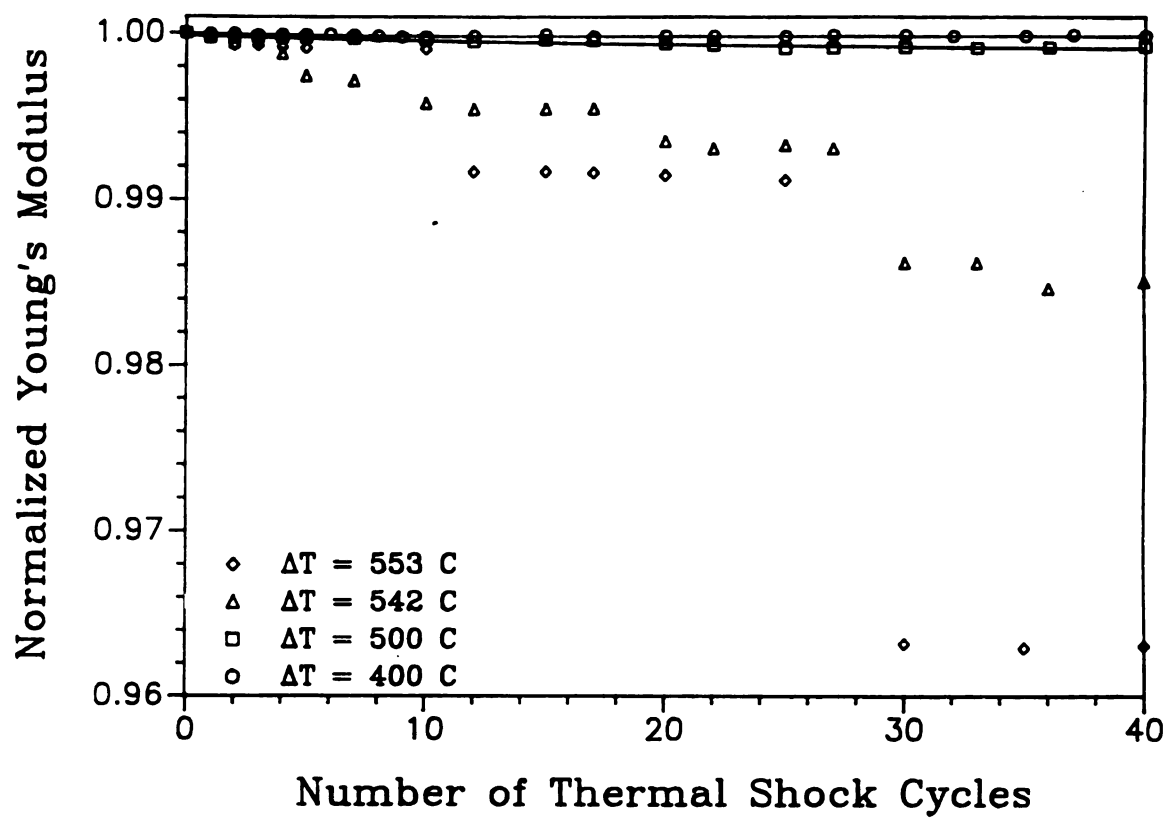


Figure 37b. Young's modulus of polycrystalline alumina quenched into silicone oil as a function of cumulative thermal shock cycles. (subset of figure 37a.)

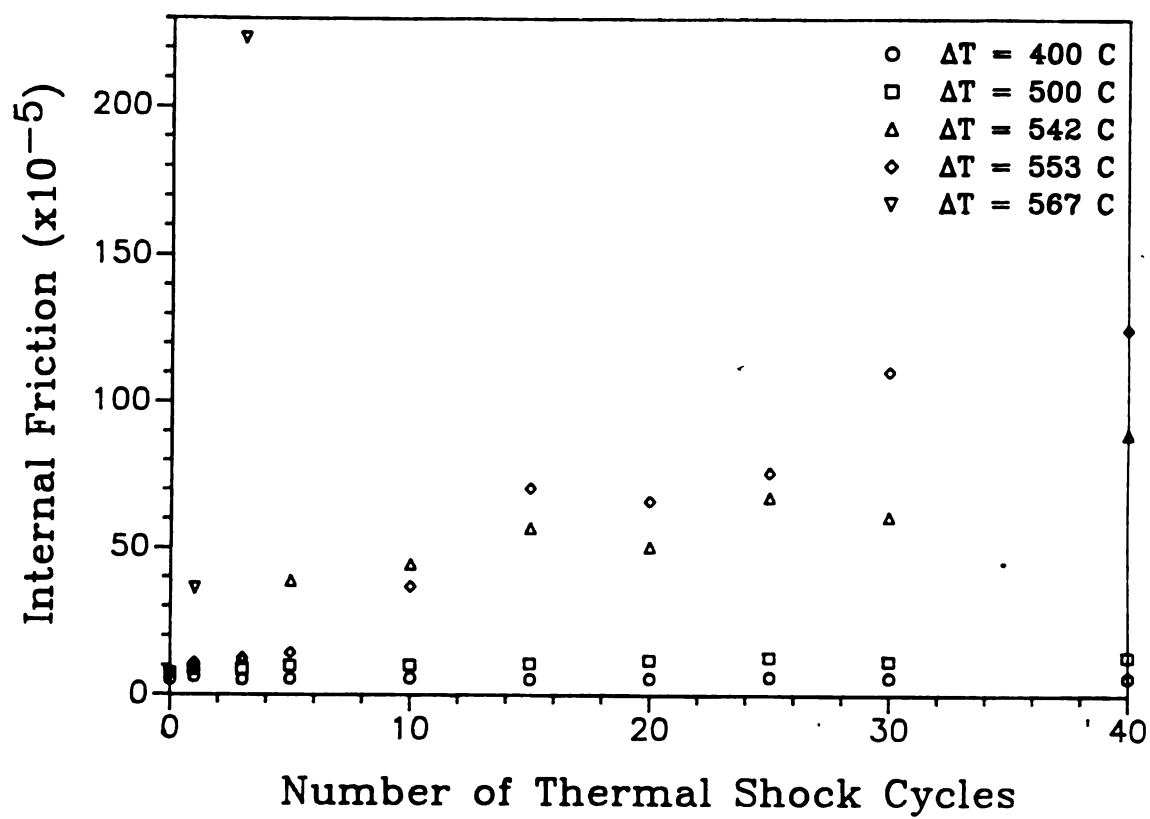


Figure 38. Internal friction of polycrystalline alumina quenched into silicone oil as a function of cumulative thermal shock cycles.

Table 22. Thermal Fatigue Parameters of Polycrystalline Alumina
Quenched into 20 degrees C Silicon Oil Bath

Specimen Label	A01	A02	A06
ΔT (C)	400	500	575
E_o (GPa)	346.38	347.41	359.2
A (GPa)	0.056	0.392	48.13
$A/E_o \times 100$ (%)	0.016	0.113	13.4
α	0.242	0.028	0.08
Q_o^{-1} ($\times 10^{-5}$)	5.1	7.7	4.9
B ($\times 10^{-5}$)	*	5.5	**
$B/Q_o^{-1} \times 100$ (%)	*	71	**
β	*	0.057	**

*. Since negative value of β was obtained, B and β are meaningless.

**Internal friction data were not able to be measured due to extremely low number of count.

For temperature differences of 542, 553 and 567 C, a "step-type" decrement in Young's modulus was observed. This may imply a step-like nature for crack propagation behavior. Specimen A05 ($\Delta T = 567$ C) showed small changes up to second thermal shock cycle. A abrupt decrease in modulus occurred after the third thermal shock cycle. Specimen A05 then showed damage saturation behavior after 6th cumulative thermal shock cycle. The "step-type" decrement in modulus may be attributed to the longer duration time in transient thermal stress (Section 4.4.6.).

4.4.3. Heat Transfer Coefficients for Various Quenching Media

In this study, we estimated the heat transfer coefficient of polycrystalline alumina for water quench, silicone oil quench and liquid nitrogen quench. After the specimens were uniformly heated to a preselected temperature, they were quenched into room temperature water or into room temperature silicone oil or liquid nitrogen bath.

The cooling rate of the silicone oil quench is far faster than that of liquid nitrogen quench and far slower than that of water quench (figures 13). With these data, the heat transfer coefficients for each quenching medium were estimated using a parameter estimation technique (Section 3.8.). The heat transfer coefficient versus surface temperature for water quench, silicone oil quench and liquid nitrogen quench shows that the maximum heat transfer coefficient for the oil bath was an order of magnitude lower than that for water bath

and an order of magnitude higher than that for liquid nitrogen bath (figures 39, 40 and 41).

The temperature dependent functional form of specific heat for polycrystalline alumina^{*}, obtained via a least squares best fit of available data [58], can be expressed as

$$C_p \text{ [J/(kg C)]} = c_1 + c_2 T + c_3 T^2 + c_4 T^3 \quad (72)$$

where C_p = specific heat

T = temperature in degrees C

c_1, c_2, c_3, c_4 = constants obtained linear regression

The best fit results for the constants c_1, c_2, c_3 and c_4 were 791.4 J/(kg C), 1.407 J/(kg C²), -1.634×10^{-3} J/(kg C³) and 7.029×10^{-7} J/(kg C⁴), respectively. The correlation coefficient was 0.995.

Table 23 lists the numerical results of the polynomial coefficients β_i that were obtained from the regression of the time versus temperature data (equation 5).

The heat transfer coefficient values obtained in this study for alumina are in broad agreement with the heat transfer coefficient values calculated for water quenched alumina by Becher and coworkers [103, 104] and for water and silicone oil quenched alumina by Hasselman and coworkers [15] (figures 42a and 42b).

The surface heat transfer coefficient, h , varies significantly

^{*} Appendix E includes the specific heat data for titanium diboride, SiC whisker reinforced alumina composite and Macor.

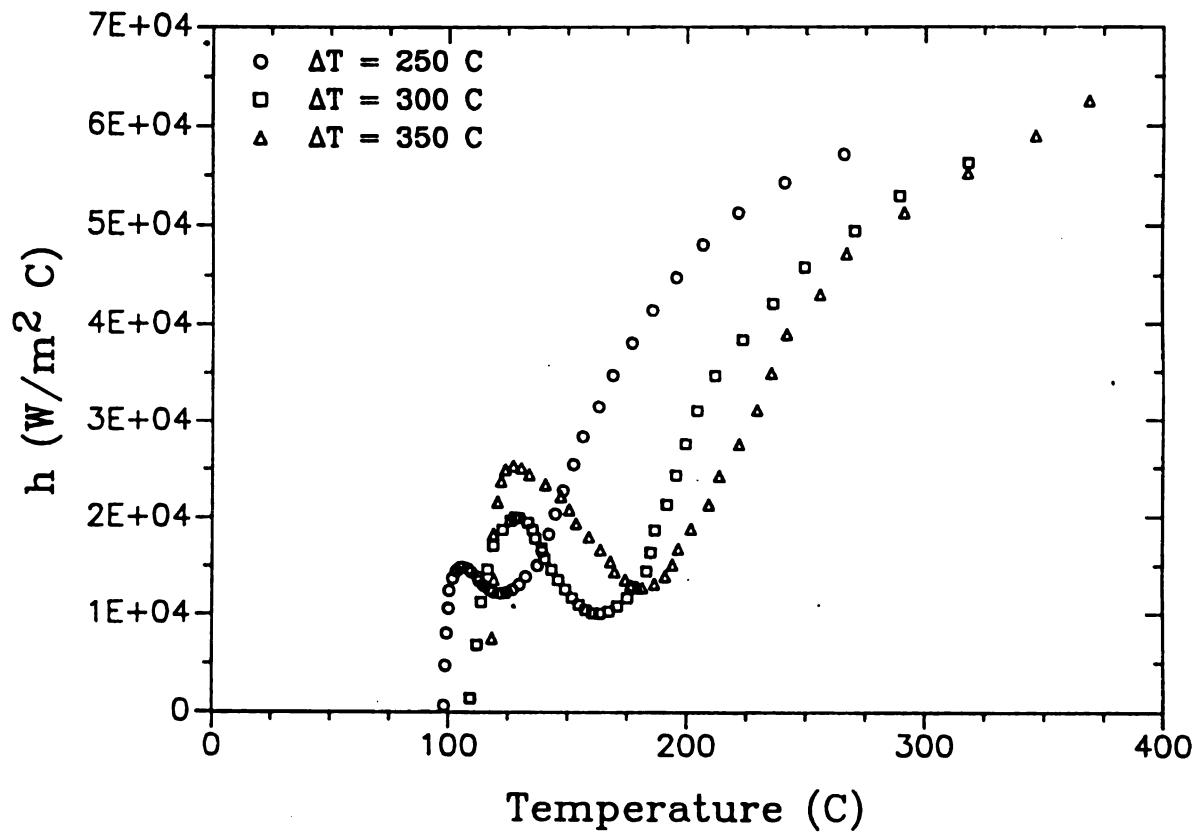


Figure 39. The estimated surface heat transfer coefficient versus temperature calculated from the experimentally obtained thermocouple voltage versus cooling time for water quenched alumina.

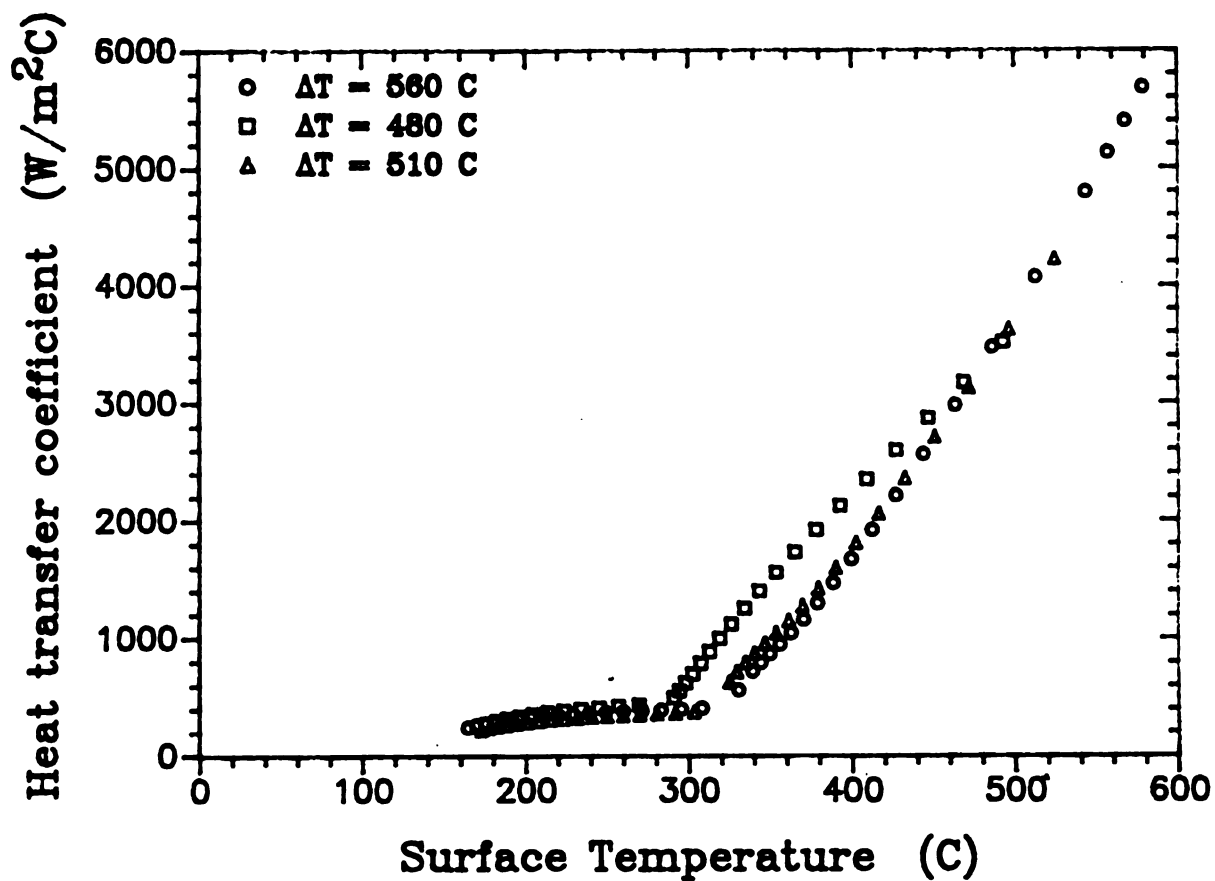


Figure 40. The estimated surface heat transfer coefficient versus temperature calculated from the experimentally obtained thermocouple voltage versus cooling time for silicone oil quenched alumina.

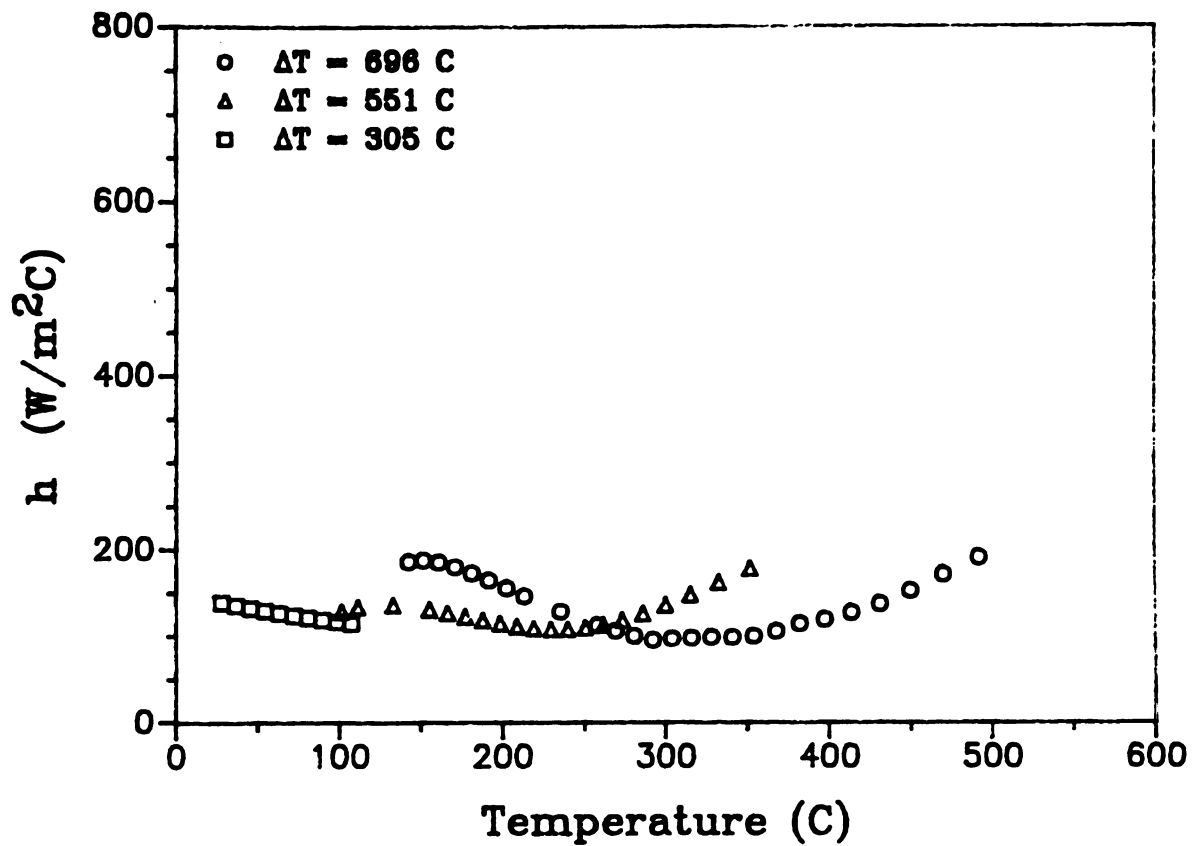


Figure 41. The estimated surface heat transfer coefficient versus temperature calculated from the experimentally obtained thermocouple voltage versus cooling time for liquid nitrogen quenched alumina.

Table 23. The Polynomial Series for Measured Temperature Changes Versus Time for Polycrystalline Alumina

$$\hat{T} = \beta_1 + \beta_2(t/\Delta t) + \beta_3(t/\Delta t)^2 + \beta_4(t/\Delta t)^3 + \beta_5(t/\Delta t)^4$$

Quench Media	ΔT ($^{\circ}\text{C}$)	β_1 ($^{\circ}\text{C}$)	β_2 ($^{\circ}\text{C}$)	β_3 ($^{\circ}\text{C}$)	β_4 ($^{\circ}\text{C}$)	β_5 ($^{\circ}\text{C}$)	Data point taken	Cor. Coeff.
Water	250	258.60	-18.88	1.09	-0.03	3.24×10^{-4}	37	0.997
(2.5 ⁺ msec)	300	309.34	-21.90	1.33	-0.04	3.91×10^{-4}	40	0.994
	350	365.74	-28.33	1.76	-0.05	5.53×10^{-4}	39	0.994

Oil ⁺⁺	480	503.31	-31.27	1.09	0.09	-4.19×10^{-3}	19	0.991
		284.14	-3.31	0.06	-0.001	7.3×10^{-6}	12	0.994
(20 ⁺ msec)	510	535.10	-33.24	0.81	0.11	-4.32×10^{-3}	17	0.996
		315.24	-2.51	0.01	-0.0001	7.2×10^{-7}	17	0.991
	560	586.05	-35.91	0.36	0.19	-8.17×10^{-3}	20	0.991
		323.69	-3.71	0.08	-0.0075	1.2×10^{-5}	17	0.972

Liquid nitrogen	696	491.85	-22.86	0.95	-0.036	5.21×10^{-4}	26	0.996
(400 ⁺ msec)	551	351.69	-20.58	1.29	-0.069	1.30×10^{-3}	20	0.997
	305	107.2	-8.83	0.34	-0.037	1.49×10^{-4}	10	0.998

+ The time specified in parenthesis represents the time step Δt .

++ The entire data is not well described by a single 4th order polynomial. Thus, we separated the data into two sets. This separation leads to a break in heat transfer coefficient (figure 40).

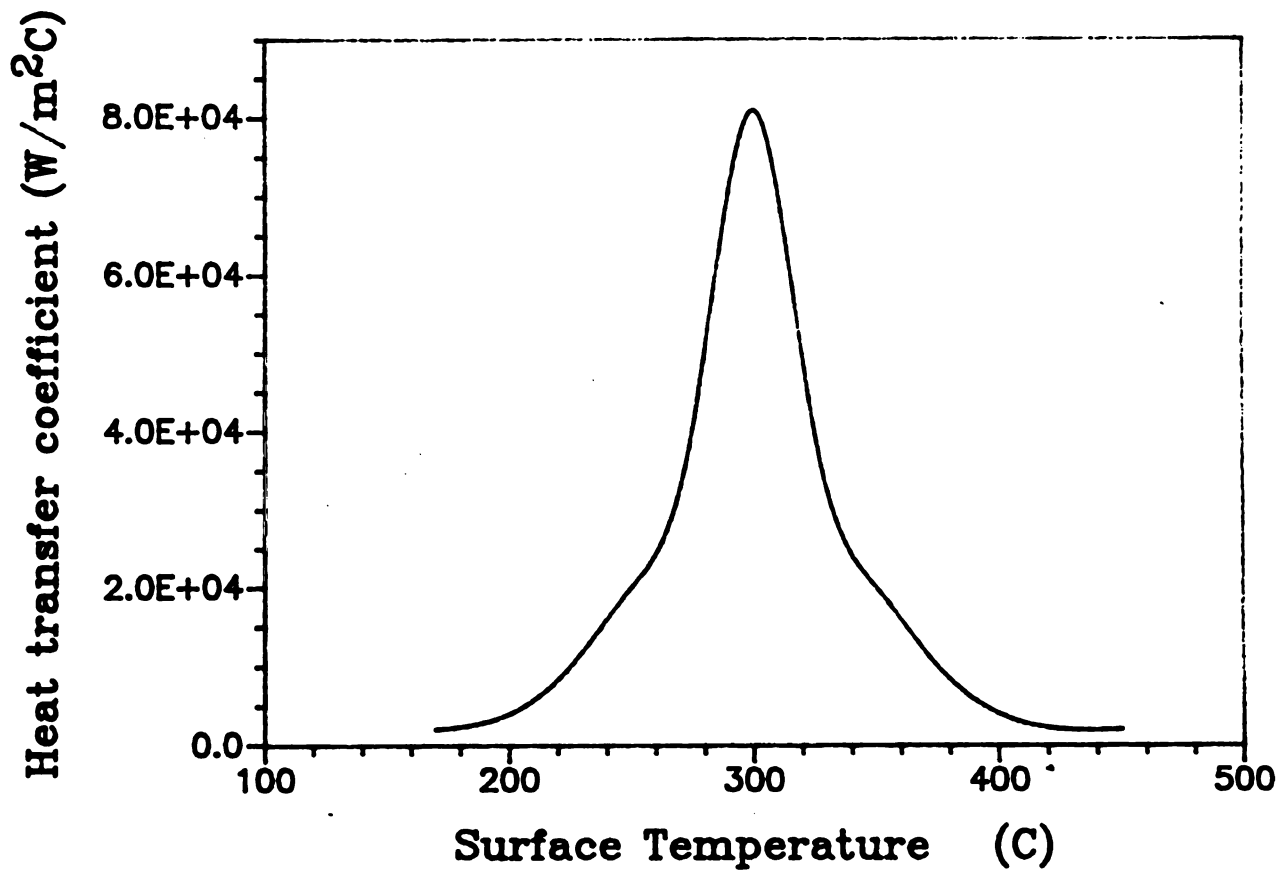


Figure 42a. Surface heat transfer coefficient of alumina for water quench inferred from the experimental ΔT_c values. (After Becher [103])

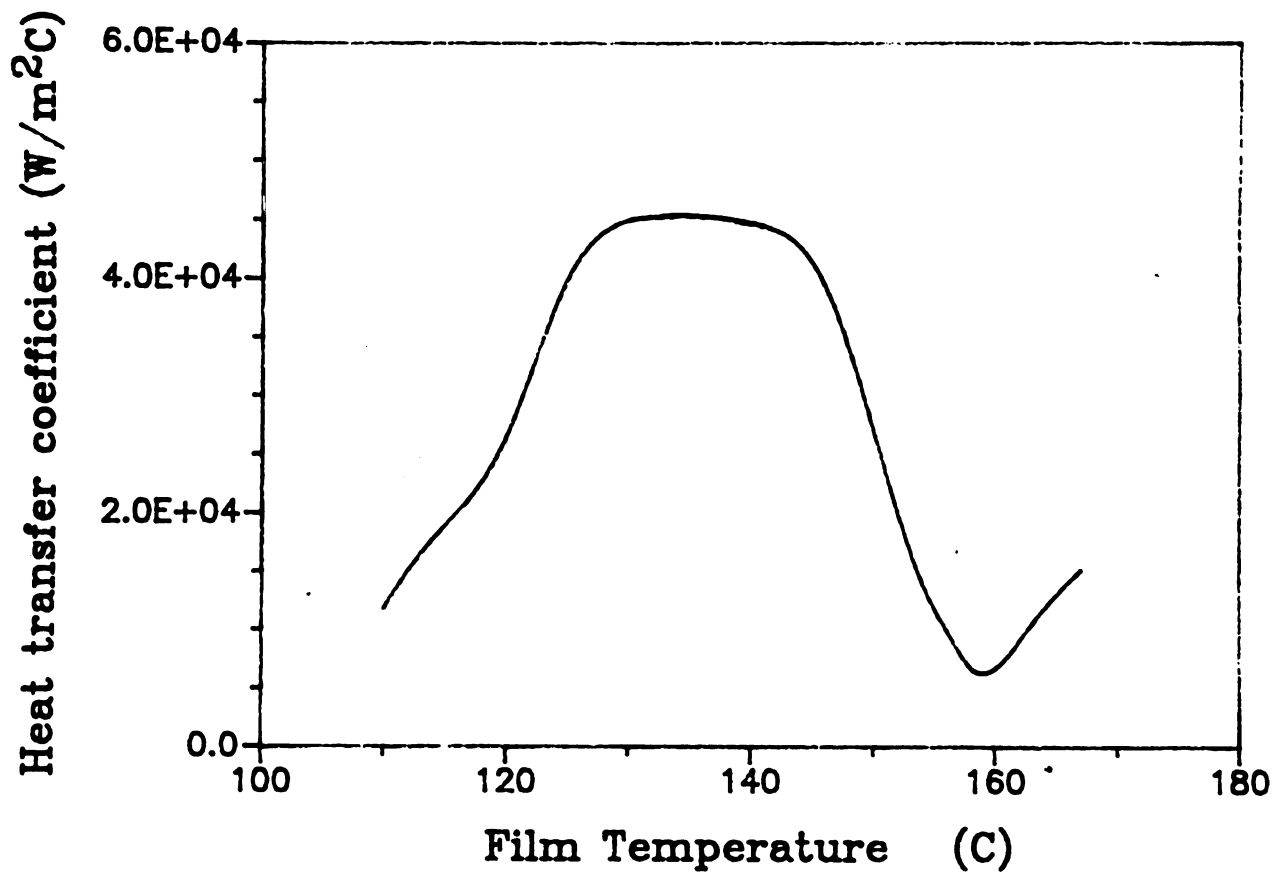


Figure 42b. Surface heat transfer coefficient of alumina for water quench inferred from the experimental ΔT_c values. (After Hasselman et al. [15])

with instantaneous temperature difference between specimen surface and fluid medium. When the quench medium boils, the heat transfer is strongly affected by the nucleation of bubbles which increases the heat transfer. The film formation at very high temperatures suppresses the transfer of heat (figure 43) since the thermal conductivity of a gas is very low [79].

Physically, the boiling nucleation regime involves the formation and movement of bubbles on the specimen surface [105, 106]. The transition regime involves an unstable film which, as temperature rises, eventually stabilizes on the surface of the hot solid. The maximum in h corresponds to the rapid creation of bubbles and their departure from the heated solid [105, 106]. Heat transfer in the boiling nucleation and film formations regimes depend on such parameters as the latent heat of vaporization of liquid, the liquid viscosity, the surface tension of the liquid vapor interface, the number density of boiling nucleation sites on the solid's surface, and empirically determined constants depending on the particular solid-liquid interface [105, 106]. Since boiling heat transfer is very complex and is only semi-empirically understood, a single equation is not available which describes h over all the temperature regimes.

Stolz et al. [107] devised a numerical technique to obtain heat transfer coefficient. A 2.54 cm radius silver sphere was heated to 870 C and quenched into 43 C oil bath. Stoltz et al. measured interior (0.254 cm from surface) temperature versus time and found the surface heat flux versus time. By means of this technique heat transfer coefficient for quenching oil were evaluated as a function of

FC = Free Convection Boiling

N = Nucleate Boiling

TR = Transition

F = Film Boiling

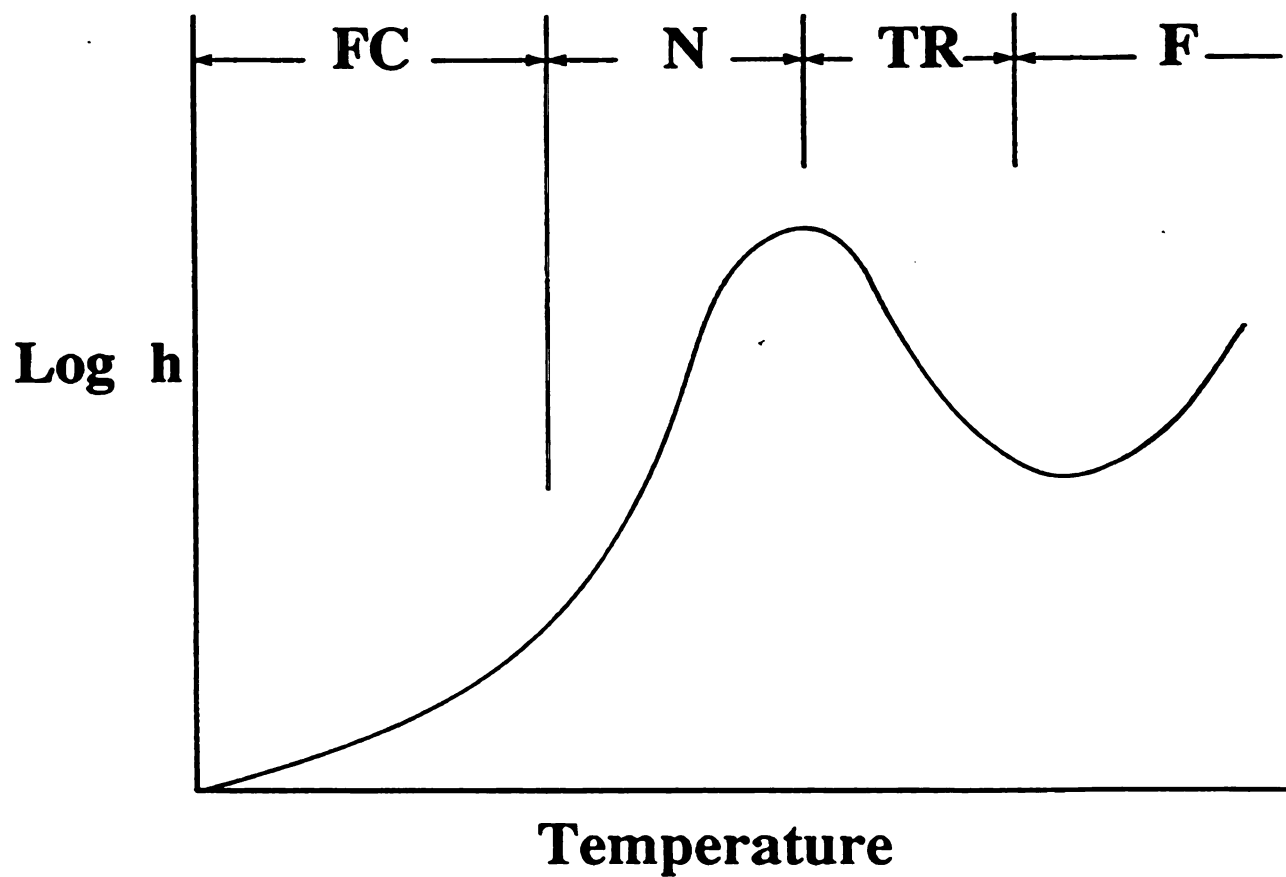


Figure 43. Schematic of heat transfer coefficient versus temperature for boiling fluid (After [106]).

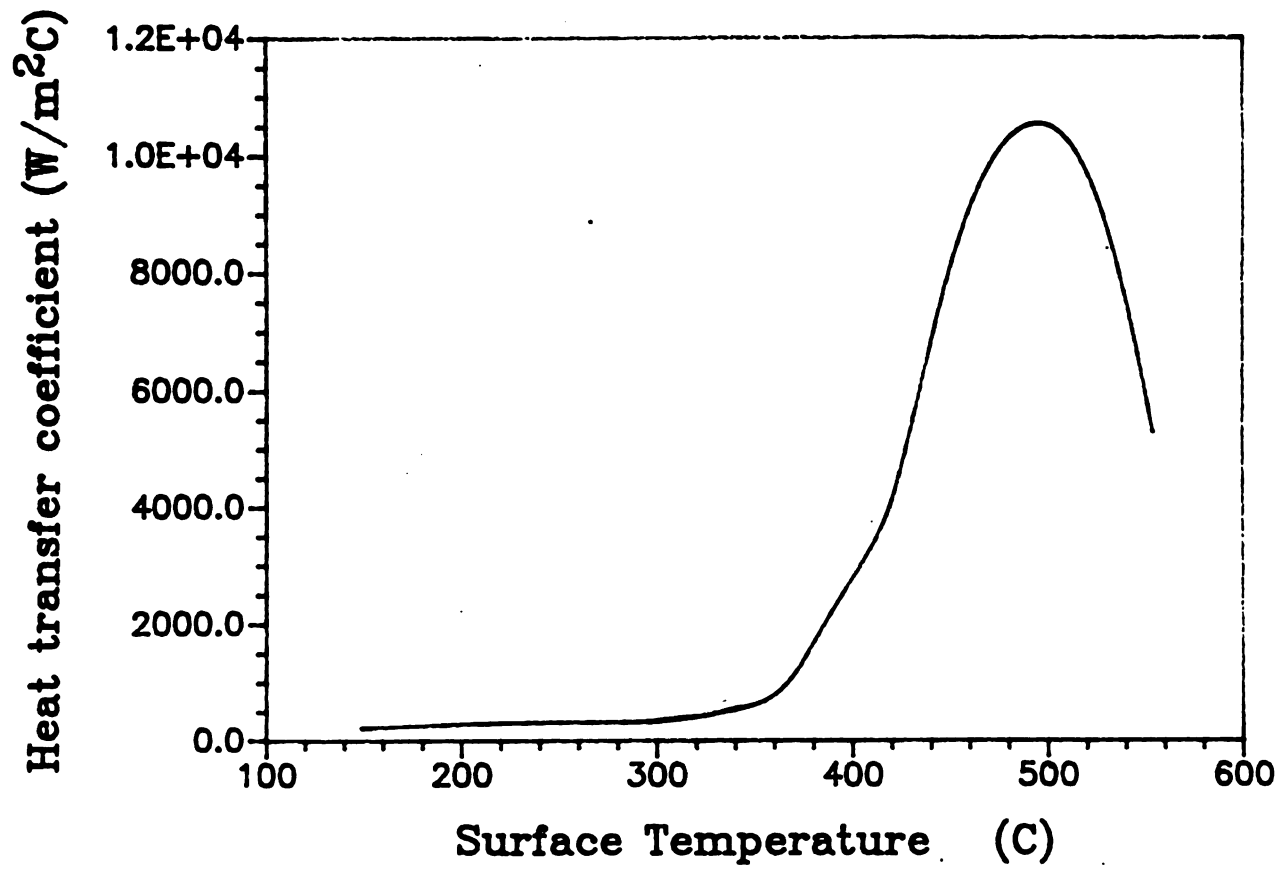


Figure 44a. Surface heat transfer coefficient of a silver sphere for a oil quench (After Stoltz et al. [107]).

temperature up to about 870 C (figure 44a). The maximum heat transfer coefficient (about $11000 \text{ W/m}^2 \text{ C}$) occurs in the surface temperature range of 520-530 C, and the value of heat transfer coefficient changes by well over an order of magnitude as the temperature ranges (figure 44a). Hachisu et al. [108] obtained similar values for the temperature dependence of heat transfer coefficient for a steel cylinder quenched into oil. The surface temperature was measured by using a thermocouple made of nickel and silver. The heat transfer coefficient was evaluated from an unsteady heat transfer analysis of the resulting cooling curve. Hachisu et al. found the maximum heat transfer coefficient ($3000 \text{ W/m}^2 \text{ C}$) in the surface temperature range 500-600 C (figure 44a).

In this study, the heat transfer coefficient of alumina quenched into silicone oil increased as surface temperature increased up to 600 degrees C (figure 40). The maximum heat transfer coefficient in this study was about $6000 \text{ W/m}^2 \text{ C}$ which was lower than the value of $11000 \text{ W/m}^2 \text{ C}$ found by Stolz et al. and higher than the value of $3000 \text{ W/m}^2 \text{ C}$ found by Hachisu et al.* According to our data, the heat transfer mechanism for oil quench seems to change from pure convection to convection with the nucleation of boiling about 350 C (see figures 40 and 43). This transition point was observed in the Stoltz's data (about 380 C) and Hachisu' data (about 200 C) (figures 44a and 44b).

 * Stolz and Hachisu didn't specify the chemical composition of the quenching oil or any of the oil properties. The heat transfer coefficient depends on the fluid properties such as the coefficient of thermal expansion, the density, the specific heat, the thermal conductivity as well as viscosity [105, 106].

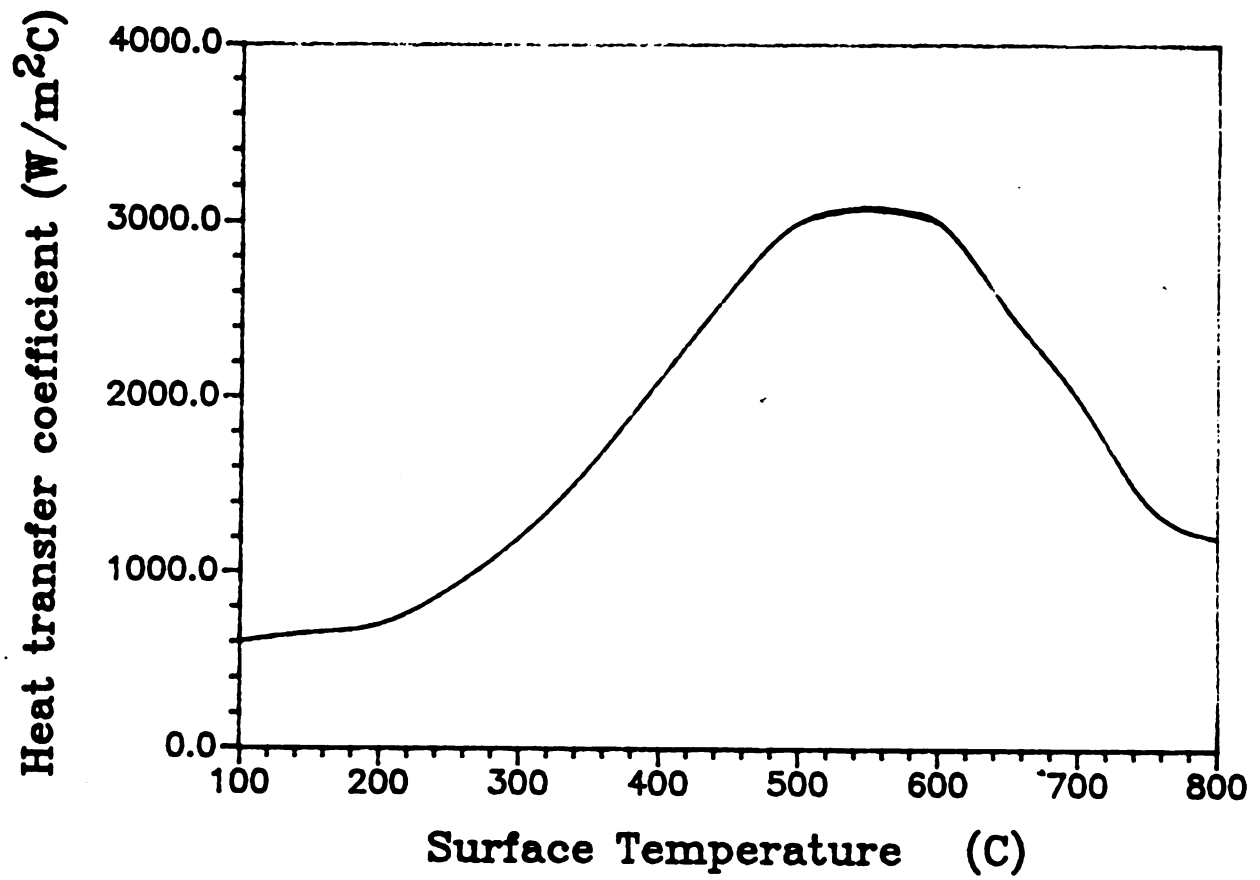


Figure 44b. Surface heat transfer coefficient of a steel cylinder for a oil quench (After Hachisu et al. [108]).

The heat transfer phenomena between a solid and liquid nitrogen is complicated. When the hot specimen reaches liquid nitrogen bath, the surface is immediately shielded by a film of nitrogen gas. Due to the film formation, the surface heat transfer rate become very small (figure 41).

In a study of nitrogen film boiling from a flat surface, Saucer and Ragsdell [109] measured heat flux of Inconel 600 plate as a function temperature from about 40 C up to 593 C. Six thirty-gage chromel-alumel thermocouples were attached on the back side of the heat transfer surface. An assembled heater then placed into the dewar. Power to the heater assembly was increased slowly until film boiling was initiated. Data was recorded after equilibrium was reached. The heat transfer coefficient was almost constant and about $290 \text{ W/m}^2 \text{ C}$ up to 590 C (figure 45) [109].

However, in this study alumina specimen was dipped into the liquid nitrogen bath virtually and transient surface temperature was measured. From 30 degrees C to 500 degrees C, the value of h determined in this study, was between 100 and $200 \text{ W/m}^2 \text{ C}$ which is very close to the results of Stoltz. This value of h is small compared to those of water or oil quench (Table 24). Thus, the maximum heat transfer coefficient for the oil bath was an order of magnitude lower than that for the water bath and an order of magnitude higher than that for the liquid nitrogen bath (Table 24). For the liquid nitrogen quench, the two most severe thermal shock conditions corresponded to specimen temperatures of 740 and 855 degrees C. Since the specimen

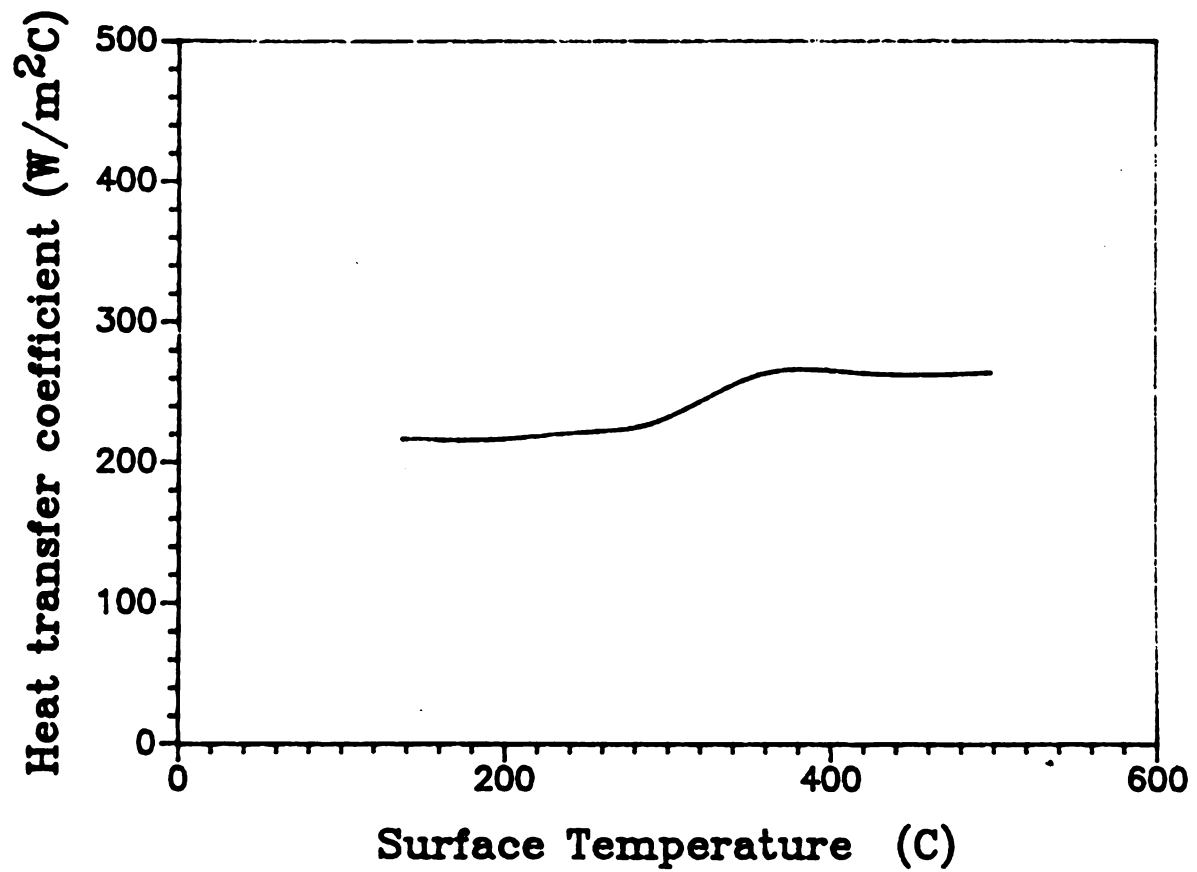


Figure 45. Surface heat transfer coefficient of a Inconel for liquid N₂ quench (After Saucer et al. [109]).

Table 24. Maximum heat transfer coefficient for various quench media

Quench Media	Maximum values (W/m ² °C)	Temperature (°C)	Material	Specimen Geometry	Reference
Water	56000	220	Steel	cylinder	[108]
	63000	370	Alumina	bar	this study
	53000	410	TiB ₂	bar	[111]
	200000	370	Macor	bar	[111]
Oil	11000	530	Silver	sphere	[107]
	3000	550	Steel	cylinder	[108]
	6000	600	Alumina	bar	this study
Liquid nitrogen	290	550-660	Inconel	plate	[109]
	200	500	Alumina	bar	this study

temperatures were above 700 C, radiation heat transfer likely becomes increasingly important as well as the film boiling heat transfer coefficient [45, 110]. Thus the liquid nitrogen quench test above 700 degrees C must consider radiation effects. The effective radiation coefficient h_{rad} [110] may be approximated as

$$h_{rad} = \frac{\epsilon_{em} \sigma_{S.B.} (T_s^4 - T_\infty^4)}{(T_s^4 - T_\infty^4)} \quad (73)$$

where h_{rad} = effective radiation heat transfer coefficient

ϵ_{em} = emissivity of the solid

$\sigma_{S.B.}$ = Stefan-Boltzmann constant

T_s = surface temperature of the solid

T_∞ = temperature of the quench media

The emissivity of the alumina at 1000 C is about 0.79 [112] and the room temperature emissivity of the alumina is approximately 0.2 [113]. Based on these two values, the emissivity at 739 and 855 C was linearly extrapolated and estimated as 0.65 and 0.72, respectively. Based on these emissivity values, the values of h_{rad} , are $41.3 \text{ W/m}^2 \text{ C}$ for 739 C specimen, $62.7 \text{ W/m}^2 \text{ C}$ for 855 C specimen and $112 \text{ W/m}^2 \text{ C}$ for 1000 C specimen. The effective heat transfer coefficient is additive. If we assume the convective heat transfer coefficient at 1000 C was about $200 \text{ W/m}^2 \text{ C}$, then the total effective heat transfer coefficient will be about $300 \text{ W/m}^2 \text{ C}$. Thus radiation effect at 1000 C will increase 50 % in heat transfer coefficient.

4.4.4. Effect of Quench Media on Thermal Shock

When a material undergoes a sudden transient temperature change, thermomechanical stresses induce in a material. An equation for the maximum thermal stresses in a homogeneous flat plate has been considered by Manson [94]. The nondimensional stress, which is the ratio of the actual stress developed to the stress that would developed from extremely rapid cooling (that is, for an infinite heat transfer coefficient), can be described as [94]

$$\sigma^* = \frac{\sigma(1-\nu)}{E\alpha_{th}\Delta T} \quad (74)$$

where σ^* = a nondimensional stress

σ = the actual thermal stress

ν = Poisson's ratio

E = Young's modulus

α_{th} = thermal expansion coefficient

and ΔT = the temperature difference.

Manson described σ_{max}^* , the maximum nondimensional thermal stress at the surface of the plate, as

$$\sigma_{max}^* = [1.5 + 3.25/N_{bi}]^{-1} \quad \text{for } 0 < N_{bi} < 5 \quad (75a)$$

$$\text{and } \sigma_{max}^* = [1.0 + 3.25/(N_{bi}^{0.67})]^{-1} \quad \text{for } 5 < N_{bi} < 20 \quad (75b)$$

$$\text{or } \sigma_{\max}^* = [1.5 + 3.25/N_{bi} - 0.5\exp(-16/N_{bi})]^{-1} \text{ for } 0 < N_{bi} < 20 \quad (75c)$$

A nondimensional parameter N_{bi} , the Biot modulus, expresses the severity of thermal shock, where

$$N_{bi} = hz/k \quad (66)$$

where h = surface heat transfer coefficient

z = the half thickness of the plate

and k = the thermal conductivity of the plate.

The maximum actual thermal stress at the surface of the plate can be evaluated by

$$\sigma_{\max} = [E\alpha_{th}\Delta T/(1-\nu)] \times \sigma_{\max}^* \quad (76)$$

$$\sigma_{\max} = [E\alpha_{th}\Delta T/(1-\nu)] \times [1.5 + 3.25/N_{bi} - 0.5\exp(-16/N_{bi})]^{-1}$$

for $0 < N_{bi} < 20$.

Equation 76 indicates that the maximum thermal stress depends material properties, E , α_{th} and ν , the dimensions of the specimen and the surface heat transfer coefficient. For thermal shock of given material with a fixed thickness into differing quenching media, the parameter most sensitive to temperature changes is the heat transfer

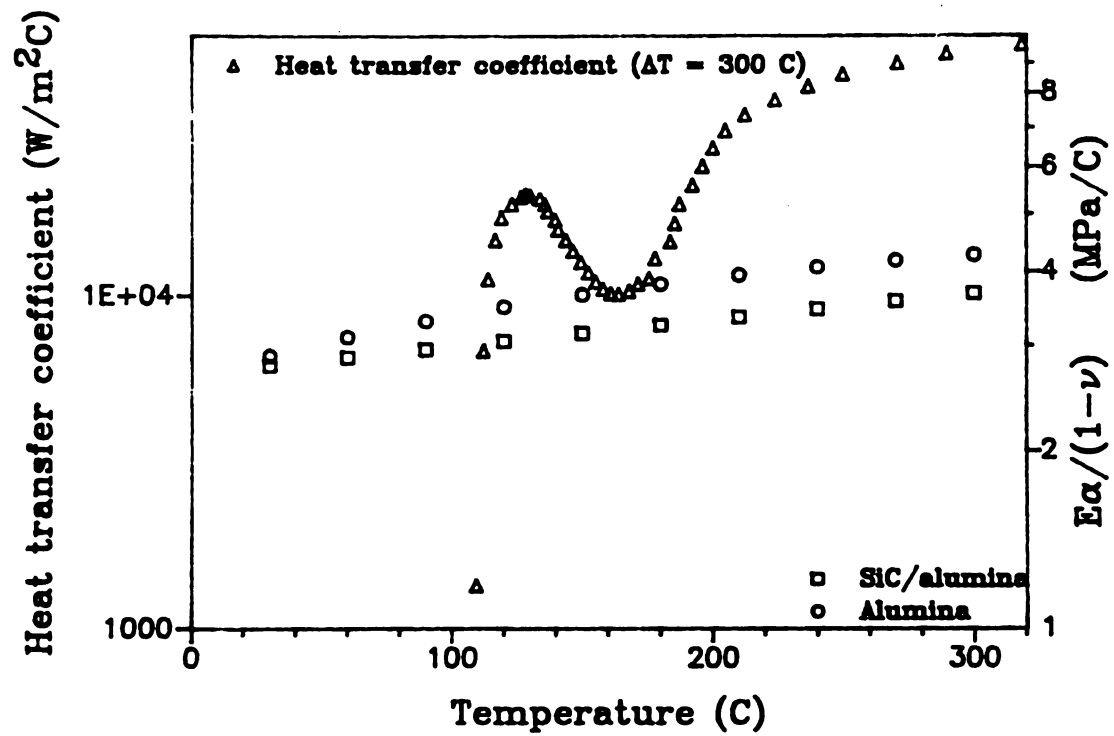


Figure 46a. Surface heat transfer coefficient of alumina and prefactor in equation 76, $E\alpha/(1-\nu)$, of alumina and SiC whisker reinforced alumina composites as a function of temperature.

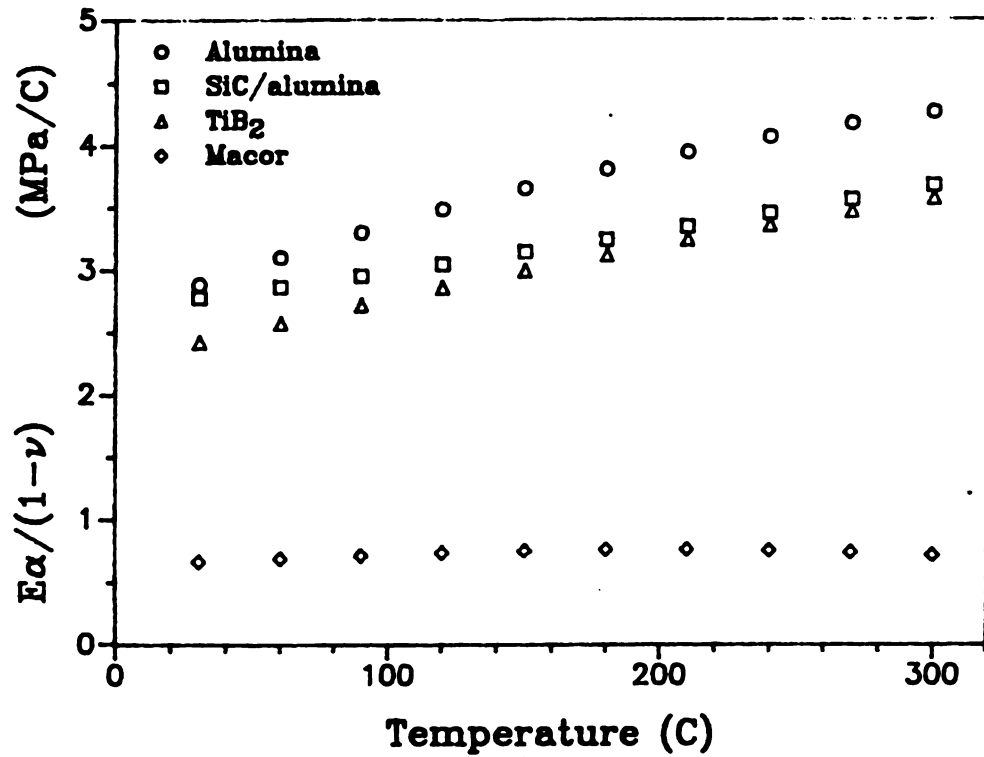


Figure 46b. Prefactor in equation 76, $E\alpha/(1-\nu)$, of titanium diboride and Macor glass ceramics as a function of temperature.

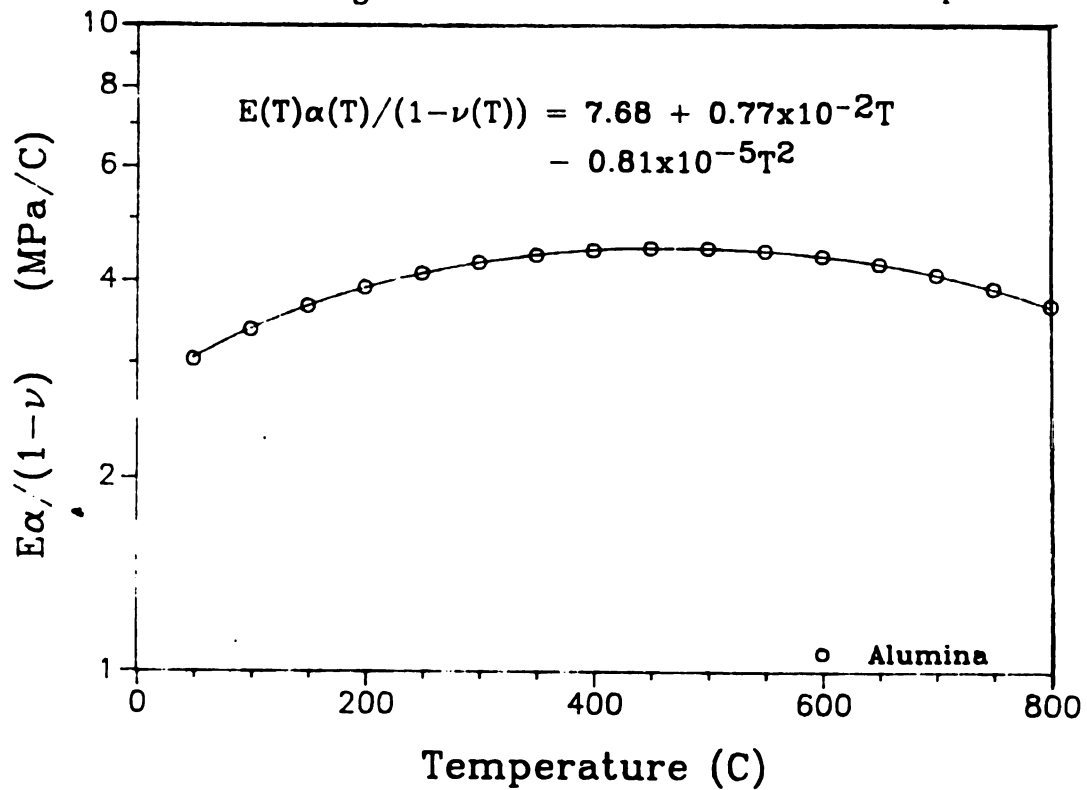


Figure 46c. Prefactor in equation 76, $E\alpha/(1-\nu)$, of alumina as a function of temperature up to 800 C.

coefficient (figure 46). The function $\sigma_{\max}^*$ in equation 76 varies significantly with Biot modulus for certain ranges of Biot modulus, Bi, e.g. $Bi < 5$.

From the previous section, $h_{\text{water}} > h_{\text{oil}} > h_{\text{liq. nitrogen}}$ for wide ranges of specimen temperature, thus $Bi_{\text{water}} > Bi_{\text{oil}} > Bi_{\text{liq. nitrogen}}$ given the material and specimen geometry is fixed.

Very small changes in modulus and internal friction were observed when the specimen quenched into liquid nitrogen bath. For the polycrystalline alumina specimens the maximum damage created by liquid nitrogen quench at $\Delta T = 1050$ degrees C ($A/E_0 = 0.115$ percent and $B/Q_0^{-1} = 50$ percent) was about same magnitude that created by 20 C silicone oil quench at $\Delta T = 500$ degrees C ($A/E_0 = 0.113$ percent and $B/Q_0^{-1} = 71$ percent) and water quench at $\Delta T = 230$ degrees C ($A/E_0 = 0.1$ percent and $B/Q_0^{-1} = 56$ percent). Thus the thermal shock damage trend mirrors the differences in h among the various media.

Although the alumina specimens were quenched into the liquid nitrogen at the highest ΔT of all the quenching media, the total damage was quite small compared to the other quench conditions. For 20 degrees C silicon oil quenched specimen, thermal shock damage became significant above the specimen temperature 564 degrees C. For the oil quench the thermal shock damage increased rapidly at a specimen temperature of about 590 degrees C.

4.4.5. The Critical Temperature Difference, ΔT_c

In this study, instead of using the classical method of determining of ΔT_c by measurements of the strength of quenched samples, we have used a non-destructive method: the measurements of dynamic young's modulus. Each specimen can give considerable information on the evolution of thermal shock damage as a function of thermal cycling.

If we observe the initial thermal shock cycle (for a virgin specimen) as a function of ΔT for the three different quenching media (20 C water, 20 C silicone oil and liquid nitrogen) we observe that at a critical temperature difference, ΔT_c , there is an abrupt decrease in modulus. ΔT_c (figure 47) was about 170 C (2mm thickness alumina, Saxonburg) and 240 C (1mm thickness alumina, Saxonburg and Coors) for 20 C water quench and 550 C (1mm thickness alumina, Coors) for 20 C silicone oil quench. However, no ΔT_c was observed for liquid nitrogen quenched specimens (1mm thickness alumina, Coors).

In equation 76, as ΔT increases, thermal shock damage occurs when $\sigma_{\max} \geq \sigma_f$, where σ_f is the tensile strength of the specimen. Using a tensile strength* of 193 MPa for the polycrystalline alumina [114] and

* Fracture strength of brittle material depends on the probability of flaw initiating fracture at a specific applied stress. Thus observed strength may vary with the manner in which the tests are conducted. In the bend strength test, the volume under maximum stress are smaller thus higher strength values are observed. When specimens are quenched, the outside of the specimen is in a tensile state and the inside in a compressive state. Thermal shock damage starts if the maximum value of these thermal stresses exceeds in a single point the tensile strength of the material.

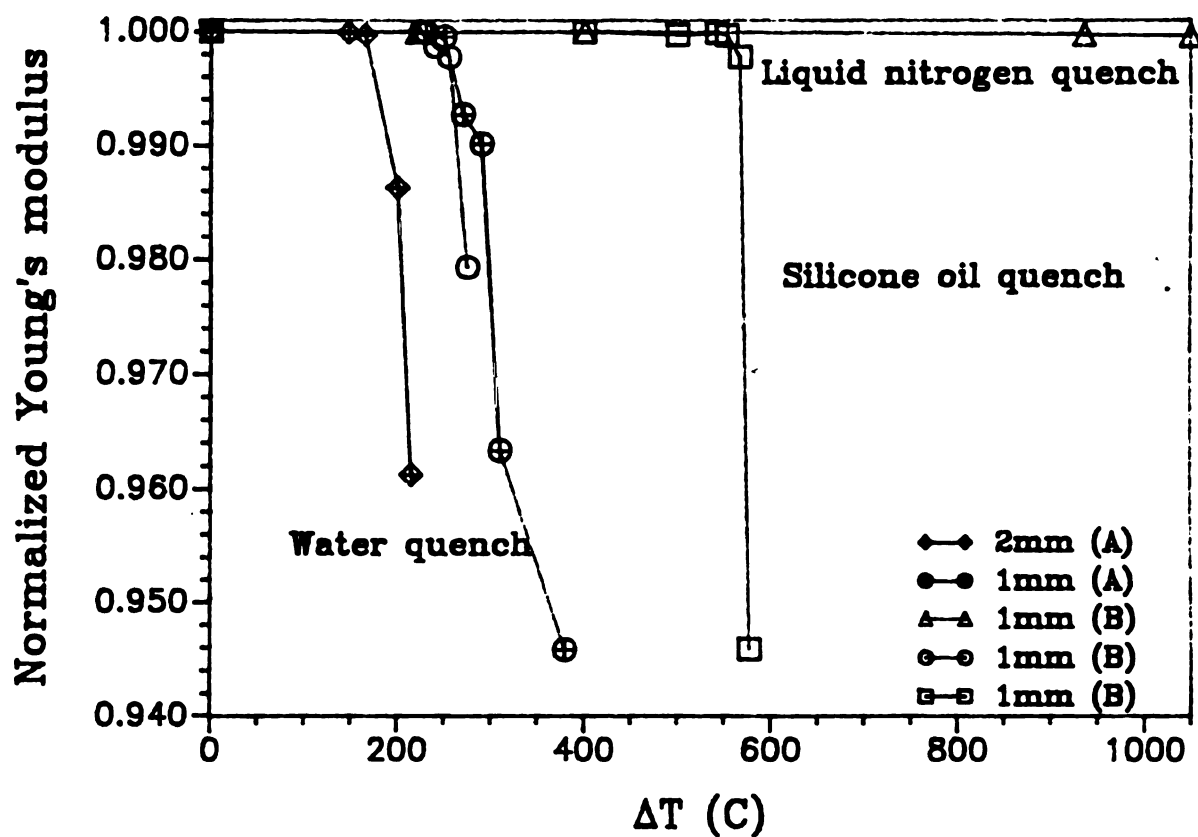


Figure 47. Young's modulus versus ΔT for various quenching media (vendor A - Saxonburg, vendor B - coors)

the temperature dependent material parameters (table 25), $\sigma_{\max}$ was computed for a range of values of ΔT . The ΔT value which gave $\sigma_{\max} = \sigma_f$ was chosen as ΔT_c . In table 26, we compare the observed ΔT_c and the calculated ΔT_c of alumina specimens as well as the properties used in this study for water quench and silicone oil quench. As the Biot modulus decreased (via a decrease in h or by a decrease in specimen size) from 2.3 to 0.3, the critical temperature difference increased from 150 degrees C to 510 degrees C. For the liquid nitrogen quench, the low Biot modulus (low heat transfer coefficient) meant that $\sigma_{\max}$ never reached σ_f for the specimen temperature 850 degrees C. The observed ΔT_c values for 2 mm thickness water quenched specimens and for 1 mm thickness oil quenched specimens agree well with the calculated values. However, 60 degrees C difference was found between the observed ΔT_c and calculated ΔT_c (equation 76) for 1 mm thickness water quenched specimens (Table 26).

4.4.6. Saturated Damage Expressed in Terms of Thermal Stress and Duration Time

For an infinite plate of half thickness z initially at uniform temperature, T_0 , quenched into a medium at a lower temperature T_∞ (figure 48), the heat conduction equation is

$$k(\partial^2 T / \partial Z^2) = \rho C_p (\partial T / \partial t) \quad (77)$$

Table 25. Temperature Dependent Material Properties of Polycrystalline Alumina. Unit of T is Celcius degree. (Result of least squares best fit of available data)

Property	Functional Form	Temp. range	cor. coeff & ref
E (GPa)	$335.49 - 0.027T$	20 C < T < 600 C,	0.99 this study
$\alpha_{th} (10^{-6}/C)$	$6.05 + 0.019T - 0.000019T^2$	20 C < T < 600 C,	0.98 [58]
ν	$0.233 - 0.149 \times 10^{-4} T$	20 C < T < 500 C,	0.99 [83]
k (W/m C)	$20.5 - 3.19 \times 10^{-2} T + 1.9 \times 10^{-5} T^2$	20 C < T < 600 C,	0.98 [58]
C_p (J/kg C)	$791.4 + 1.41T - 1.63 \times 10^{-3} T^2 + 7.03 \times 10^{-7} T^3$	20 C < T < 800 C	0.99 [58]

Table 26. Critical Quenching Temperature of Polycrystalline Alumina for Various Quenching Media and Various Geometry

Media	Specimen Vendor	Dimensions + (cm)	Bi	ΔT_c^{+++} (cal.)	ΔT_c (obs.)	ref
Water	Saxonborg	th. = 0.2 w = 0.9-1.0 l = 9.4-10.0	2.3	155 C	170 C	*
Water	Saxonborg	th. = 0.1 w = 1.2-1.3 l = 11.4-11.5	1.6	180 C	240 C	*
Water	Coors	th. = 0.1 w = 1.2-1.3 l = 7.0-10.7	1.6	180 C	240 C	*
Oil	Coors	th. = 0.1 w = 1.3 l = 8.5-10.5	0.34	490 C	550 C	*
Liq.N ₂	Coors	th. = 0.1 w = 1.2 l = 7.0	< 0.04	>> 2000 C	**	*
Oil	McDanel++	0.63 cm diameter cylindrical rod	1.06 ^{***}	***	430 C	[15]

+ th. = thickness, w = width, and l = length

++ 998 Alumina (E = 393 GPa)

+++ ΔT_c was calculated by using equation 76.

* this study

** ΔT_c was not observed.

*** Bi^C modulus was not obtained from the measured heat transfer coefficient but calculated from the observed ΔT_c thus calculated ΔT_c is same as observed ΔT_c .

Note. Although the two kind of polycrystalline alumina (Coors and Saxonborg) are equally 96 % alumina, the properties (grain size, porosity and modulus) were somewhat different. The grain size, porosity range and Young's modulus ranges for Saxonborg vendor used in this study were about 10 μm , 6 - 7.4 % and 323 - 338 GPa, respectively. For Coors vendor used in this study the grain size, porosity range and Young's modulus ranges were about 7 μm , 3 - 5.9 % and 337 - 361 GPa, respectively.

where k - thermal conductivity of the specimen

C_p - specific heat

ρ - density of the specimen

Then the transient temperature distribution along the thickness direction of the plate may be obtained using the boundary conditions,

$$T = T_0 \quad \text{at } t = 0 \quad (78a)$$

$$(\partial T / \partial Z) = -h(T - T_\infty) / k \quad \text{on } Z = z \quad (78b)$$

$$(\partial T / \partial Z) = 0 \quad \text{on } Z = 0 \quad (78c)$$

where h - surface heat transfer coefficient

z - half thickness of the plate

The thermal stress on the surface can be expressed as follows [21]

$$\sigma(f) = \sum_{k=0}^{\infty} \{ [2 \exp(-\eta_k^2 f) / (1 + R + R^2 \eta_k^2)] * (\eta_k^{-2} - R) \} \quad (79)$$

where η_k - roots of the transcendental equation

$$(R \eta_k \tan \eta_k = 1)$$

R - inverse of Biot modulus

or

$$\sigma(f) = [E \alpha_{th} \Delta T / (1 - \nu)] \{ (1 + 1/Bi) (1 - \exp(f \cdot Bi^2) \cdot \operatorname{erfc}(f^{0.5} Bi)) - 2(f/\pi)^{0.5} \} \quad (80)$$

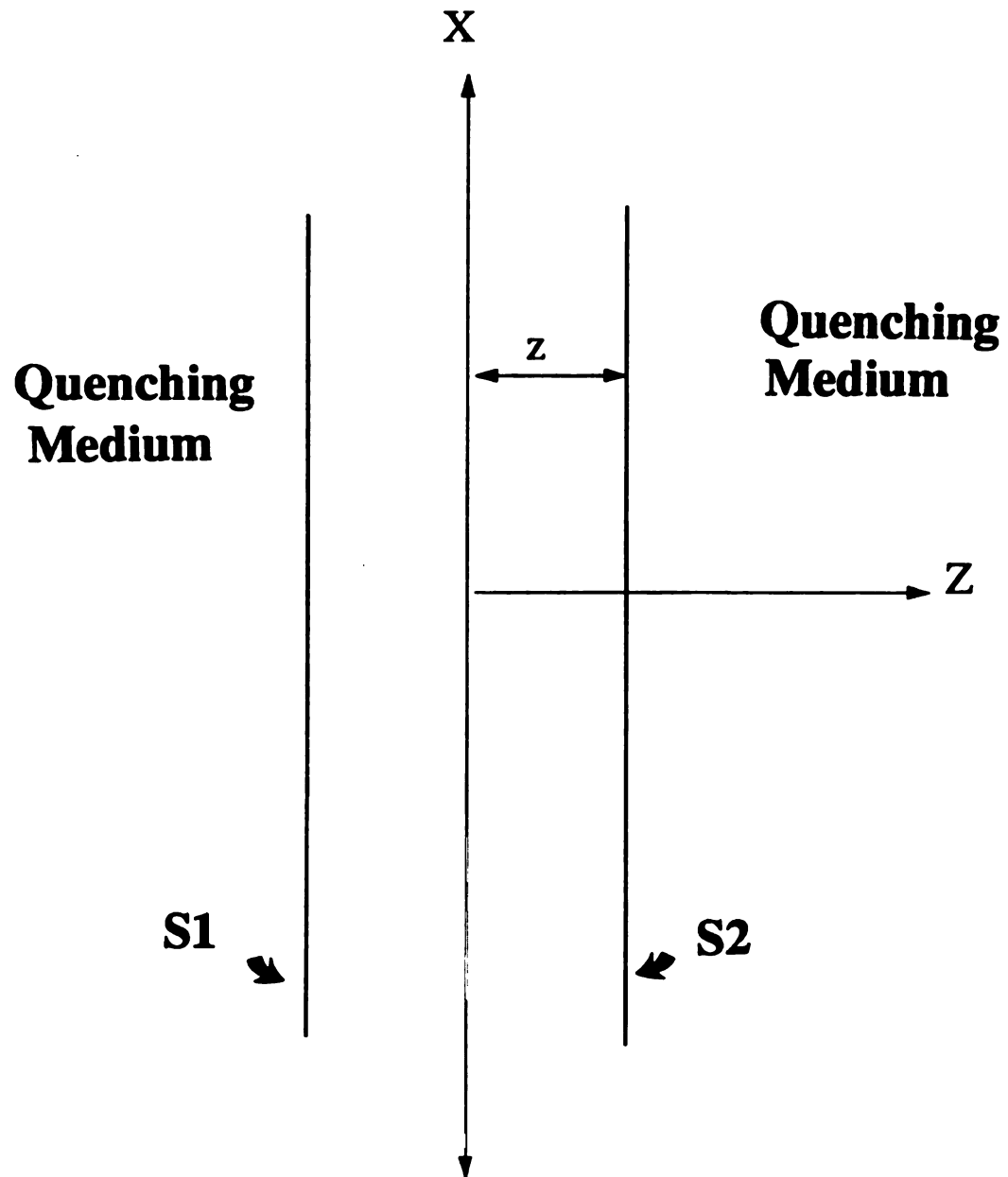


Figure 48. Flat plate initially at T_o quenched into a medium at a lower temperature T_∞ . The quenching medium contacts the plate at both of the transverse surfaces of the plate (labeled here as $S1$ and $S2$).

where $\sigma(t)$ - thermal stress on the surface as a function of time

E - Young's modulus

α_{th} - the coefficient of thermal expansion

ΔT - initial temperature difference between bath and specimen

ν - Poison's ratio

Bi - Biot modulus, $(1/R - hz/k)$

f - dimensionless time, $kt/C_p \rho z^2$

k - thermal conductivity of the specimen

C_p - specific heat

ρ - density

z - half thickness of the specimen

Cheng assumes the material to be homogeneous and isotropic, with properties that do not depend on temperature. Also the heat transfer coefficient, h , is assumed constant. In practice, the heat transfer coefficient and thermal conductivity vary significantly with temperature.

We calculate time dependent surface thermal stress in two steps:

(1) we use the temperature dependent functional forms of E , α_{th} , and ν (Table 25) along with the Biot modulus of initial specimen temperature value (Table 27). Thus, the time dependent surface stress increases rapidly, follows by maximum, then decreases slowly (figure 49). The maximum thermal stress is obtained at the time of 11 msec for water quench and 35 msec for oil quench. The peak stress reflects the nature of crack initiation and at the surface where the tensile surface

stresses are a maximum. We assume that crack initiation occurs when $\sigma(f) = \sigma_f$ (at time f_1), where σ_f is the tensile strength of the material. The crack presumably arrests when $\sigma(f) = \sigma_f$ (at time f_2) (figure 50). The time interval, Δf , between f_2 and f_1 as well as the thermal stress magnitude also affect the thermal shock damage (crack growth). For the water quench, the peak stress is larger than that for oil quench, however the duration time is much shorter than that for oil quench (figure 49). We must consider both factors for thermal shock damage. The product (area) between stress, $\sigma(f)$ and duration time $\Delta f \cdot (f_2 - f_1)$ could be related to the thermal shock damage.

Since the heat transfer coefficient changes very rapidly with time (temperature) especially for water quench, the Biot modulus also changes rapidly (figure 51). When the maximum stress is reached, the Biot modulus and the thermal conductivity are not the same as the values prior to commencing the thermal quench. We averaged the Biot modulus* between the crack initiation time f_1 ($\sigma(f_1) = \sigma_f$) and the crack arrest time f_2 ($\sigma(f_2) = \sigma_f$), since the rest of the stress portion may not significantly affect the crack growth. With these

.....

$$* Bi_{ave} = \frac{\int_{f_1}^{f_2} Bi(f) df}{(f_2 - f_1)} \quad (79)$$

$Bi(f)$ was expressed in terms of polynomial via a least square best fit of the data in figure 51.

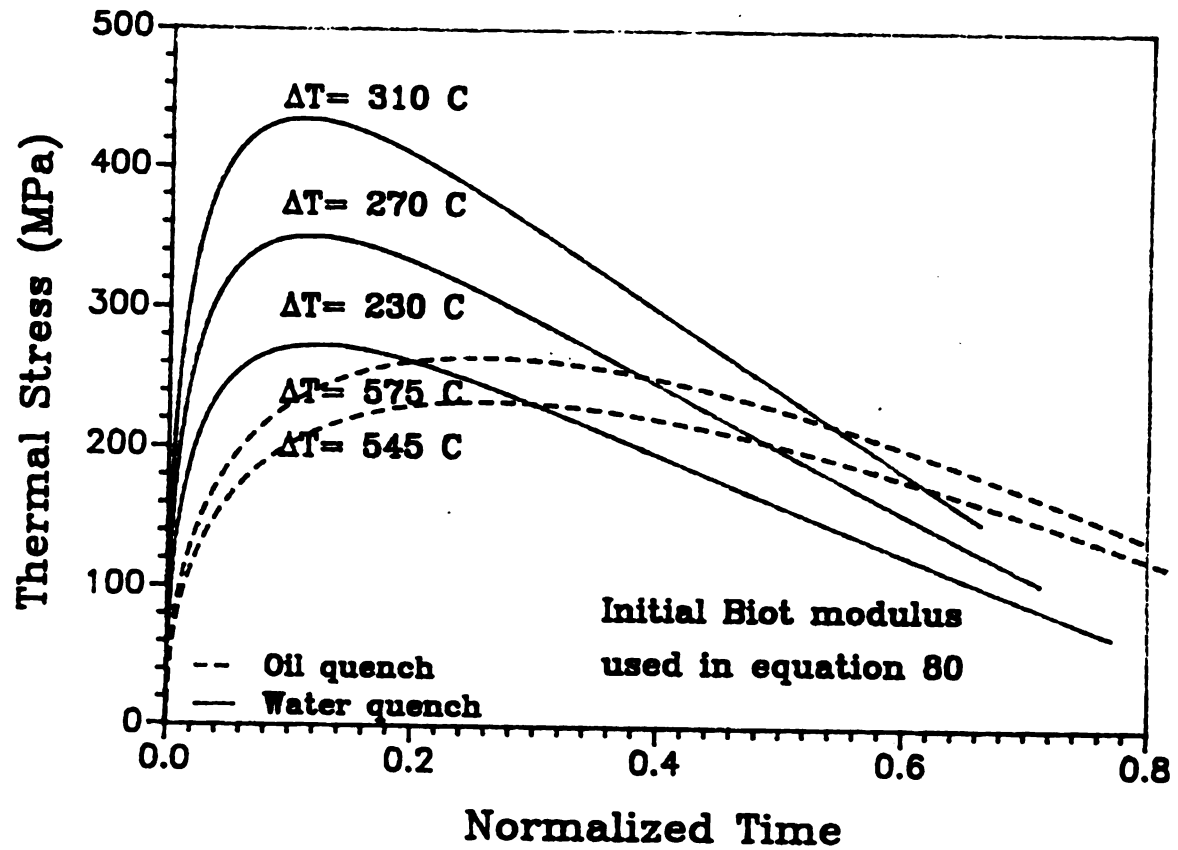


Figure 49a. Surface tensile stress as a function of nondimensional time for 1mm thickness alumina using initial Biot modulus.

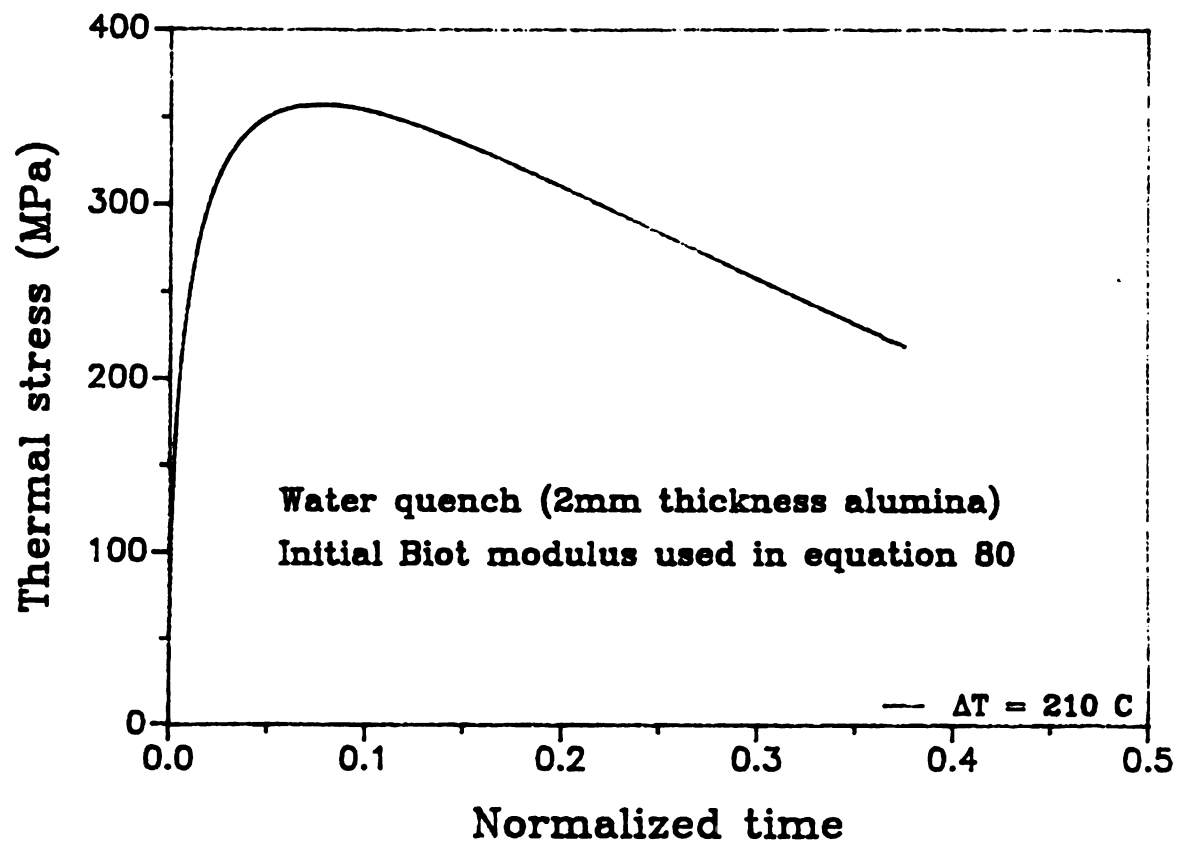


Figure 49b. Surface tensile stress as a function of nondimensional time for 2mm thickness alumina using initial Biot modulus.

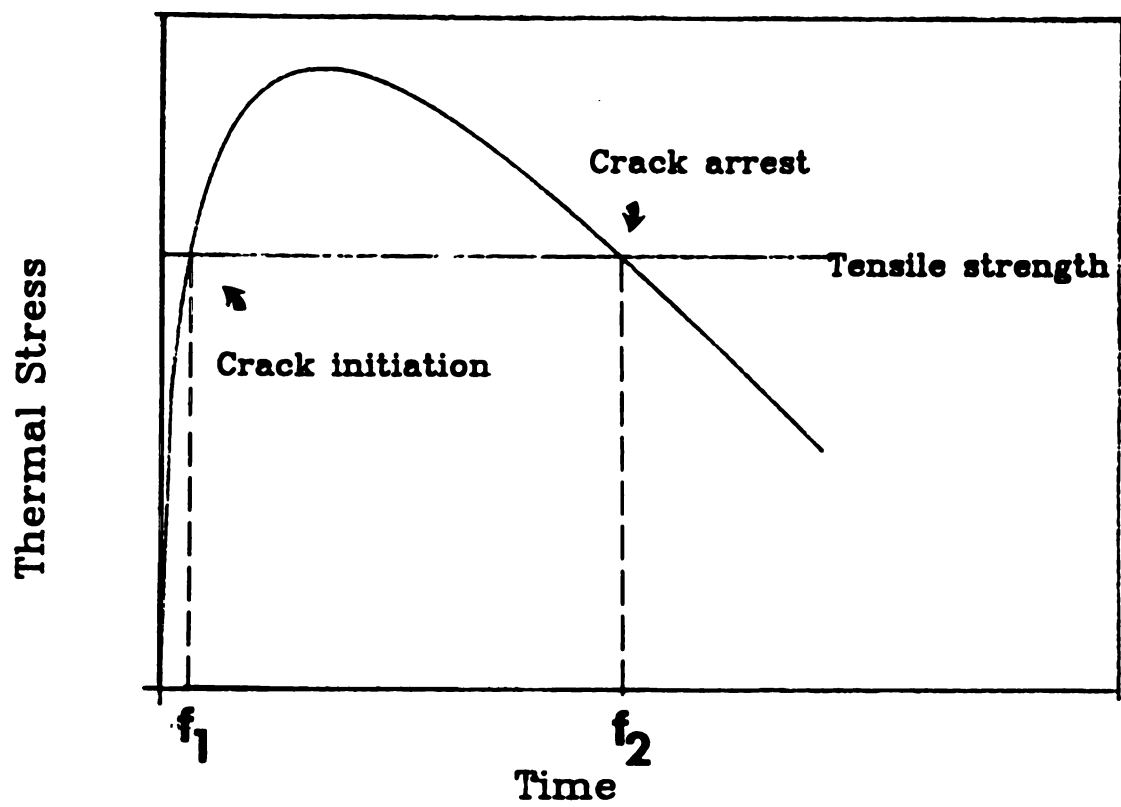


Figure 50. Schematic of surface stress as a function of time showing crack initiation and crack arrest

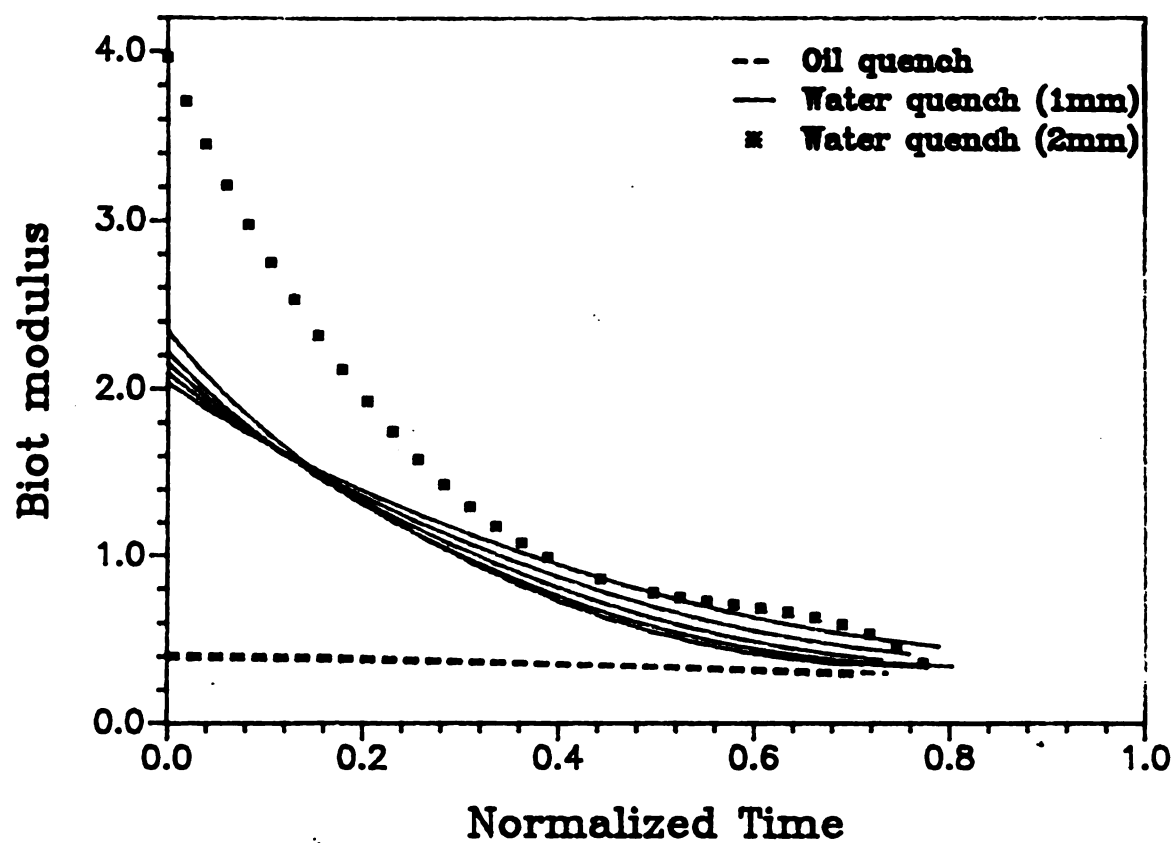


Figure 51. Biot modulus for alumina as a function of nondimensional time. For 1mm thickness water quenched alumina, five different ΔT (310, 290, 270, 250 and 230 C) were included.

averaged Biot modulus (Table 27), improved thermal stress as a function of time was obtained (figure 52). Since the average Biot modulus values of water quench were much lower than the initial values, the maximum thermal stress values of water quench were considerably reduced. However, for oil quench, the average Biot modulus doesn't differ greatly from the initial Biot modulus, so the peak stress wasn't reduced considerably. As an example, for water quench ($\Delta T = 310$ degrees C) the maximum tensile stress is reduced from 428 MPa to 282 MPa and for oil quench ($\Delta T = 575$ degrees C) the maximum tensile stress is reduced from 268 MPa to 250 MPa.

A power law relationship* can be written in terms of the maximum thermal stress for the polycrystalline alumina quenched into water and oil bath, such that

$$A/E_o = c_1 \cdot (\sigma_{\max}/\sigma_f)^{r_1} \quad (81)$$

where $\sigma_{\max}$ - maximum surface thermal stress at a fixed ΔT

σ_f - tensile strength of the material

c_1, r_1 - thermal fatigue parameters

However, the plot of $\ln(A/E_o)$ versus $\ln(\sigma_{\max}/\sigma_f)$ for the polycrystalline alumina quenched into water and oil bath (figure 53), show two different sets of curves for the two different quenching

* This power law relationship is similar to the power law equation in terms of normalized temperature (equation 42 in Section 4.1.7.).

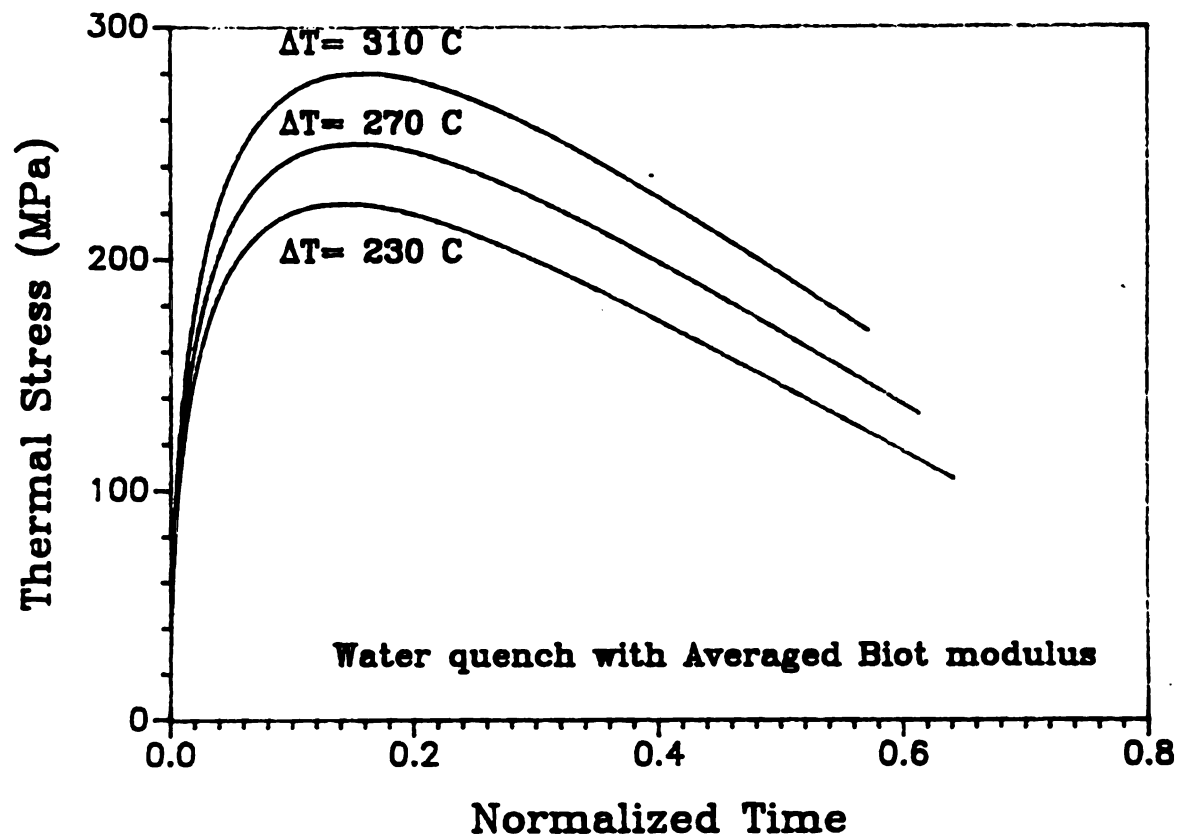


Figure 52a. Surface tensile stress as a function of nondimensional time for 1mm thickness alumina quenched into water using average Biot modulus.

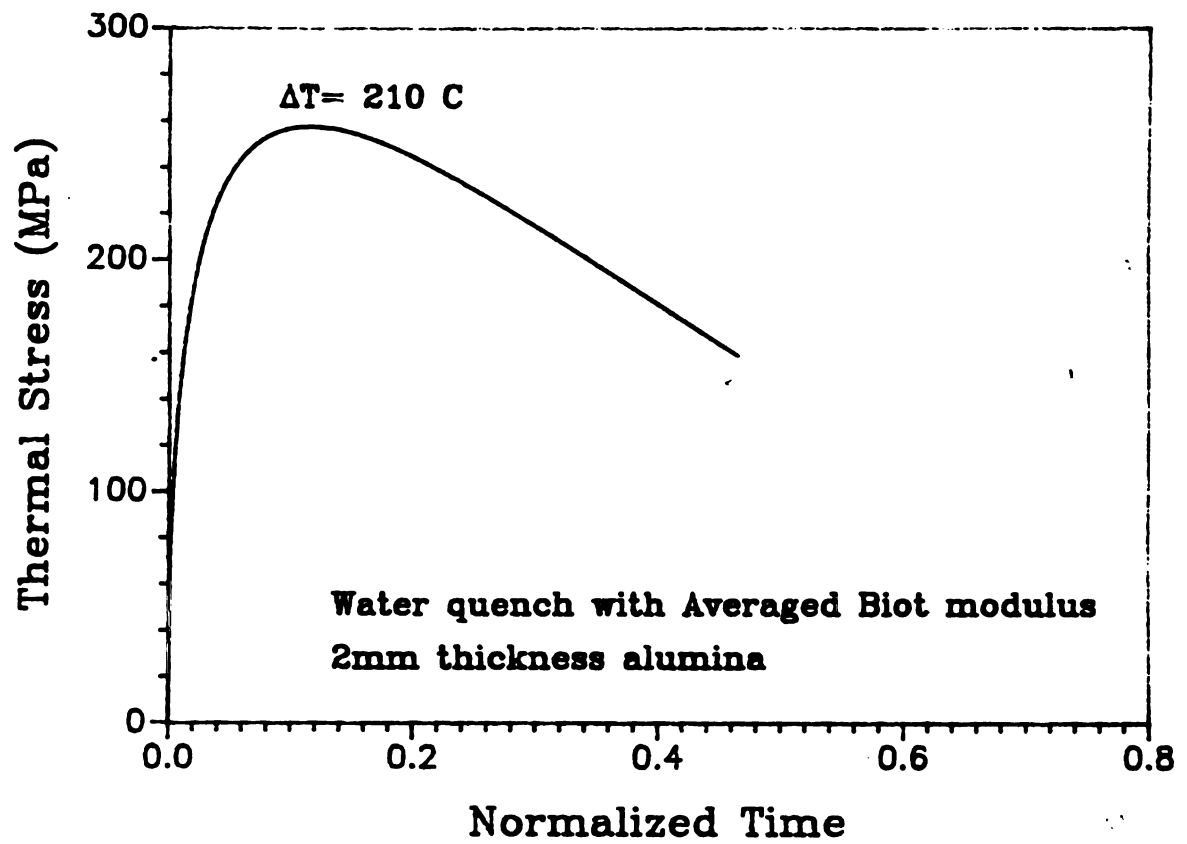


Figure 52b. Surface tensile stress as a function of nondimensional time for 1mm thickness alumina quenched into oil using average Biot modulus.

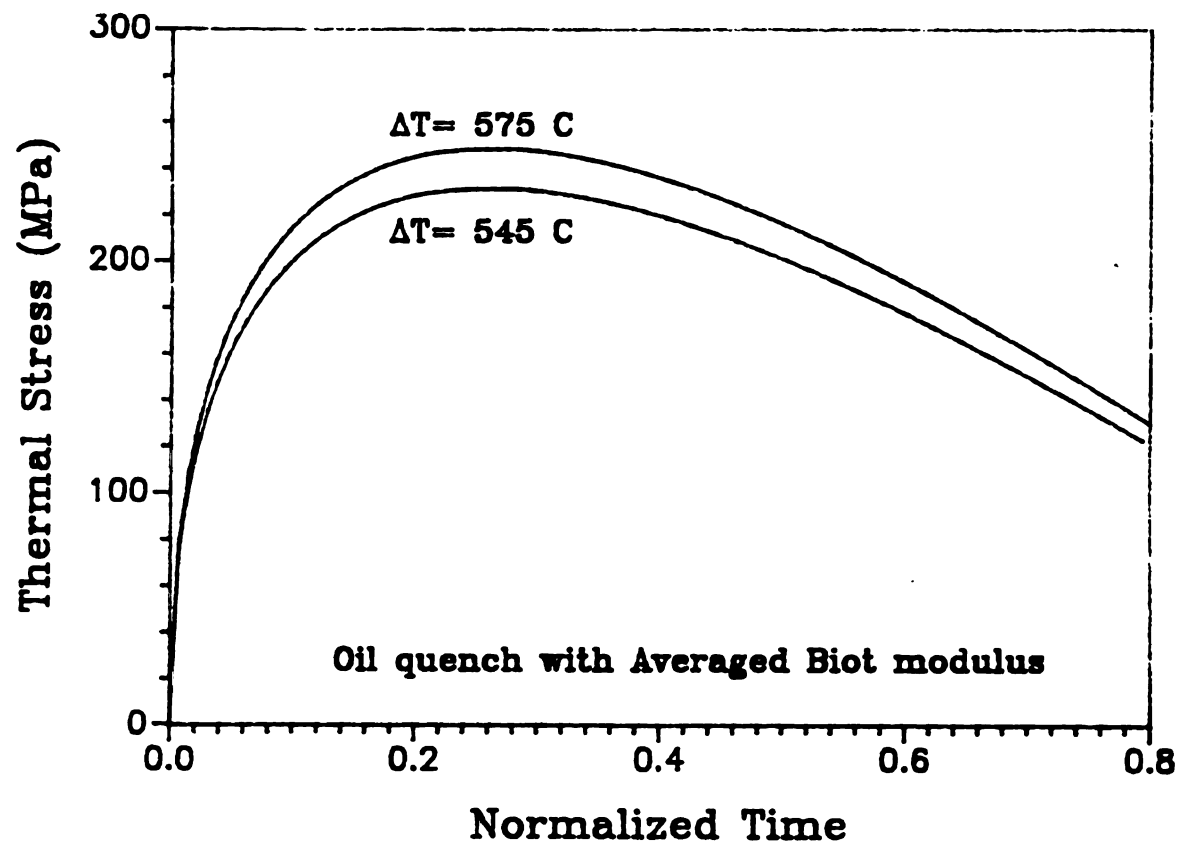


Figure 52c. Surface tensile stress as a function of nondimensional time for 2mm thickness alumina quenched into water using average Biot modulus.

Table 27 Average Biot Modulus and Initial Biot Modulus for Water Quench and the Oil Quench for Different Temperature Regimes

Quench Medium	ΔT (C)	Initial Biot Modulus	Average Biot Modulus
Water +	210 *	3.96	2.15
	230	2.03	1.19
	250	2.09	1.11
	270	2.15	1.00
	290	2.21	0.93
	310	2.33	0.88

Oil ++	542	0.388	0.357
	553	0.392	0.359
	575	0.410	0.367

+ Saxonburg

++ Coors

* 2mm thickness specimen

media. The linear regression values of the slopes r_1 and intercept c_1 are 8.08 ± 1.63 , -9.44 ± 1.06 for water quench and 22.72 ± 2.83 , -9.59 ± 0.81 for oil quench. Note that the intercepts for two different quench media are almost same. Thus if the $\sigma_{\max} = \sigma_{t.s.}$, then the saturation thermal shock damage for oil quench and water quench will be same. We may expect the same saturation thermal shock damage if the $\sigma_{\max}$ were same for water quenched specimen and oil quenched specimen. However, we observed a big difference in saturation thermal shock damage between oil quenched specimen and water quenched specimen. This difference may attribute to the difference in transient stress profile.

Thus, if we consider the duration time as well as the stress, the differences between the curves in figure 53 could be reduced. A plot of normalized damage, A/E_0 versus the product between stress $\sigma(f)$ and duration time Δf for the polycrystalline alumina quenched into water and oil bath (figure 54a and 54b) shows a power law relation

$$A/E_0 = c_1 * [(\sigma(f)/\sigma_f) * \Delta f]^r \quad (92)$$

where c_1 = constant

$\sigma(f)$ = time dependent thermal stress

Δf = duration time ($f_2 - f_1$)

Using the Biot modulus of initial specimen temperature, we calculate time dependent surface stress (equation 80) and the product

between stress $\sigma(f)$ and duration time Δf for the polycrystalline alumina quenched into water and oil bath. Then the plot of $\ln(A/E_o)$ versus $\ln(\sigma(f)/\sigma_f \cdot \Delta f)$ for the polycrystalline alumina quenched into water and oil bath (figure 54a), was improved. The values of $r = 4.03 \pm 1.90$ and $c_1 = -2.61 \pm 0.78$ were obtained from the least squares procedure. The correlation coefficient for the least squares fit was 0.62.

With the average Biot modulus (equation 80), the two curves in figures 53 collapse to single curve (figure 54b). With the average Biot modulus, a least squares best fit yielded value of $r = 5.4 \pm 0.81$ and $c_1 = -0.063 \pm 0.62$ with 0.93 correlation coefficient. Equation 92 is similar to the power law equation in A/E_o and $\Delta T/T_r$ (equation 42 in Section 4.1.7.).

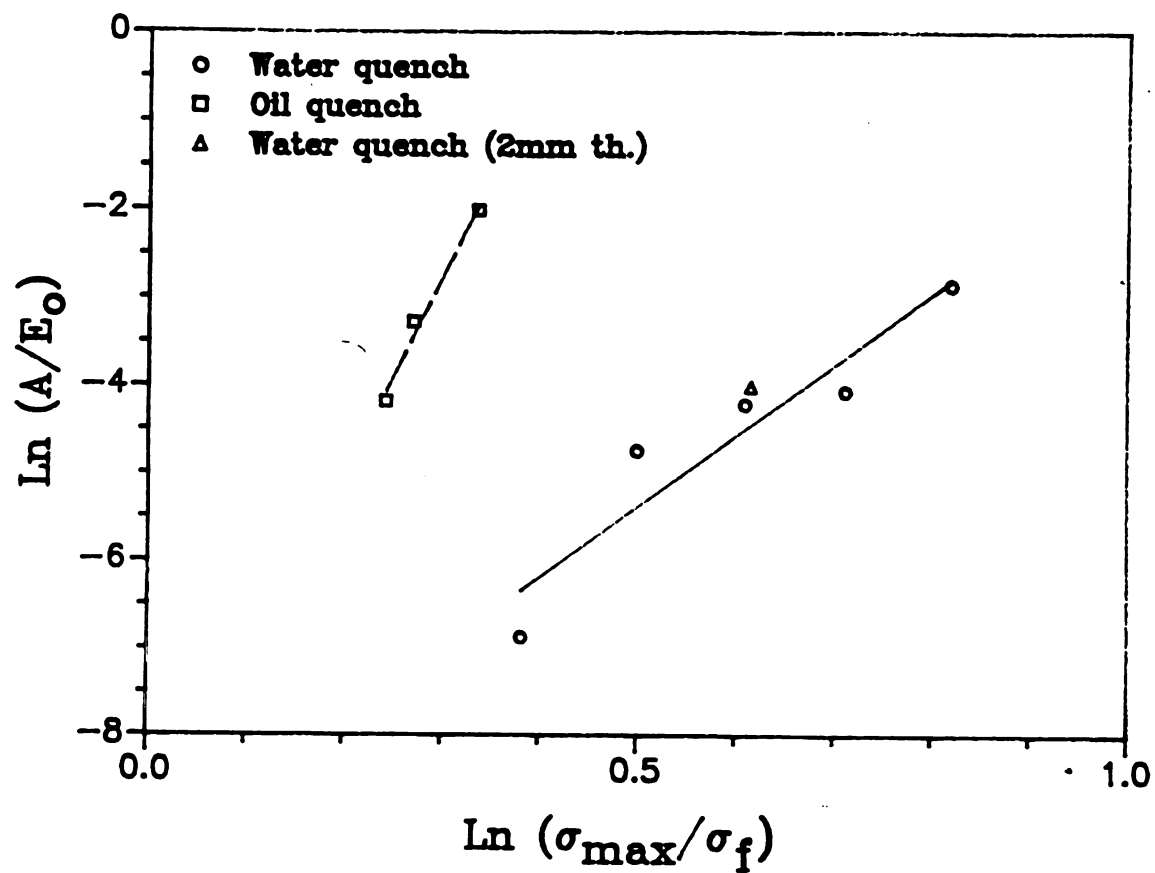


Figure 53. Normalized saturation damage parameter, A/E_0 versus the normalized maximum thermal stress, $(\sigma_{\max}/\sigma_{t.s.})$.

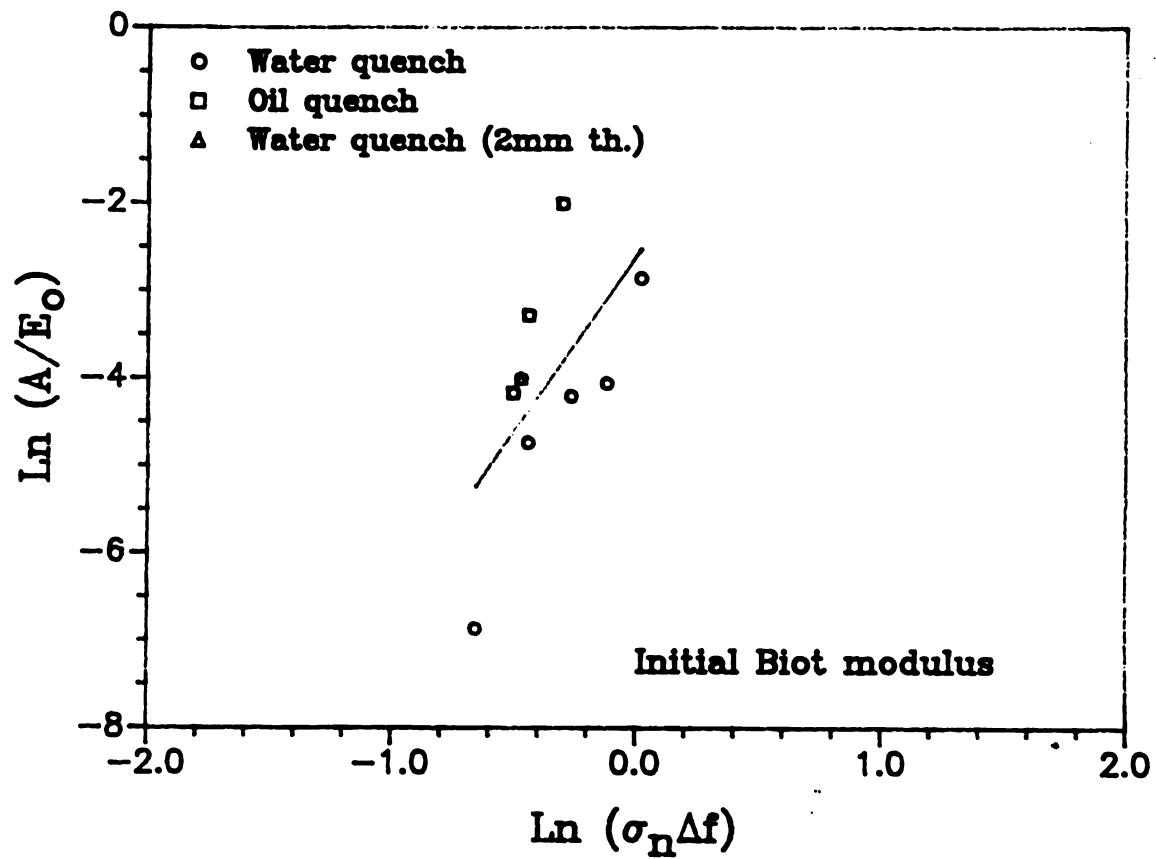


Figure 54a. Normalized saturation damage parameter, A/E_0 , versus the product of normalized thermal stress and duration time, $\sigma_n \cdot \Delta f$ using the initial Biot modulus.

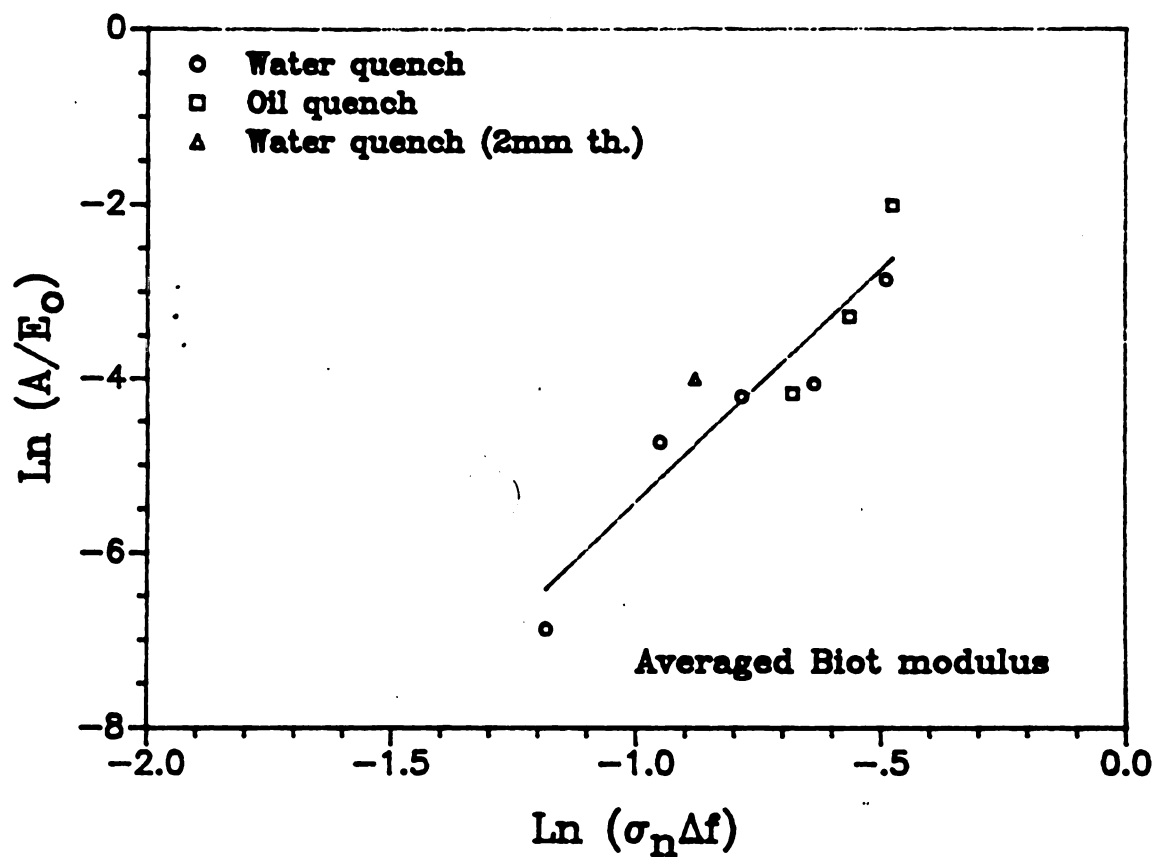


Figure 54b. Normalized saturation damage parameter, A/E_0 versus the product of normalized thermal stress and duration time, $\sigma_n \cdot \Delta f$ using the average Biot modulus.

5. Summary and Conclusions

5.1. Cyclic Thermal Shock of SiC Whisker Reinforced Alumina Composites

The Young's modulus and internal friction values for thermally cycled SiC whisker/alumina composites were observed to change as a function of the cumulative number of thermal shock cycles, presumably due to the accumulation of thermal shock induced microcrack damage. However, the thermal shock damage, as measured by both the modulus and internal friction, saturates at a sufficiently high number of thermal cycles. For a ΔT of 270°C , the saturation in internal friction ($1/\beta_{\text{crit}}$) occurred at about 49 cycles, while for 380°C , the saturation in internal friction occurred at about 3 cycles. It should be noted that in this work, the maximum number of thermal shock cycles per specimen was 100. Further research needs to be done to determine if the saturation behavior is still observed for thermal shock specimens subjected to a much higher cumulative number of thermal shock cycles, or whether in fact the damage once again increases when the cumulative number of thermal shock cycles is considerably greater than 100.

A plot of the relative change in internal friction versus the damage parameter ϵ can be represented as a straight line for the four specimens. In figure 16, four curves (one curve for each ΔT value) depict the internal friction change as a function of increasing cumulative thermal cycle number n . The data contained in the four Q^{-1} versus n curves in figure 16 is replotted in figure 20 in terms of

ΔQ^{-1} versus ϵ damage parameter. The four nonlinear internal friction curves from figure 16 thus collapse onto a single linear plot of ΔQ^{-1} versus ϵ . Thus the thermal shock damage, at least for the specimens included in this study, can be expressed as a simple linear function of the crack damage parameter.

The linear relation between the change in internal friction and the crack damage parameter can be understood in terms of fundamental processes if we assume that the microcracking contribution to internal friction is proportional to the integrated crack surface area. The ratio of the crack damage parameter (third moment) to the crack area (second moment) can be written in terms of the shape parameters for a given distribution, for several probability distributions (see section 3.4.). When one fixes the shape parameters for a given distribution, one then fixes the parameters relating the ratio of the moments, so that the crack damage parameter can be expressed as a constant multiple of the integrated crack area. Therefore, the observed linear relation between ΔQ^{-1} , the crack-damage induced change in internal friction, and ϵ , crack damage parameter can be explained if we assume ΔQ^{-1} is proportional to the total crack area.

A power law relation was found between the normalized temperature difference $\Delta T/T_r$ and the normalized modulus and internal friction saturation values A/E_0 (equation 42) and B/Q_0^{-1} (equation 43), respectively. Equations 42 and 43 thus relate the temperature difference to the saturation damage levels as measured by the modulus in the case of A/E_0 and as measured by internal friction in the case

of B/Q_0^{-1} . The linearity of thermomechanical stress with the temperature change ΔT implies a power law relation of the total accumulated crack damage with stress. Such relations, which can be expressed in terms of a stress intensity factor can apply to crack growth by either a fatigue mechanism akin to that of metals (equation 1) or to slow crack growth.

5.2. Cyclic Thermal Shock into Water Bath of Polycrystalline Alumina

The accumulation of thermal shock induced microcrack damage for thermally cycled alumina was monitored by modulus and internal friction changes. For low and intermediate values of ΔT (thermal shock severity) the thermal shock damage tends to saturate at a sufficiently high number of thermal cycles. This saturation value increases as thermal shock temperature difference increases.

The relative change in internal friction versus the damage parameter ϵ can be expressed as a power law relation for each of the five thermally shocked specimens. The five Q^{-1} versus n curves in figure 25 are replotted in figure 11 in terms of ΔQ^{-1} versus ϵ damage parameter. The five internal friction curves from figure 25 thus collapse onto a single curve plot of ΔQ^{-1} versus ϵ . Thus the thermal shock damage, at least for the specimens included in this study, can be expressed as a power law function of the crack damage parameter.

A power law relationship between the saturation damage level and thermal shock difference implies a fatigue-like power law relation in

stress. The crack growth rates for the polycrystalline are far more sensitive to ΔT than SiC whisker reinforced alumina composites. The exponent in the power law was determined to be about 12 for the unreinforced polycrystalline alumina (about 2 times larger than that of SiC whisker reinforced alumina composites).

5.3. Thermal Fatigue Parameters and Their Relation to Material Parameter

Exponents p_1 and p_2 in the power law fatigue relations (equations 42 and 43) are functions of thermal shock resistance parameters such as R'''' and hence p_1 and p_2 are likely also functions of materials parameters such as fracture toughness. In addition, the thermal fatigue parameters $A\alpha$ and $B\beta$ are apparently functions of fracture toughness. The results of this study suggest the existence of threshold fracture toughness (or strength) above which thermal shock damage will not accumulate for a given ΔT (figure 34).

5.4. Effect of Quenching Media (Water, Silicone Oil and Liquid Nitrogen) on Cyclic Thermal Shock of Polycrystalline Alumina

The accumulation of thermal shock induced microcrack damage for thermally cycled alumina was monitored by modulus and internal friction changes. For liquid nitrogen quench, the thermal shock damage tends to saturate at a sufficiently high number of thermal cycles. For silicone oil quench, at low and intermediate values of ΔT (thermal shock severity) the thermal shock damage tends to saturate

at a sufficiently high number of thermal cycles. At intermediate temperature differences, a "step-type" decrement in Young's modulus was observed.

A technique for measuring the surface heat transfer coefficient, h , is developed for ceramics quenched into a water bath that combines a parameter estimation method with a fast-response capability of recently developed thin-film thermocouples. The surface heat transfer coefficient values were measured for alumina quenched into water, oil and liquid nitrogen. Measurements of heat transfer coefficient, h , for water, silicone oil and liquid nitrogen quenches show that h for silicone oil is an order of magnitude higher than for liquid nitrogen and an order of magnitude lower than for water.

The surface tensile stress developing during quenching was calculated from the measured surface heat transfer coefficient as a function of temperature using temperature dependent parameters, Young's modulus, the coefficient of thermal expansion, Poisson's ratio, and thermal shock severity. For polycrystalline alumina, plots of $\ln(A/E_0)$ versus $\ln(\sigma_{\max}/\sigma_f)$ for water and silicone quench show very different slopes (figure 53). However these two different curves collapse into a single curve if we consider the duration time as well as the stress. A plot of normalized damage, A/E_0 versus the product between stress $\sigma(f)$ and duration time Δf for the polycrystalline alumina quenched into water and oil bath (figure 54a and 54b) shows a power law relation (equation 74) which is similar to the power law equation in A/E_0 and $\Delta T/T_r$ (equation 37 in Section 4.1.7.).

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**THERMAL FATIGUE IN CERAMICS
AND CERAMIC MATRIX COMPOSITES**

Volume II

By

WON JAE LEE

A Dissertation

**Submitted to
MICHIGAN STATE UNIVERSITY
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APPENDICES

Appendix A. Young's Modulus, Shear modulus and Internal Friction Calculation

The modulus program calculates the Young's modulus and shear modulus of bar shape specimen from the flexural resonance frequency and resonance torsional frequency (Section 3.2.).

```

20 REM Young's modulus and Shear modulus Calculation
25 REM
30 DIM L$(90),H$(90),L1$(90),DT$(90)
35 CLS:PRINT " "
100 PRINT"Do you want to estimate the flex and tors freq given A"
105 INPUT "given a set of dimensions? (input yes or no) ",Q$
120 PRINT " "
150 REM
160 IF Q$="yes" THEN GOSUB 7000: IF Q$ = "yes" THEN END
230 PRINT"how many pairs of flexural and torsional frequencies"
240 INPUT" do you wish to calculate for this specimen? ",NN
250 FOR LL = 1 TO NN
260 PRINT " ": INPUT"specimen label ",L$
400 INPUT "Delta temperature ",H$
410 INPUT"Date and time of experiment ",DT$
420 CLS
510 PRINT" "
910 REM
920 REM
930 REM
940 IF LL >1 THEN GOTO 1000
950 PRINT "Specimen: ",L$:PRINT "At delta temperature ",H$:
960 PRINT"Date and time of experiment ",DT$: PRINT " "
1000 IF LL = 1 THEN INPUT "mass in grams ",M1
1020 IF LL = 1 THEN INPUT "thickness in cm ",T1
1035 IF LL = 1 THEN INPUT "width in cm ",W1
1040 IF LL = 1 THEN INPUT "length in cm ",L1:PRINT " "
1045 REM r1 is the initial guess for Poisson's ratio
1050 R1 = .25
1052 IF LL = 1 THEN GOTO 1060
1055 PRINT "Specimen: ",L$:PRINT "at delta temperature ",H$:
1058 PRINT"Date and time of measurement ",DT$
1060 INPUT"flexural frequency in hz ",F2
1080 INPUT"torsional frequency in hz ",T2
1090 PRINT" "
1094 REM
1096 REM
1097 REM

```



```

1100 REM Program will give an estimate of torsional frequency,when
1110 REM it is unknown.
1120 REM
1200 REM Let A = thickness/width and B1 = thickness/length
1210 A=T1/W1 :B=T1/L1
1220 REM shape factor R
1230 R=(A+(1/A))/((4*A)-2.52*(A^2)+.21*(A^6))
1240 REM approx bulk density D9=(mass/thick*width*length)
1250 D9=M1/(T1*W1*L1)
1260 REM
1270 REM
1280 REM let C = cross sectional area
1290 C=T1*W1
1295 IF T2=1 THEN GOTO 3000
1300 REM calculate shear modulus G
1310 G=(4*L1*R*M1*(T2^2))/C
1450 S1=1.1
1500 IF ABS(S1-1)<.000001 THEN GOTO 2500
1510 REM C1 is a shape factor that will be calculated
1520 REM in terms of four subfactors designated by I.M.N.P
1600 O5=R1
1620 GOSUB 4000
1630 R1=(Y/(2*G))-1
1640 B5=(Y*G)/(3*((3*G)-Y))
1650 S1=O5/R1 :GOTO 1500
2300 REM
2400 REM
2410 REM
2420 REM
2500 REM output result
2540 PRINT "Approximate bulk density (gm/cm3) ",D9
2545 PRINT "Young's modulus (dyne/cm2) ",Y
2550 PRINT "Shear modulus (dyne/cm**2) ",G
2560 PRINT "Poision's ratio ",R1
2570 PRINT "Bulk modulus ( dyne/cm**2) ",B5
2580 PRINT " "
2600 PRINT "*****":PRINT " "
2700 GOSUB 8000
2900 REM
2905 REM
2910 REM
3000 REM Estimate of torsional frequency
3010 R1=.25
3020 GOSUB 4000
3025 G=Y/(2*(R1+1))
3030 T9=SQR((G*C)/(4*L1*R*M1))
3050 REM PRINT "Estimated torsional frequency in hz " ,T9
3060 REM PRINT "Estimated Young's modulus (dyne/cm**2) ",Y
3600 REM
3610 REM

```

```

3620 REM
3990 NEXT LL
3995 END
4000 REM Shape factor C1
4010 L=1+6.585*(1+.075*O5+.8109*O5^2)*R^2
4020 M=-100.083*(1+.2023*O5+2.173*O5^2)*B^4
4030 N=12+76.06*(1+.14081*O5+1.536*O5^2)*B^2
4040 P=.868*B^4
4050 C1=(1+(M/N)+P)*B^-3
4060 Y=(.94645*C1*M1*(F2^2))/W1
4210 REM
4990 RETURN
5000 REM Calculation of estimated flexural and torsional frequencies
5005 REM given dimensions, estimated density, and estimated Young's
      modulus
5010 REM
5020 REM
5110 INPUT "Estimated Young's modulus (dyne/cm**2) " ,Y3
5120 INPUT "Estimated bulk density (gm/cm**3) " ,D3
5130 M3=T1*W1*L1*D3
5140 G=Y3/(2*(R1+1))
5160 REM Shape factor C1
5170 L=1+6.585*(1+.075*O5+.8109*O5^2)*R^2
5180 M=-100.083*(1+.2023*O5+2.173*O5^2)*B^4
5190 N=12+76.06*(1+.14081*O5+1.536*O5^2)*B^2
5200 P=.868*B^4
5210 C1=(1+(M/N)+P)*B^-3
5220 REM
5230 REM
5260 F3=SQR((Y3*W1)/(.9464999*C1*M3))
5270 T3=SQR((G*C)/(4*L1*R*M3))
5310 PRINT "Estimated flexural frequency in Hz " ,F3
5330 PRINT "Estimated torsional frequency in Hz " ,T3
5500 RETURN
7000 REM
7020 INPUT "thickness in cm " ,T1
7035 INPUT "width in cm " ,W1
7040 INPUT "length in cm " ,L1:PRINT " "
7050 R1=.25
7090 PRINT" "
7094 REM
7096 REM
7097 REM
7200 REM
7210 A=T1/W1 :B=T1/L1
7220 REM shape factor R
7230 R=(A+(1/A))/((4*A)-2.52*(A^2)+.21*(A^6))
7290 C=T1*W1
7295 GOSUB 5000
7900 RETURN

```



```

8000 REM print results to line printer
8100 LPRINT " "
8120 LPRINT "Specimen: ",L$:LPRINT "at delta temperature ",H$
8140 LPRINT "date and time of experiment",DT$:LPRINT " "
8300 LPRINT "Mass in grams ",M1
8320 LPRINT "Thickness in cm ",T1
8335 LPRINT "Width in cm ",W1
8340 LPRINT "Length in cm ",L1:LPRINT " "
8360 LPRINT "Flexural frequency in hz ",F2
8380 LPRINT "Torsional frequency in hz ",T2:LPRINT " "
8500 REM output result
8510 REM PRINT DATE$
8540 LPRINT "Approximate bulk density (gm/cm3) ",D9
8545 LPRINT "Young's modulus (dyne/cm2) ",Y
8550 LPRINT "Shear modulus (dyne/cm**2) ",G
8560 LPRINT "Poisson's ratio ",R1
8570 LPRINT "Bulk modulus ( dyne/cm**2) ",B5
8580 LPRINT " "
8600 LPRINT "*****":PRINT " "
8900 REM
8905 REM
8910 REM
8990 RETURN

```

This program is a non-linear least-squares fitting program for determining specimen suspension effects on internal friction data. Measured internal friction and specimen support position data is fit to equation 7 in section 3.3..

```

10000 REM *****
12000 REM Non-linear least-squares fitting program for determining
12020 REM specimen suspension effects on internal friction data.
12040 REM Internal friction and specimen support position data is
12060 REM fit to equation (4), page 519 of J. B. Wachtman, Jr. and
12080 REM W. E. Tefft, "Effect of Suspension Position on Apparent
12100 REM Values of Internal Friction Determined by Forster's
12120 REM Method", Review of Scientific Instruments, 29: 517-520,
14000 REM 1958.
14020 REM *****
14040 REM
16000 N=15 : REM number of iteration
16010 C1=.00003 : REM c1 is a convergence parameter
16060 I1=1 : I2=N : C2=3
16110 DIM A(C2,C2),B(C2),V(C2),D(C2,I2)

```

```

16120 DIM C(C2),Y(I2),X(I1,I2),C3(C2)
16122 DIM XX(N), XR(N), XF(N), YR(N), Z(N), COSH(N), SINH(N)
16124 DIM QM(N), QA(N)
16200 DEF FNY(I)=(C(1) + (X(1,I)*C(2)*C(3)))/(1! + C(2)*X(1,I))
16210 DEF FNW(I)=1
16230 GOSUB 18000 : REM read data and X matrix
16240 GOSUB 17310 : REM standard deviation of Y's
16243 REM
16245 FOR LL=1 TO 15
16250 GOSUB 16310
16260 GOSUB 16420
16270 GOSUB 16540
16280 GOSUB 16660
16290 GOSUB 17070
16300 NEXT LL
16301 REM
16302 GOSUB 19000: GOSUB 20000 : GOSUB 21000 :REM print results
16305 END
16307 REM
16309 REM
16310 REM *** calculating the derivative ***
16320 FOR K=1 TO C2
16330 FOR I=1 TO I2
16340 Y = FNY(I)
16350 C(K)=C(K)*(1.001)
16360 Y5=FNY(I)
16370 D(K,I)=(Y5-Y)/(C(K)*.001)
16380 C(K)=C(K)/1.001
16390 NEXT I
16400 NEXT K
16410 RETURN
16414 REM
16416 REM
16420 REM *** first initialize the [a] and [b] matrices ***
16430 FOR K=1 TO C2
16440 FOR J=1 TO C2
16450 B(J)=0 : A(J,K)=0
16460 NEXT J
16470 NEXT K
16480 FOR J=1 TO C2
16490 FOR I=1 TO I2
16500 B(J)=B(J)+(Y(I)-FNY(I))*D(J,I)
16510 NEXT I
16520 NEXT J
16530 RETURN
16534 REM
16538 REM
16540 REM *** calculating the sums ***
16550 FOR K=1 TO C2
16560 FOR J=1 TO C2

```

```

16570     FOR I=1 TO I2
16580     A(K,J)=A(K,J)+FNW(I)*D(K,I)*D(J,I)
16590     NEXT I
16600     NEXT J
16610     NEXT K
16620     FOR J=1 TO C2
16630     A(J,J)=(A(J,J))*(1+C1)
16640     NEXT J
16650     RETURN
16660     N=C2 : Q=0 : V=0
16670     N1=N-1 : J=1
16680     FOR I=1 TO N1
16690     L1=0
16700     FOR P=1 TO N
16710     G=ABS(A(P,I))
16720     IF(G>L1)THEN L1=G : J=P
16730     NEXT P
16740     IF(L1=0)THEN I=N1 : NEXT I
16750     IF(L1=0)THEN RETURN
16760     FOR K=I TO N
16770     T1=A(I,K)
16780     A(I,K)=A(J,K)
16790     A(J,K)=T1
16800     NEXT K
16810     T1=B(I)
16820     REM
16830     B(I)=B(J)
16840     B(J)=T1
16850     I5=I+1
16860     FOR K=I5 TO N
16870     Q=-A(K,I)/A(I,I)
16880     A(K,I)=0
16890     B(K)=Q*B(I)+B(K)
16900     FOR J=I5 TO N
16910     A(K,J)=Q*A(I,J)+A(K,J)
16920     NEXT J
16930     NEXT K
16940     REM NEXT I
16950     IF(A(N,N))=0 THEN RETURN
16960     V(N)=B(N)/A(N,N)
16970     N6=N+1
16980     FOR K=1 TO N1
16990     Q=0
17000     N5=N-K
17010     FOR J=1 TO K
17020     Q=Q+A(N5,N6-J)*V(N6-J)
17030     NEXT J
17040     V(N5)=(B(N5)-Q)/A(N5,N5)
17050     NEXT K
17060     RETURN

```

```

17065 REM
17068 REM
17070 REM update coefficients c(i) and print results
17080 PRINT" "
17090 FOR I=1 TO C2
17100 C(I)=C(I)+V(I)
17110 PRINT"c(",I,")-",C(I)
17120 NEXT I
17130 REM calculating the variance
17140 FOR I=1 TO I2
17150 V=V+FNW(I)*(Y(I)-FNY(I))^2
17160 NEXT I
17170 REM r2 is the sqr of the correlation coefficient
17180 R2=1-(V/I2/V5)
17190 PRINT"corr. coeff. sqr",R2
17200 IF R2<R3 THEN 17250
17210 FOR J1=1 TO C2
17220 C3(J1)=C(J1)
17230 NEXT J1
17240 R3=R2
17250 PRINT"best so far",R3
17260 FOR I=1 TO C2
17270 PRINT"c(i)",I,")= ",C3(I)
17280 NEXT I
17290 PRINT" " : PRINT"best fit corr. sqr=",R3
17300 RETURN
17305 REM
17310 REM
17350 REM std dev of y's
17360 FOR I=1 TO I2
17370 V1=Y(I)+V1 : V2=Y(I)^2+V2
17380 NEXT I
17390 V5=V2/I2-(V1/I2)^2
17400 RETURN
18000 REM
18020 REM
18040 REM *** read data and make X matrix
18080 A0 = 1.018 : B0 = 2.036
18085 REM l$ is the specimen label, i2 is the number of data pairs,
18087 REM and L0 is the length of the specimen in cm.
18091 READ l$ : READ I2 : READ L0 : MIN = 99999!
18160 FOR J = 1 TO I2
18180 READ Y(J),XX(J) :
      REM Vector y contains measured internal friction.
18185 REM Vector xx contains the position data (position,
18187 REM in cm of the support threads on the specimen.
18190 IF Y(J) < MIN THEN MIN = Y(J)
18220 XR(J) = (4.73*XX(J))/L0
18240 REM XF(J) = XX(J)/L0
18260 NEXT J

```

```

18280      C(1) = MIN : C(2) = .01 : C(3) = MIN/C(2)
18340 FOR M = 1 TO I2
18360      Q = XR(M)
18380      COSH(M) = .5*(EXP(Q) + EXP(-Q))
18400      SINH(M) = .5*(EXP(Q) - EXP(-Q))
18420      Y1 = A0*(COSH(M) + COS(Q))
18440      Y2 = SINH(M) + SIN(Q)
18460      YR(M) = (Y1 - Y2)/B0
18480      X(1,M) = YR(M) *YR(M)
18500 NEXT M
18520 RETURN
18540 REM
18580 REM
19000 REM print results on CRT
19010 REM
19030 IF (C(1) < 0 OR C(2) < 0 OR C(3) < 0) THEN PRINT
      "NEGATIVE PARAMETERS are silly"
19400 REM
19420 PRINT "Internal friction parameters for specimen ",L$
19440 PRINT "Parameters obtained from non-linear least squares"
19450 PRINT "where the number of internal friction-
      position data pairs = ",I2
19460 PRINT " " : PRINT " "
19480 PRINT " Specimen internal friction, Qs-1 = ",C(1)
19500 PRINT " Energy ratio, k = ",C(2)
19520 PRINT " Apparatus internal friction, Qa-1 = ",C(3)
19530 PROD = C(2)*C(3) : PRINT " Product k*Qa-1 = ",PROD
19535 PRINT " Correlation coefficient = ",R3
19540 PRINT " ": PRINT"*****":PRINT " "
19600 IF R3 < .95 THEN PRINT"correlation coefficient unacceptable"
19990 RETURN
19994 REM
19996 REM
20000 REM Print results to the line printer
20010 IF R3 < .95 THEN RETURN
20030 IF (C(1) < 0 OR C(2) < 0 OR C(3) < 0) THEN RETURN
20400 REM
20420 LPRINT "Internal friction parameters for specimen ",L$
20440 LPRINT "Parameters obtained from non-linear least squares"
20450 LPRINT "where the number of internal friction-
      position data pairs = ",I2
20460 LPRINT " " : LPRINT " "
20480 LPRINT " Specimen internal friction, Qs-1 = ",C(1)
20500 -21000
20520 LPRINT " Apparatus internal friction, Qa-1 = ",C(3)
20530 LPRINT " Product k*Qa-1 = ",PROD
20535 LPRINT " Correlation coefficient = ",R3
20540 LPRINT " ": LPRINT"*****":LPRINT " "
20990 RETURN
20994 REM

```



```

20996 REM
21000 REM *** print Qm-1(exp), Qm-1(theo) and difference
21010 REM
21020 LPRINT " ": LPRINT " "
21040 A$ = " ##.###      ##.###      ##.####      ##.#### "
21060 LPRINT " x(cm)    Qm-1(exp)  Qm-1(theo)  Diff " : LPRINT " "
21080 SUM = 0!
21100 FOR I = 1 TO I2
21120     THEO(I)=(C(1) + (X(1,I)*C(2)*C(3)))/(1! + C(2)*X(1,I))
21140     DIFF(I) = (Y(I) - THEO(I))/Y(I)
21160     LPRINT USING A$;XX(I),Y(I),THEO(I),DIFF(I)
21180     SUM = ABS(SUM) + ABS(DIFF(I))
21200 NEXT I
21220 AD = SUM/I2 : REM Avediff is the average residual
21240 LPRINT " ": LPRINT "avediff = ",AD : LPRINT " "
21260 LPRINT "The data for which the residuals exceed the mean value"
21280 LPRINT " of the residual are: " : LPRINT " "
21300 B$ = " ##.###      ##.###      ##.###      ##.####"
21320 LPRINT " x(cm)      Qm-1(exp)  Qm-1(theo)  Difference
21340 FOR L = 1 TO I2
21360     IF ABS(DIFF(L)) > AD THEN LPRINT
                USING B$;XX(L),Y(L),THEO(L),DIFF(L)
21380 NEXT L
21400 LPRINT " ": LPRINT " "
30000 REM
30050 REM
40000 DATA Internal friction Wachtman and Tefft
40020 DATA 4
40030 DATA 1.0
40040 DATA 5.4,0.0667
40060 DATA 1.1,0.1667
40100 DATA 1.8,0.3330
40110 DATA 2.0,0.1333

```

Appendix B Slow Crack Growth

As noted in the Section 4.1.2., some authors equate "thermal fatigue" with the environmentally assisted slow crack growth that can occur during cyclic stressing of a ceramic [B1, B2]. On the other hand, Evans separates environmentally assisted slow crack growth from fatigue [B3].

Slow crack growth has been observed for polycrystalline alumina under quasistatic loading [B4]. However, for unreinforced polycrystalline alumina, several researchers have observed a degradation of mechanical properties during cyclic loading. These authors [B5, B6-B8, B9] each claim that the crack damage during cyclic loading of the alumina was not due the slow crack growth, but rather was due to a "true" fatigue effect, in that crack extension was affected by the cyclic nature of the stress instead of the stress-crack tip chemistry interaction that characterizes slow crack growth.

The thermal shock work of Bradt et al. on polycrystalline alumina [B9] includes key results on thermal shock damage and slow crack growth. Sets of 100 thermally shocked specimens were divided into two subsets of 50 specimens each, with one subset fractured in bend in a room temperature water bath and the other subset of 50 specimens fractured in a liquid nitrogen bath (77°K) [B9]. The liquid nitrogen inhibited slow crack growth during the retained strength fracture testing. The probability of failure as a function of the quench temperature difference ΔT was found to be very similar for alumina

specimens fractured in four point bend in a room temperature water bath and in liquid nitrogen, indicating that slow crack growth was not significant during the retained strength testing [B9].

The thermal shock itself applies a tensile stress under very high stress rate conditions, during which slow crack growth should be minimal. For alumina specimens of a similar size to those in this study, thermal shock into a water bath with comparable quench temperature differences, specimen surface stresses reached a maximum within only 60 msec after the initial instant of immersion [B9]. Furthermore, acoustic emission measurements performed by Bradt et al. on their polycrystalline alumina specimens indicated that only about 10 to 20 msec elapsed from the instant of quenching until the first cracks propagated, again for quench temperature differences comparable to this study [B9].*

* Bradt et al. [B9] specimens' were alumina cylinders, however the characteristic dimensions of the bar specimens included in this study were similar to Bradt's specimens. In particular, the diameter of the Bradt's specimens was 4 mm, while the thickness of the bars in this study were about 3.0 mm. In terms of the temporal development of thermal gradients, Bessel functions characteristic of cylindrically bound media will be replaced by Fourier series characteristic of rectilinearly bound media as one passes from cylindrical specimens to prismatic specimens [B9], thus the times characterizing the temperature change for Bradt's 4.0 mm diameter alumina cylinders will be similar to the times characterizing the 3.0 mm thick alumina bars in this study. However, the details of the stress fields produced by the thermal transients will differ considerably for the two specimen geometries [B10].

Appendix B References

- B1. D. P. H. Hasselman, R. Badalian, and E. P. Chen, "Thermal Fatigue and Its Failure Prediction for Brittle Ceramics", pp. 55-68 in Thermal Fatigue of Materials and Components, ASTM 612, D. A. Spears and D. F. Mowbray, editors, American Society for Testing and Materials, Philadelphia, PA, 1976.
- B2. D. P. H. Hasselman, "Thermal Stress Resistance of Engineering Ceramics", *Mater. Sci. and Eng.*, 71: 251-264, 1985
- B3. A. G. Evans, "Fatigue in Ceramics", Lawrence Berkeley Laboratory Report LBL-9529, 1979.
- B4. R. F. Pabst and G. Popp, "Critical and Subcritical Crack Extension in Alumina and Silicon Carbide as Functions of Temperature, Environment and Loading Rate", pp. 305-315 in Fracture Mechanics of Ceramics. Vol. 5, edited by R. C. Bradt, A. G. Evans, D. P. H. Hasselman, and F. F. Lange, Plenum Press, NY, 1983.
- B5. F. Guiv, "Cyclic Fatigue of Polycrystalline Alumina in Direct Push-Pull", *J. Mater. Sci.*, 13: 1357-1361, 1978.
- B6. D. Lewis, "Cyclic Mechanical Fatigue in Ceramic-Ceramic Composites--An Update", *Ceram. Eng. Sci. Proc.*, 4 [9-10]: 874-881, 1983.
- B7. D. Lewis and R. W. Lewis, "Thermal Shock Fatigue of Monolithic Ceramics and Ceramic-Ceramic Composites", *Ibid.*, 2 [7-8]: 712-718, 1981.
- B8. D. Lewis and R. W. Rice, "Comparison of Static, Cyclic, and Thermal Shock Fatigue in Ceramic Composites", *Ibid.*, 5 [9-10]: 714-721, 1982.
- B9. W. P. Rogers, A. F. Emery, R. C. Bradt, and A. S. Kobayashi, "Statistical Study of Thermal Fracture of Ceramic Materials in the Water Quench Test", *Ibid.*, 70 [6]: 406-412, 1987.
- B10. B. A. Boley and J. H. Weiner, Theory of Thermal Stresses, John Wiley and Sons, New York, 1960.

Appendix C Ratio of Third Moment and the Second Moment of the Lognormal and Rayleigh Probability Density Functions

In section 4.1.6., it was shown that the ratio of the third and second moments of the gamma distribution is given by a constant which depends on the "shape" parameters of the distribution. This ratio of the third and second moments of a distribution has implications concerning such properties as the crack damage parameter (which is proportional to the third moment of the crack size distribution) and the crack area (which is proportional to the second moment of the crack size distribution). In this appendix, we shall extend those results to two additional distributions, namely the Rayleigh and the lognormal distributions, since grain size distribution in a polycrystalline material have been discussed in terms of Rayleigh or lognormal distribution [C1]. A general form of the probability density function for the Rayleigh distribution (figure C1), $p_r(a)$ is

$$p_r(a) = \rho x \exp(-\rho x^2) \quad \text{for } x \geq 0 \quad (C1)$$

For the Rayleigh pdf, the second moment m_2 and third moment m_3 are

$$m_2 = 2 \int_0^{\infty} \rho x^3 \exp(-\rho x^2) dx = 1/\rho \quad (C2)$$

and

$$m_3 = 2 \int_0^{\infty} \rho x^4 \exp(-\rho x^2) dx = (3/2)(1/\rho)(\pi/4\rho)^{1/2} \quad (C3)$$

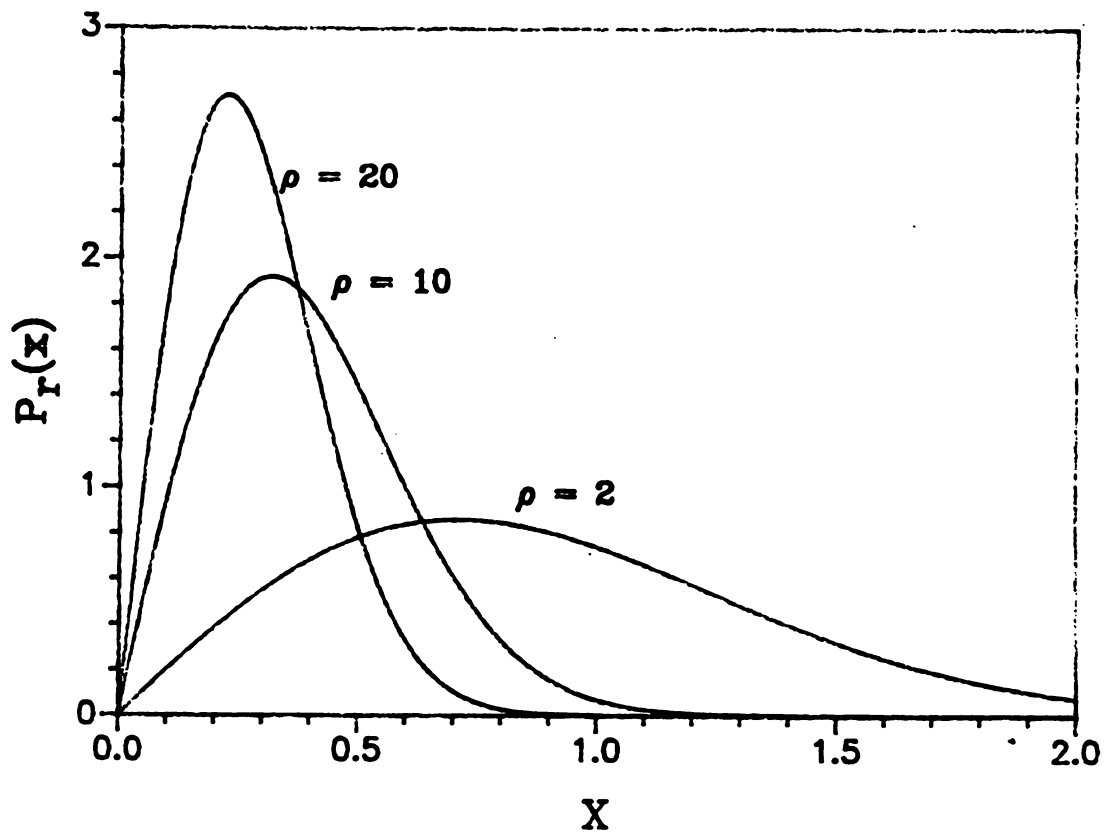


Figure C1. Plot of Rayleigh distribution functions for a range of values of ρ .

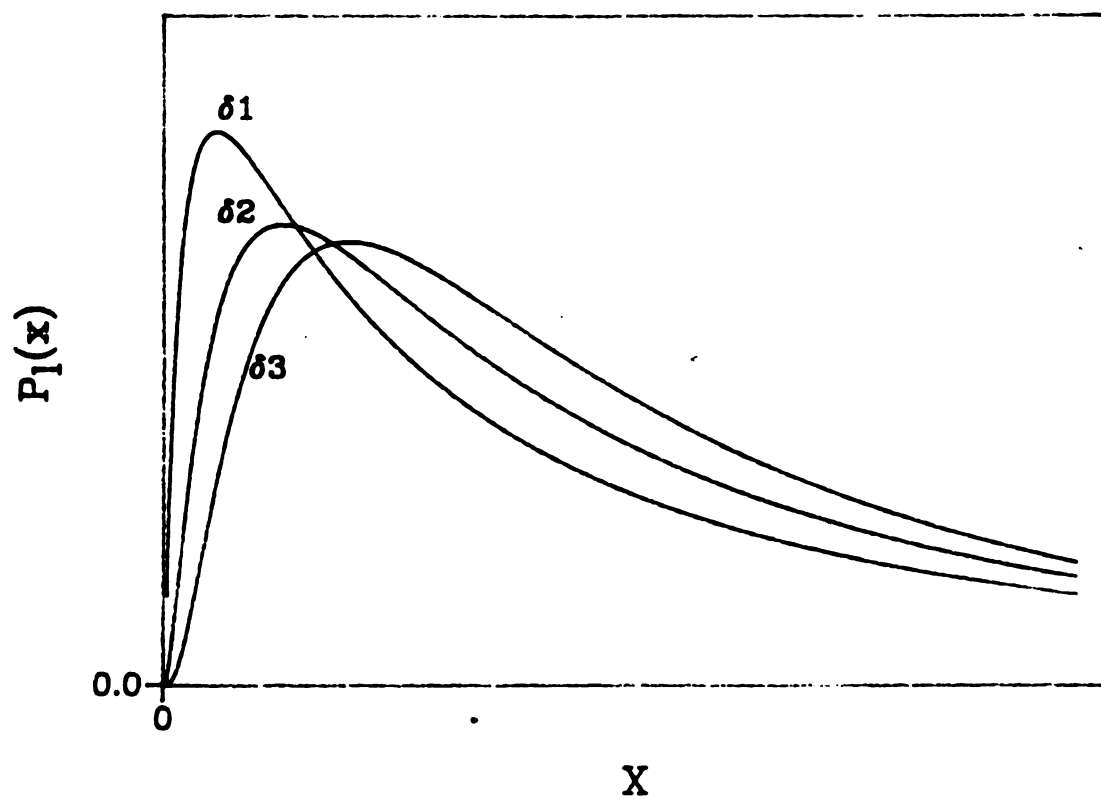


Figure C2. Plot of lognormal distribution function as a function of δ .

Thus the ratio m_3/m_2 is $(3/2) (\pi/4\rho)^{1/2}$. The lognormal distribution function (figure C2) $p_1(a)$ is given by

$$p_1(a) = (2\pi\delta^2)^{-2/1} D^{-1} \exp[-(\ln a - \mu)^2 / 2\delta^2] \quad (C4)$$

where δ - the standard deviation of $\ln(a)$

μ - the value of $\ln(a)$ averaged over the pdf.

The j th moment of the lognormal distribution is given by

$$\langle a^j \rangle = a_{\text{med}}^j \exp[j^2 \delta^2 / 2] \quad (C5)$$

where a_{med} is the median value of a . The ratio of the third and second moments of the lognormal distribution is given by

$$m_3/m_2 = a_{\text{med}} \exp(5 \delta^2 / 2) \quad (C6)$$

Thus the ratio of the third and second moments of the Rayleigh and lognormal distributions are both functions of the shape parameters of the distribution.

Appendix C References

- C1. S. K. Kurtz and F. M. A. Carpay, "Microstructure and Normal Grain Growth in Metals and Ceramics", J. Appl. Phys., 51 [11]: 5745-5754, 1981.

Appendix D. Moduli and Internal Friction as a Function of Porosity for Polycrystalline Alumina

Generally, the modulus decreases and internal friction increases as the porosity increases. In this study, the volume fraction porosity, P , of the polycrystalline alumina specimens ranged from 0.03 to 0.074. The measured elastic modulus for each of the undamaged polycrystalline alumina specimen used in this study are not identical because of the porosity differences among the specimens. In this appendix, we show that these differences can be treated in terms of well-known functional forms for modulus-porosity relations. Using these modulus-porosity relations, we extrapolate to zero porosity and find that the unshocked, zero porosity extrapolations of modulus agree well with the elastic moduli calculated from single crystal data.

Modulus-porosity relations for these alumina specimens were adequately represented by the following semi-empirical exponential forms

$$E = E_0 \exp(-aP) \quad (D1)$$

$$G = G_0 \exp(-bP) \quad (D2)$$

where E_0 , G_0 - the Young's modulus and shear modulus of
nonmicrocracked, theoretically dense alumina
 P - volume fraction porosity
 a , b - empirical constants.

For small P values, the empirical exponential equations can be approximated by linear functions, which are

$$E/E_0 = 1 - cP \quad (D3)$$

$$G/G_0 = 1 - dP \quad (D4)$$

where c, d = the porosity-modulus slope.

The results of least-squares best fit to equations D1 - D4 are listed in table D1 and shown in figures D1 and D2. In all cases, the correlation coefficients are greater than 0.91.

The expected bounds for the Young's modulus and shear modulus of a theoretically dense polycrystalline bodies can be calculated from the elasticity data of that single crystal. The Voigt and Reuss bounds calculated from measurements on single crystal alumina [D1] (Table D2) are upper and lower bounds of the theoretically dense materials' elastic constants. Since these bounds are calculated from single crystal data, these aggregate bounds may be used to estimate the elastic moduli of theoretically dense polycrystalline specimens. The least-square best fit to the exponential equations D1 and D2 differs by 6.8 percent and 4.8 percent with the E_0 and G_0 from mean of Voigt and Reuss bounds (Table D1 and D2). In contrast, the least-square best fit to the linear equations D3 and D4 yields $E_0 = 374.25$ GPa and $G_0 = 154.81$ GPa, which differs by 6.9 percent and 3.6 percent

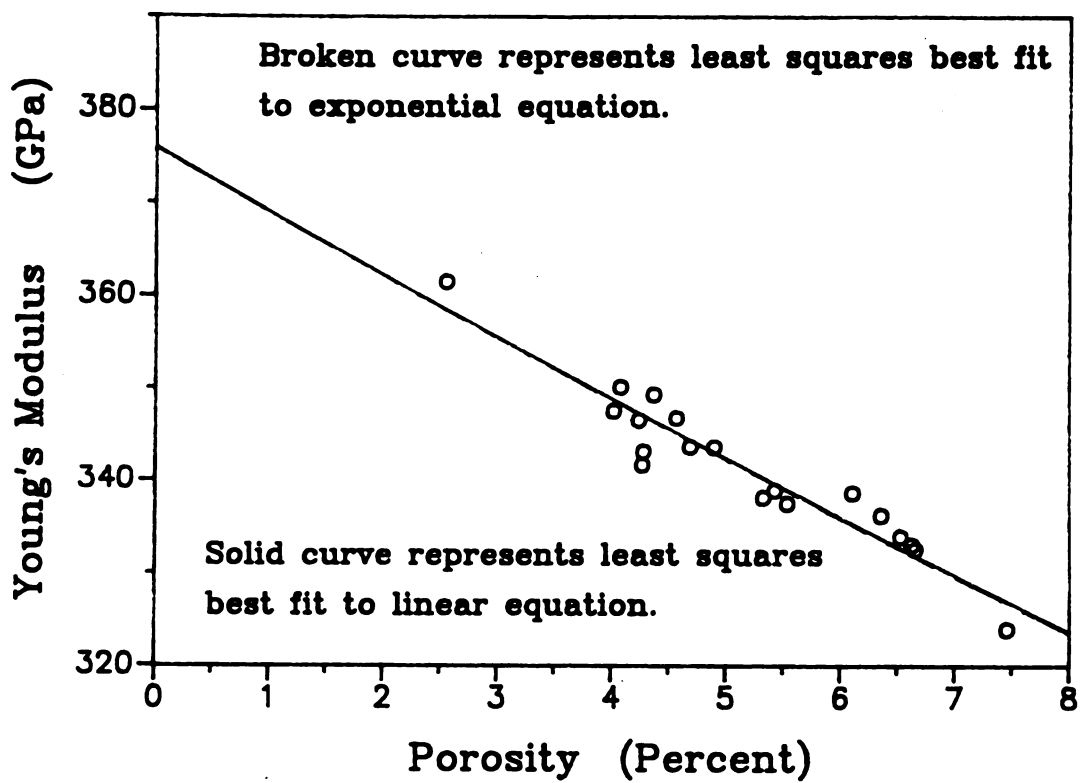


Figure D1. Young's modulus of the alumina as a function of porosity prior to thermal shock damage.

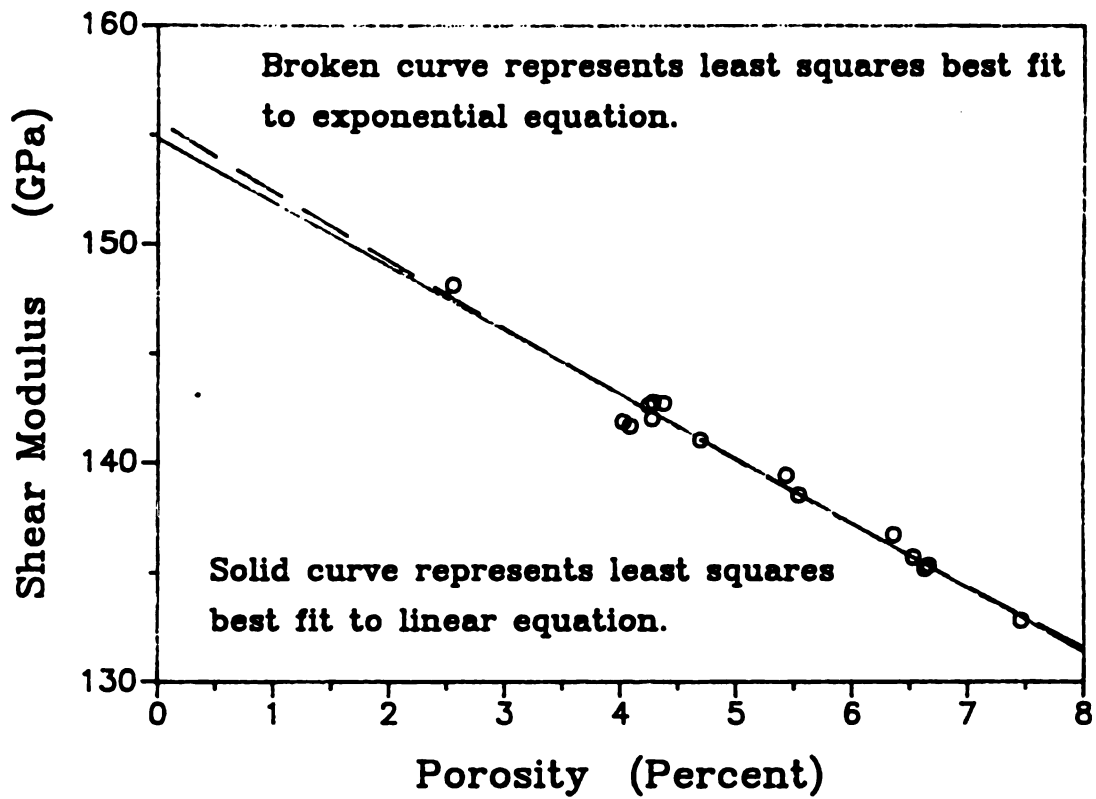


Figure D2. Shear modulus of the alumina as a function of porosity prior to thermal shock damage.

Table D1. Results of Least Squares Best Fit to Porosity-Modulus Equation

	Exponential form (eq. D1 and D2)	Linear form (eq. D3 and D4)
E_o (GPa)	375.9	374.3
Constant a, c	0.019	6.37
Cor. Coeff.	0.91	0.91

G_o (GPa)	155.7	154.8
Constant b, d	0.021	2.93
Cor. Coeff.	0.98	0.98

ν_o	0.229	0.222

Table D2. The Voigt and Reuss Bounds Calculated from Measurements on Single Crystal Alumina [D1] along with the Arithmetic Mean of Those Bounds for the Elastic Moduli

Bound Type	Young's modulus	shear modulus	Poisson's ratio
Voigt	408.5 GPa	165.7 GPa	0.223
Reuss	398.3 GPa	160.7 GPa	0.239
Mean of Voigt and Reuss bounds	403.4 GPa	163.2 GPa	0.236

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with the E_0 and G_0 from mean of Voigt and Reuss bounds (Table D1 and D2).

In the present study, the internal friction of the alumina specimens showed a linear dependence on porosity (figure D3) for the restricted range of porosity exhibited by the specimens in this study.

In studies of polycrystalline yttria [D2] and magnesia [D3], Marlowe [D2] and Hanna et al. [D3] fit their internal friction data to a power function of porosity, P , such as

$$Q^{-1} = F P^r \quad (D5)$$

where F , r - empirical constant.

For Marlowe's data on polycrystalline yttria, F and r are 0.009 and 1.925, respectively. However, for a zero value of porosity, Q^{-1} will not be zero, as predicted by equation A5. There are a number of physical mechanisms other than porosity that contribute to internal friction [D4, D5]. Thus equation D5 should be modified as follows

$$Q^{-1} = Q_0^{-1} + F' P^{r'} \quad (D6)$$

where Q_0^{-1} - zero porosity internal friction

F' , r' - empirical constants.

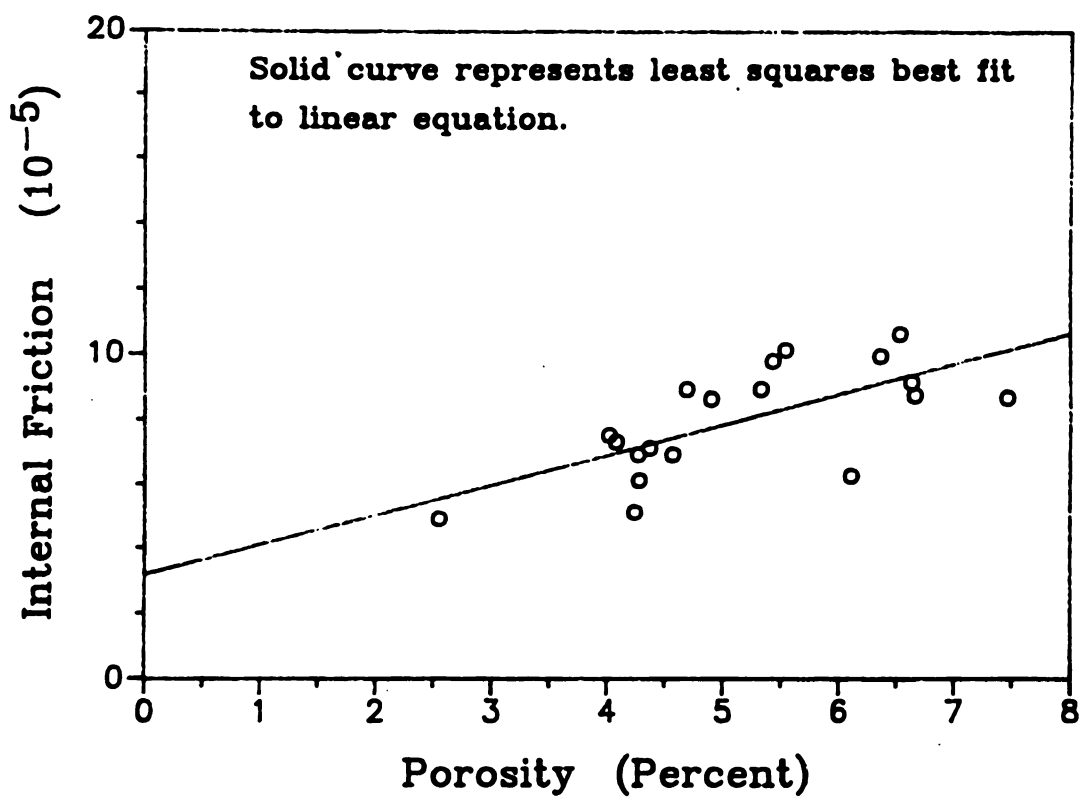


Figure D3. Internal friction of the polycrystalline alumina as a function of porosity.

Figure D4 shows a least-squares best fit of revised equation D6 to Marlowe's internal friction data. In the present study, the porosity range was so narrow (3 to 7.5 percent) that the effect of porosity on internal friction can't be monitored well by equation D6 (figure D3).

Appendix D References

- D1. G. Simmons and H. Wang, Single Crystal Elastic Constants and Calculated Aggregate Properties: A Handbook, The M. I. T. Press, Cambridge, MA, 1971.
- D2. M. O. Marlowe, "Elasticity and Kilocycle Internal Friction of Y_2O_3 ", M. S. Thesis, Iowa State University, Ames, Iowa, 1963.
- D3. R. Hanna and W. B. Crandall, "The Young's Modulus and Internal Friction of Polycrystalline MgO at Room Temperature", U. S. Atomic Energy Commission Report AROD-2891.1., Army Research Office, Durham, NC, 1961. (cited from ref. D2)
- D4. C. Zener, Elasticity and Anelasticity of Metals, University of Chicago Press, Chicago, 1948.
- D5. T. S. Ke, "Internal Friction and Precipitation from the Solid Solution of Nitrogen in Tantalum", Physical Review [74]: 914-916, 1948.

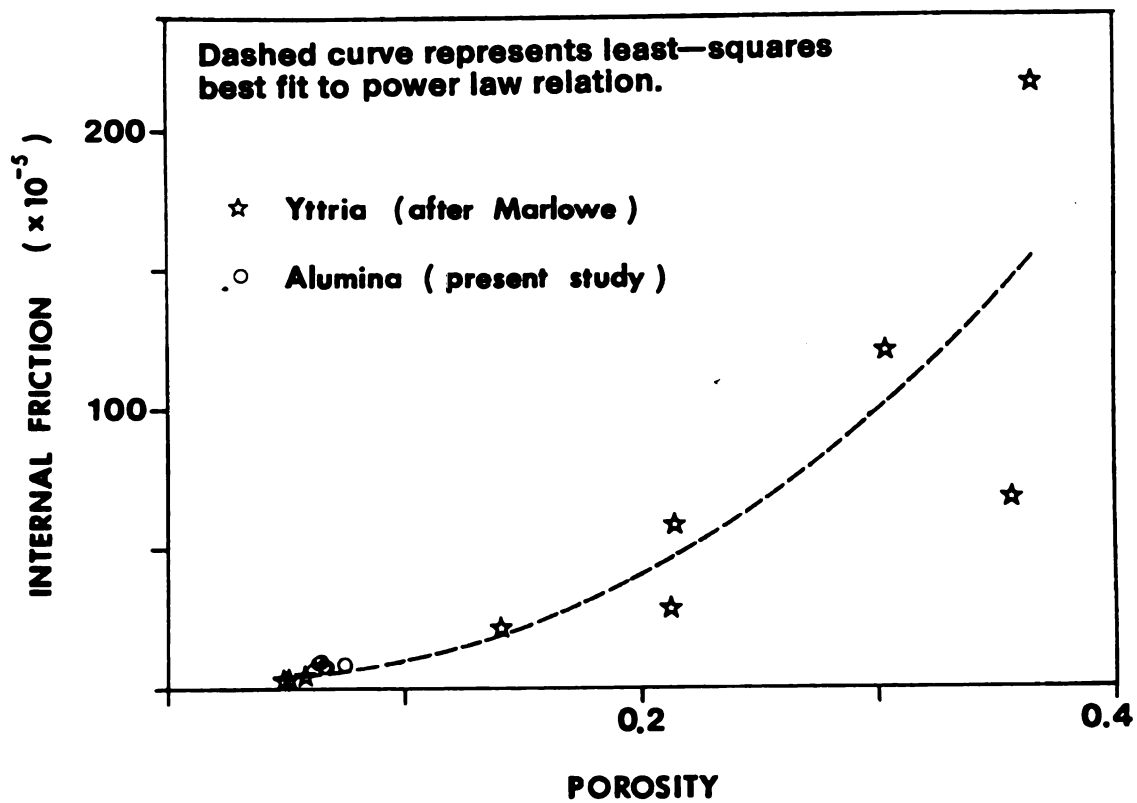


Figure D4. Internal friction of the Y_2O_3 as a function of porosity (after Marlowe [D2]).

Appendix E Maximum Tensile Thermal Stresses at the Surface During Thermal Shock

The maximum actual thermal stresses at the surface of the plate can be evaluated by

$$\begin{aligned}\sigma_{\max} &= [E\alpha\Delta T/(1-\nu)] \times \sigma_{\max}^* \\ &= [E\alpha\Delta T/(1-\nu)] \times [1.5 + 3.25/N_{bi} - 0.5\exp(-16/N_{bi})]\end{aligned}\quad (E1)$$

Equation E1 indicates that the maximum thermal stress depends on a number of material properties, the dimensions of the specimen and the surface heat transfer coefficient. If all these material properties and heat transfer coefficient are known, then it is relatively simple to calculate the maximum tensile thermal stress at the surface of the specimen. However, the material properties are not temperature independent but temperature-dependent. The importance of the temperature-dependence of the material properties was reported in study of transient thermal stress intensity calculations [E1]. Hasselman et al. [E2] reported that the usual assumption that the material properties and heat transfer coefficient are constant* may result in significant errors in the quantitative interpretation of thermal shock results. Thus we calculate the maximum tensile thermal stress at the surface by considering the temperature dependent

* The constant value selected is typically the room temperature value of the material parameters and heat transfer coefficient.

material properties (Table E1 and figures E1-E5). Also we have to consider a temperature dependent heat transfer coefficient (figures E6 and E7).

The temperature dependent Young's modulus, Poisson's ratio, thermal conductivity, specific heat and thermal expansion coefficient for alumina, $\text{SiC}_w/\text{alumina}$ composites, titanium diboride and Macor glass ceramics are readily measureable. The modulus of alumina (figure 22), $\text{SiC}_w/\text{alumina}$ composites (figure 14), SiC fiber reinforced aluminosilicate (figure E5a) and Macor (figure E4a) were directly obtained by the author. Also the thermal expansion of SiC whisker reinforced alumina composites (figure 8b) and SiC fiber reinforced aluminosilicate (figure E5b) were directly obtained by the author. Poisson's ratio of alumina [E3], titanium diboride [E7] and Macor [E8], the specific heat of alumina [E4], $\text{SiC}_w/\text{alumina}$ [E4, E5], titanium diboride [E7], and Macor [E8], thermal conductivity of alumina [E4], $\text{SiC}_w/\text{alumina}$ [E6], titanium diboride [E7], and Macor [E8], and thermal expansion coefficient of alumina [E4], titanium diboride [E7] and Macor [E8] were obtained from literatures (Table E1). In this study, we measured the heat transfer coefficient of alumina. Although the SiC whisker reinforced alumina composites contained 20 volume percent of SiC whisker^{*}, we approximate the heat

* Specific heat of 20 vol % SiC/Alumina composites is not available. This functional form was evaluated from the data of alumina and SiC by Rule of Mixture.

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Table E1. Temperature Dependent Material Properties of Polycrystalline Alumina and SiC Whisker Reinforced Alumina Composites. Unit of T is Celcius degree.
(Result of least squares best fit of available data)

Property	Functional Form	Temp. range	cor. coeff & reference
Alumina			
E (GPa)	$335.49 - 0.027T$	20 C < T < 600 C	0.99 this study
$\alpha_{th} (10^{-6}/C)$	$6.05 + 0.019T - 0.000019T^2$	20 C < T < 600 C	0.98 [E4]
ν	$0.233 - 0.149 \times 10^{-4} T$	20 C < T < 500 C	0.99 [E3]
k (W/m C)	$20.5 - 3.19 \times 10^{-2} T + 1.9 \times 10^{-5} T^2$	20 C < T < 600 C	0.98 [E4]
C_p (J/kg C)	$791.4 + 1.41T - 1.63 \times 10^{-3} T^2 + 7.03 \times 10^{-7} T^3$	20 C < T < 800 C	0.99 [E4]
SiC whisker reinforced alumina			
E (GPa)	$409.5 - 0.047T$	20 C < T < 900 C	0.99 This study
$\alpha_{th} (10^{-6}/C)$	$5.13 + 5.7 \times 10^{-3} T + 4.5 \times 10^{-6} T^2$	20 C < T < 500 C	0.99 This study
ν^*	0.22		This study
k (W/m C)	$35 - 6.82 \times 10^{-2} T + 5.1 \times 10^{-5} T^2$	20 C < T < 900 C	0.99 [E6]
C_p (J/kg C)**	$762.0 + 1.56T - 1.95 \times 10^{-2} T^2 + 9.19 \times 10^{-7} T^3$	20 C < T < 800 C	0.99 [E4, E5]

* Since temperature dependent data are not available and Poisson's ratio is weak function of temperature [E3], we used room temperature data.

** Specific heat of 20 vol % SiC/Alumina composites is not available. This functional form was evaluated from the data of Alumina and SiC by Rule of Mixture.

Table E1. (Continued)

Property	Functional Form	Temp. range	cor. coeff
Titanium diboride			
E (Gpa)	496.7 - 0.0384T	20 C < T < 600 C	0.99 [E7]
$\alpha_{th}(10^{-6}/C)$	$4.10 + 0.01T - 5.4 \times 10^{-6} T^2$	20 C < T < 600 C	0.98 [E7]
ν	$0.099 + 1.138 \times 10^{-5} T$	20 C < T < 600 C	0.99 [E7]
k (W/m C)	$32.2 - 8.3 \times 10^{-4} T + 6.5 \times 10^{-6} T^2$ $- 8.78 \times 10^{-10} T^3$	20 C < T < 600 C	0.99 [E7]
C_p (J/kg C)	$608.4 + 1.62T - 2.0 \times 10^{-3} T^2$ $+ 9.200 \times 10^{-7} T^3$	20 C < T < 600 C	0.99 [E7]
Macor			
E (Gpa)	$65.7 - 0.013T + 5.15 \times 10^{-5} T^2$ $- 7.86 \times 10^{-8} T^3$	20 C < T < 500 C	0.99 [E8]
$\alpha_{th}(10^{-6}/C)$	$7.04 + 1.13 \times 10^{-2} T$	0 C < T < 900 C	0.99 [E8]
ν	$0.27 - 1.88 \times 10^{-5} T + 5.31 \times 10^{-7} T^2$ $- 9.443 \times 10^{-9} T^3$	20 C < T < 500 C	0.99 [E8]
k (W/m C)	$1.29 + 2.42 \times 10^{-5} T + 5.85 \times 10^{-8} T^2$ $+ 8.965 \times 10^{-10} T^3$	20 C < T < 600 C	0.99 [E8]
C_p (J/kg C)	$709.3 + 1.92T - 5.26 \times 10^{-3} T^2$ $+ 5.631 \times 10^{-6} T^3$	20 C < T < 600 C	0.99 [E8]
SiC _f /AS*			
E (GPa)	126.1 - 0.011T (on heating) 124.2 - 0.009T (on cooling)	20 C < T < 600 C 20 C < T < 600 C	0.96 0.99
$\alpha_{th}(10^{-6})$	$2.58 + 3.16 \times 10^{-2} T + 1.46 \times 10^{-6} T^2$	20 C < T < 500 C	this study 0.99 this study

* ν , k and C_p of SiC_f/AS were not available.

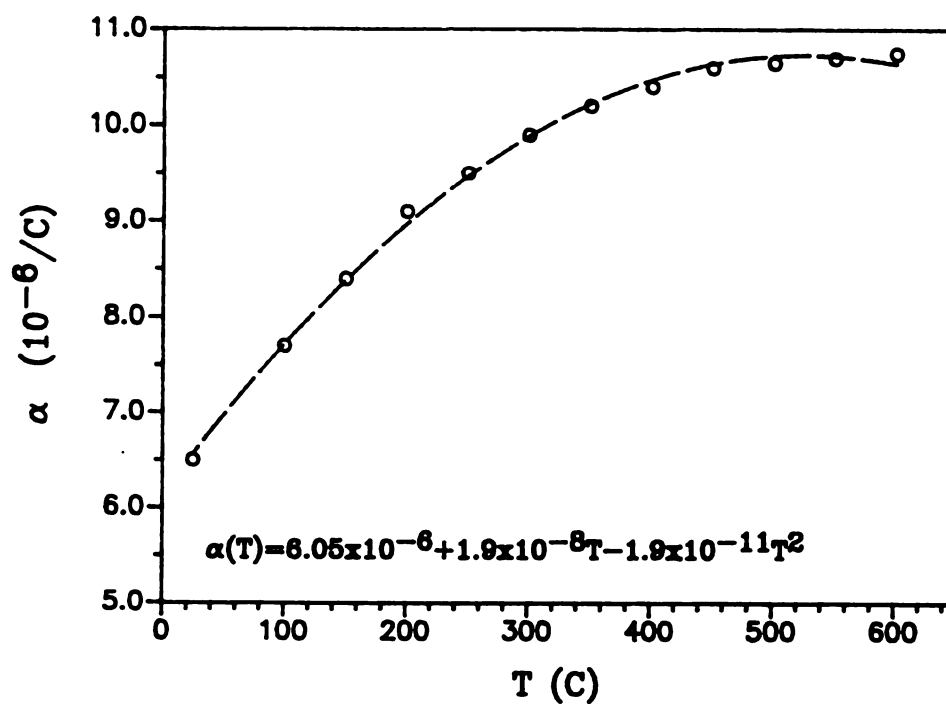


Figure Ela. Thermal expansion coefficient of alumina as a function of temperature.

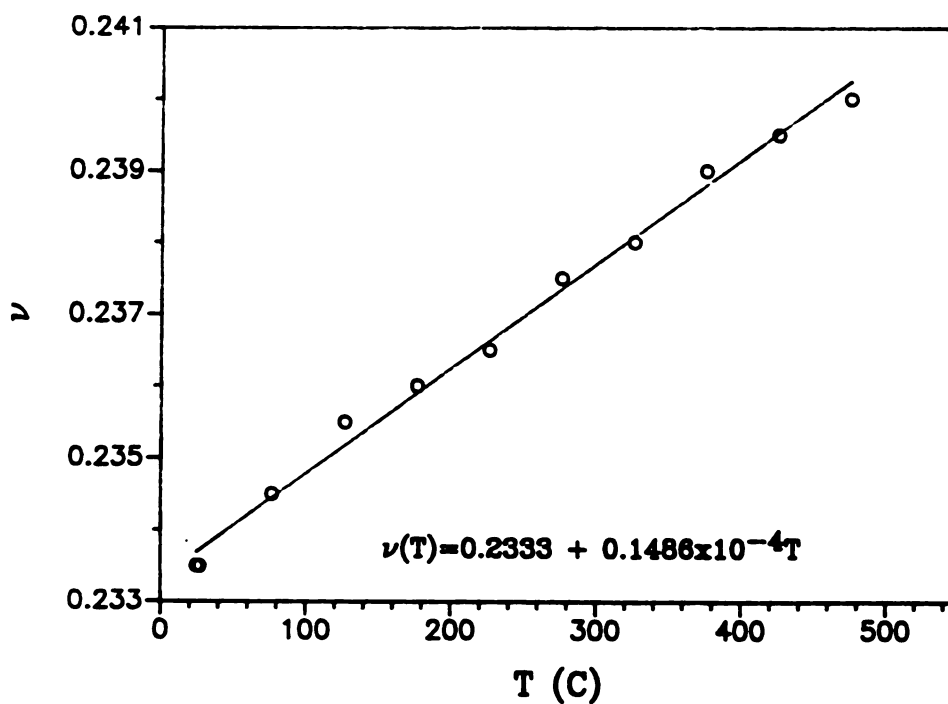


Figure Elb. Poisson's ratio of alumina as a function of temperature.

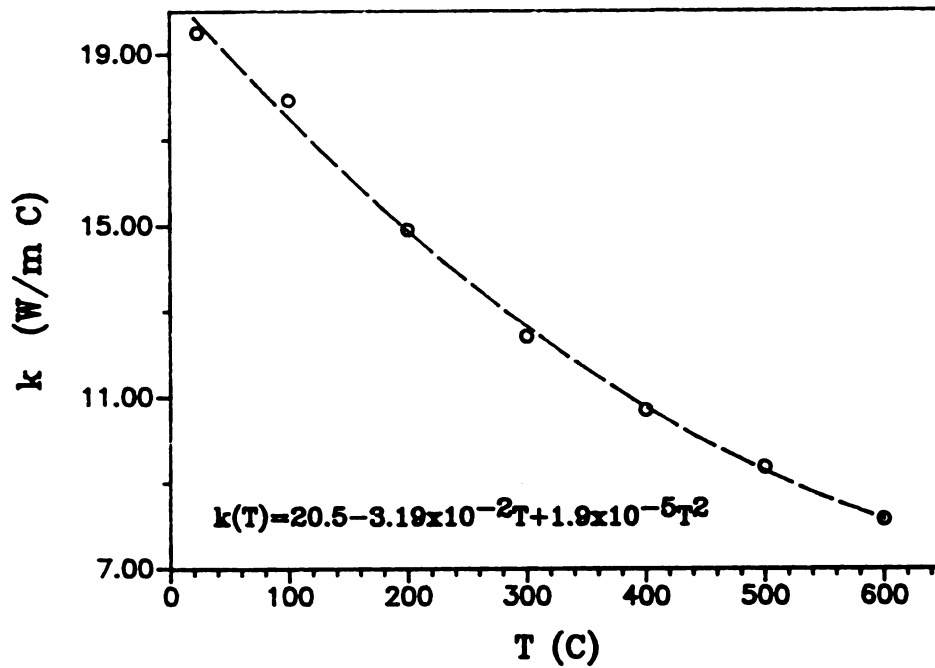


Figure Elc. Thermal conductivity of alumina as a function of temperature.

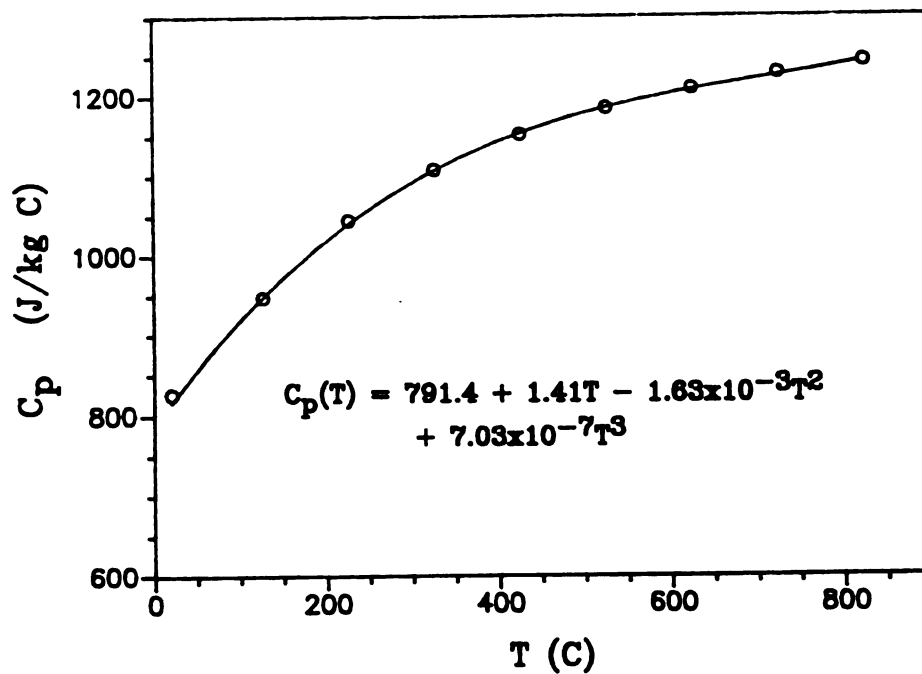


Figure Eld. Specific heat of alumina as a function of temperature.

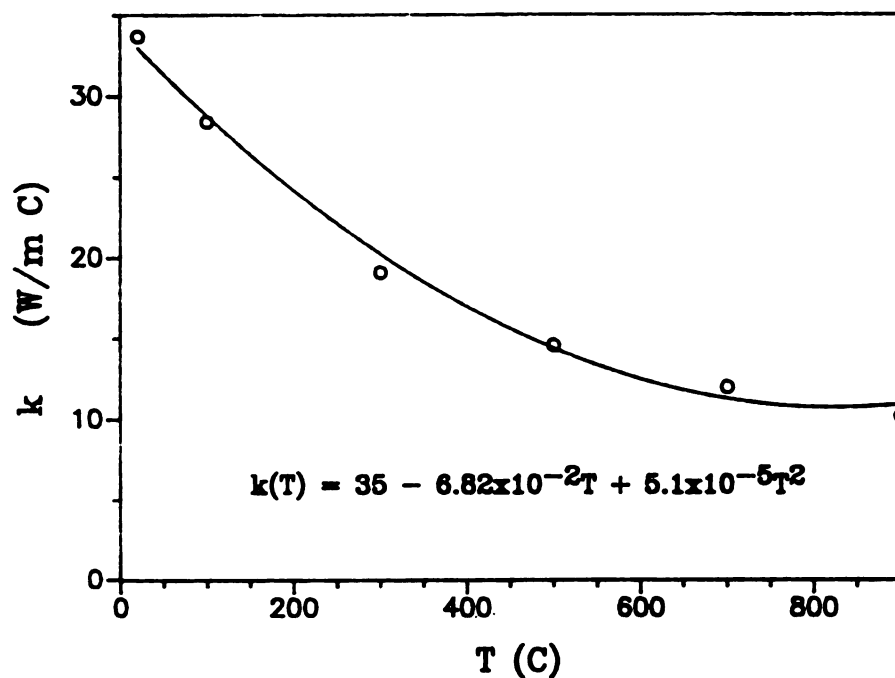


Figure E2a. Thermal conductivity of $\text{SiC}_w/\text{alumina}$ as a function of temperature.

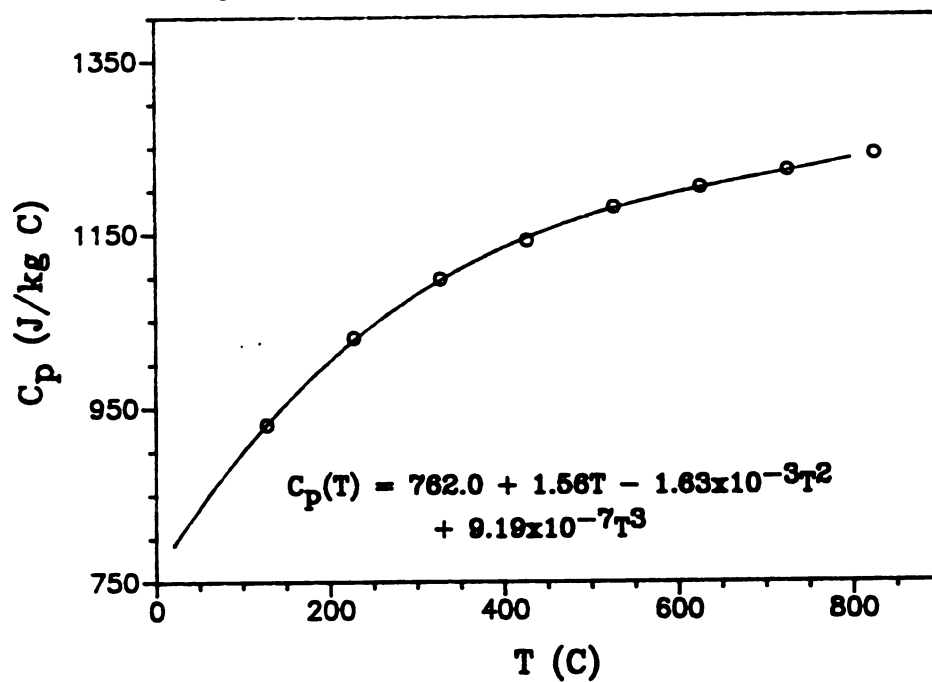


Figure E2b. Specific heat of $\text{SiC}_w/\text{alumina}$ as a function of temperature.

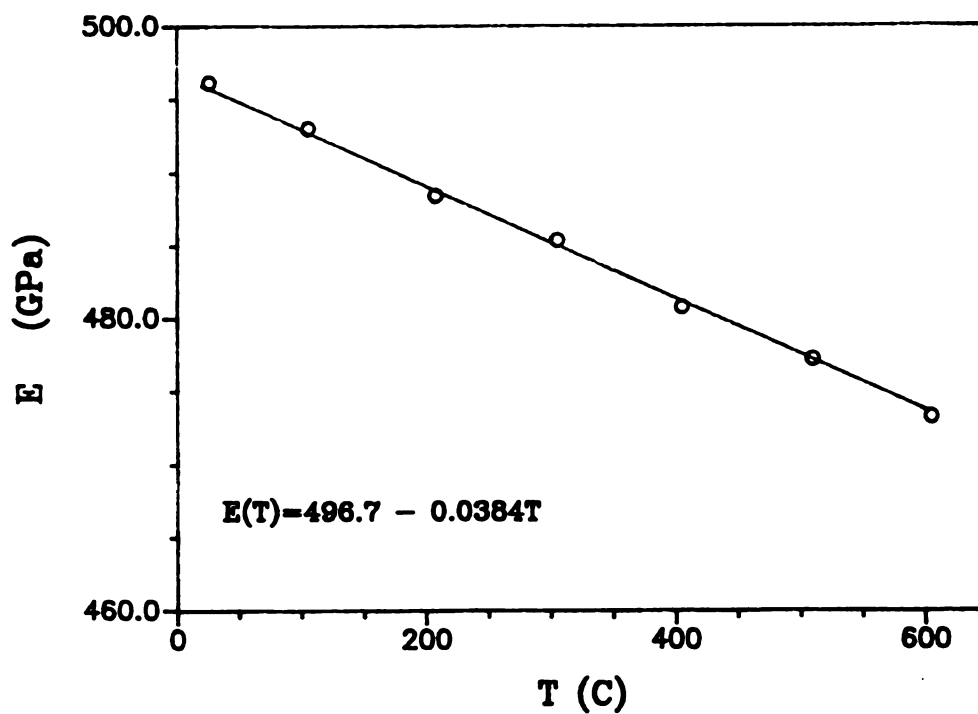


Figure E3a. Young's modulus of TiB_2 as a function of temperature.

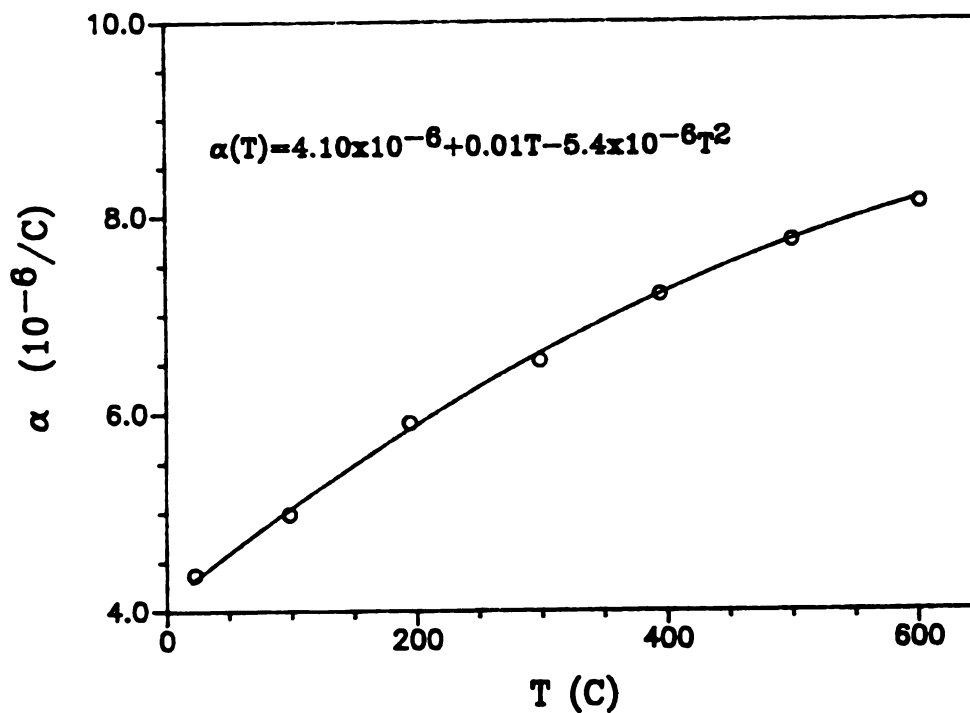


Figure E3b. Thermal expansion coefficient of TiB_2 as a function of temperature.

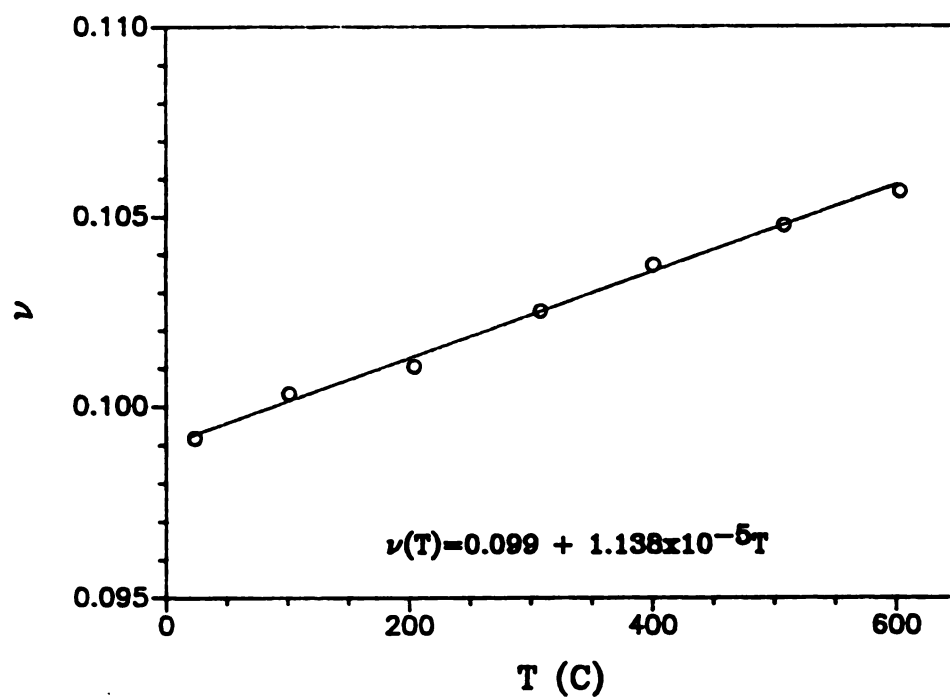


Figure E3c. Poisson's ratio of TiB_2 as a function of temperature.

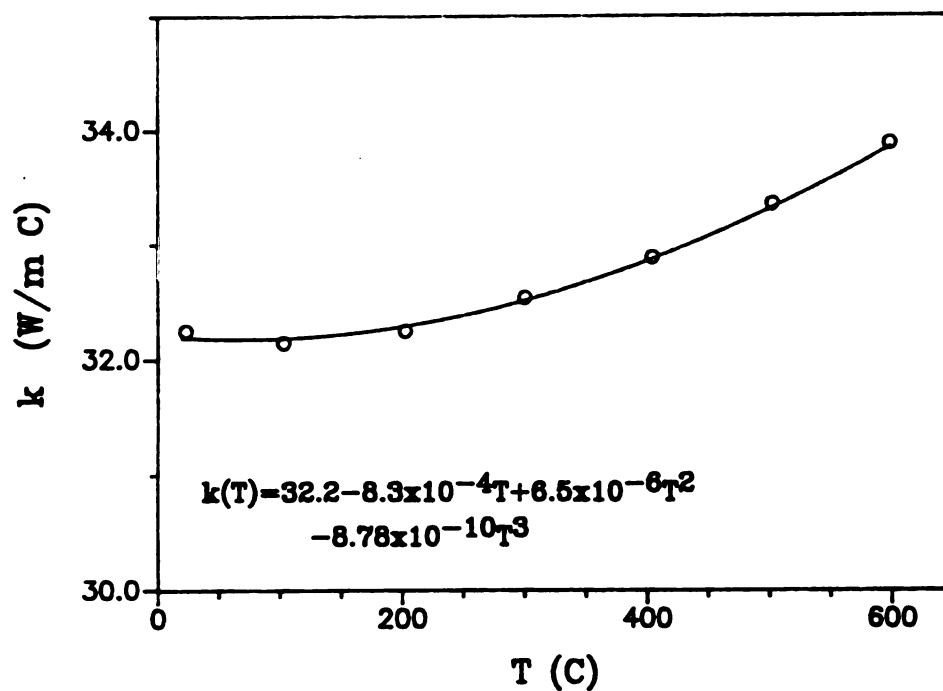


Figure E3d. Thermal conductivity of TiB_2 as a function of temperature.

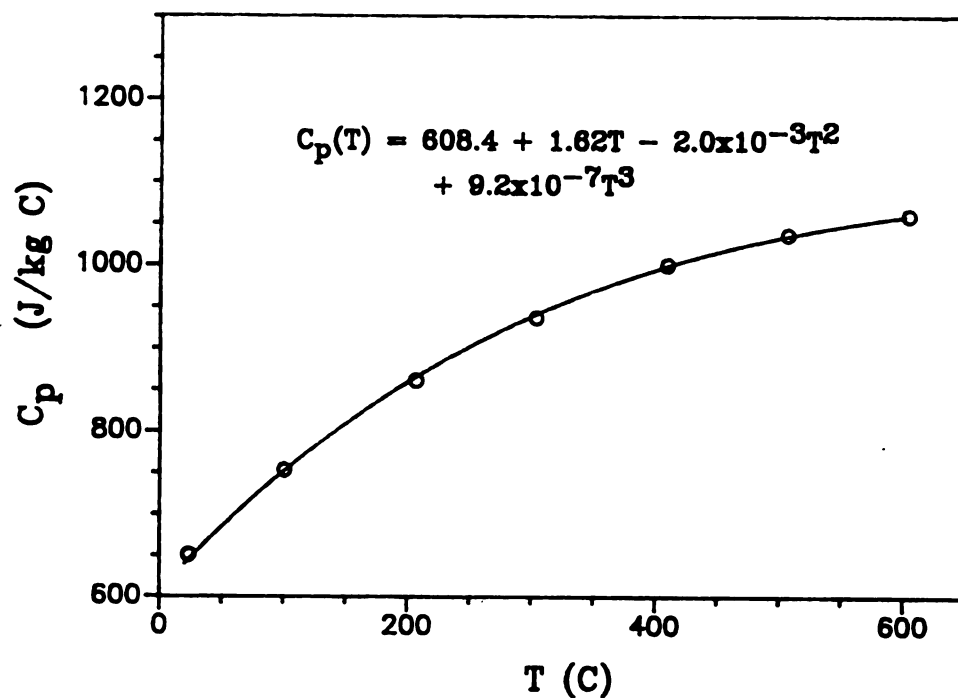


Figure E3e. Specific heat of TiB_2 as a function of temperature.

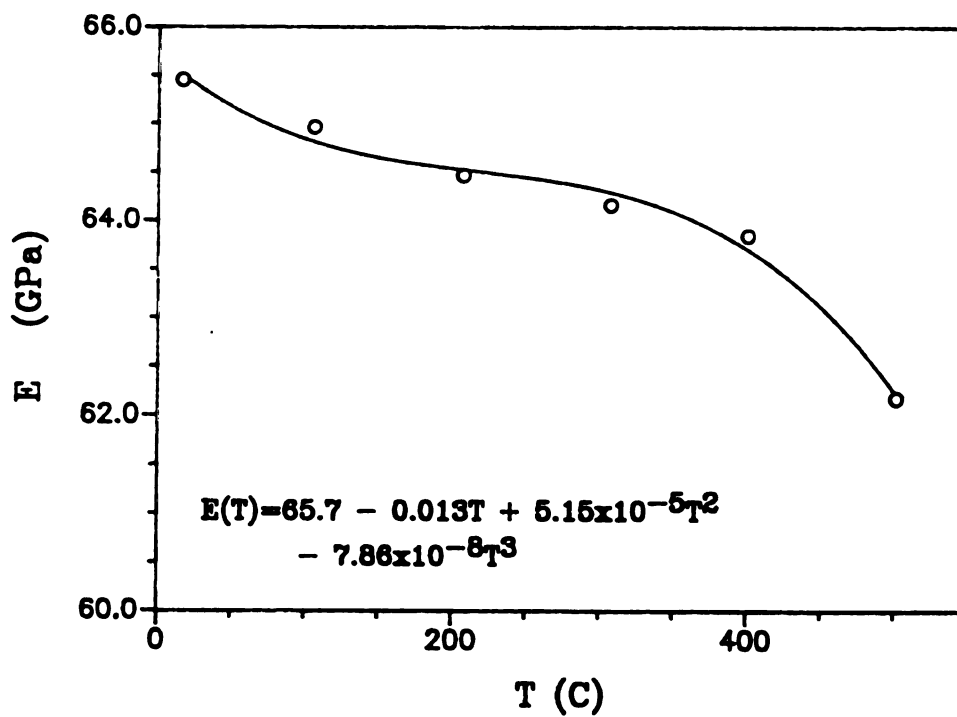


Figure E4a. Young's modulus of Macor as a function of temperature.

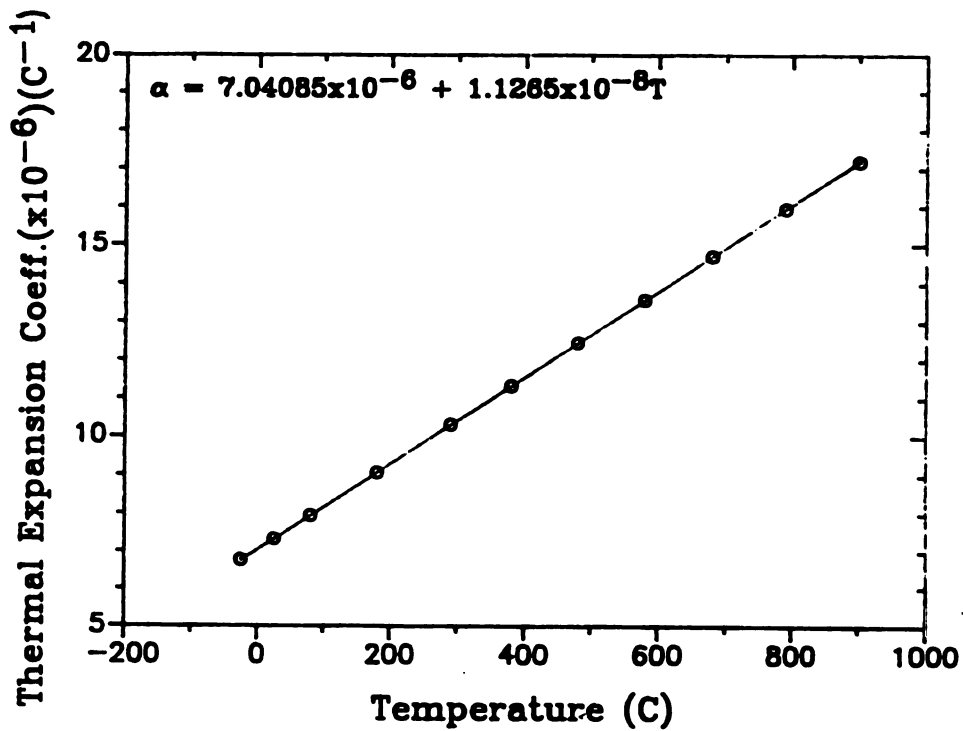


Figure E4b. Thermal expansion coefficient of Macor as a function of temperature.

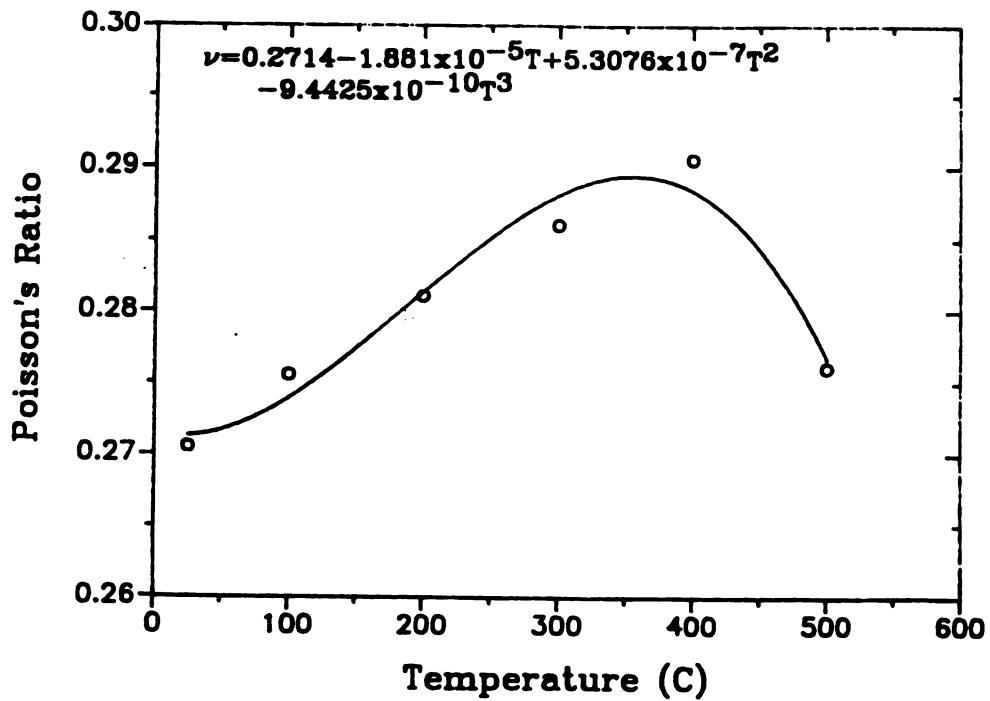


Figure E4c. Poisson's ratio of Macor as a function of temperature.

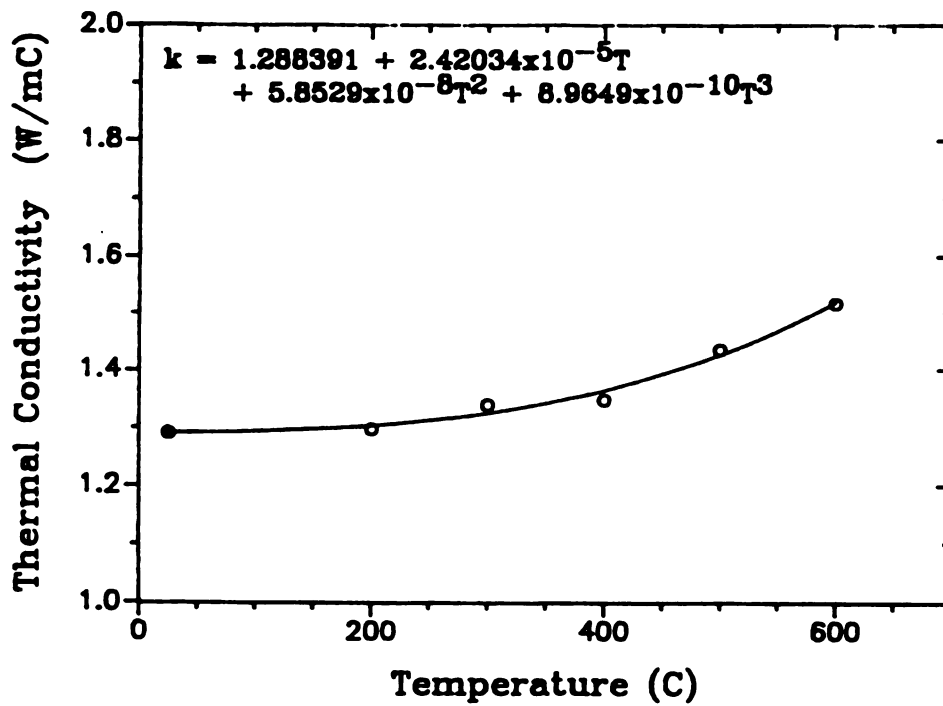


Figure E4d. Thermal conductivity of Macor as a function of temperature.

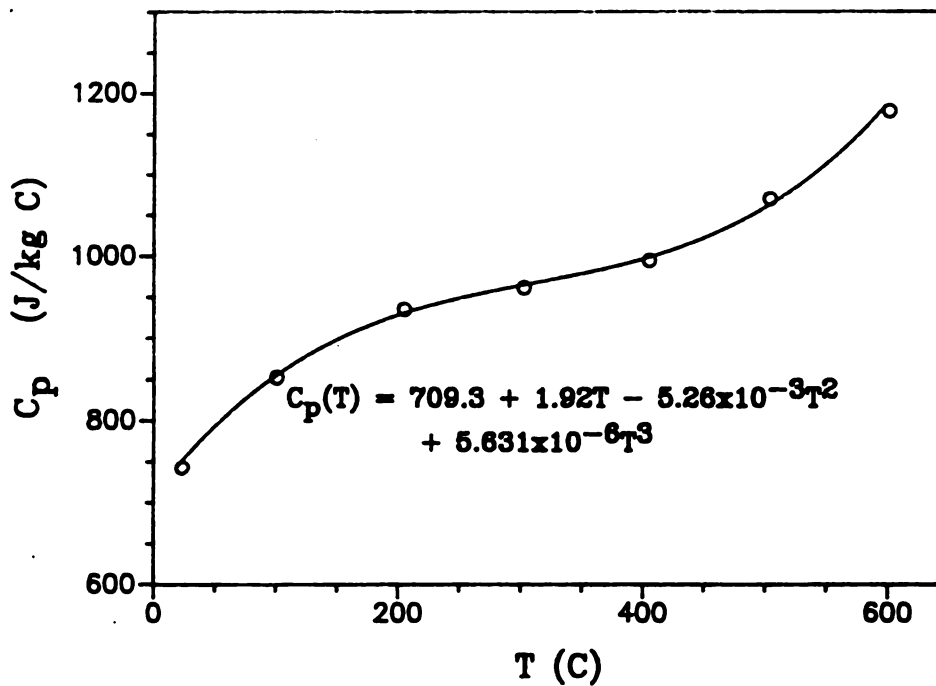


Figure E4e. Specific heat of Macor as a function of temperature.

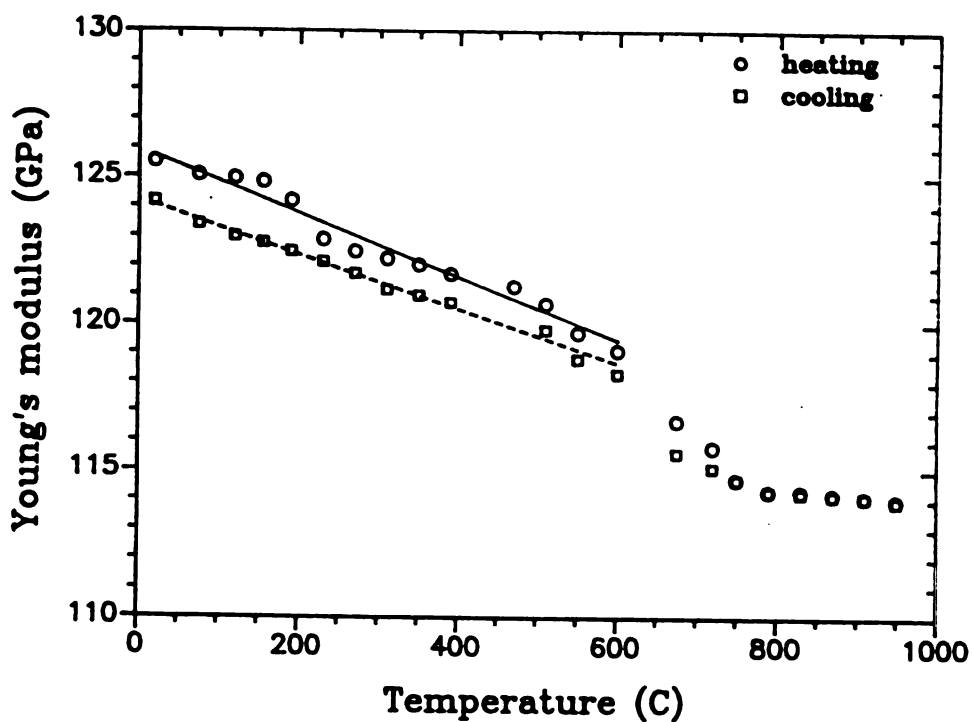


Figure E5a. Young's modulus of SiC_f/AS as a function of temperature.

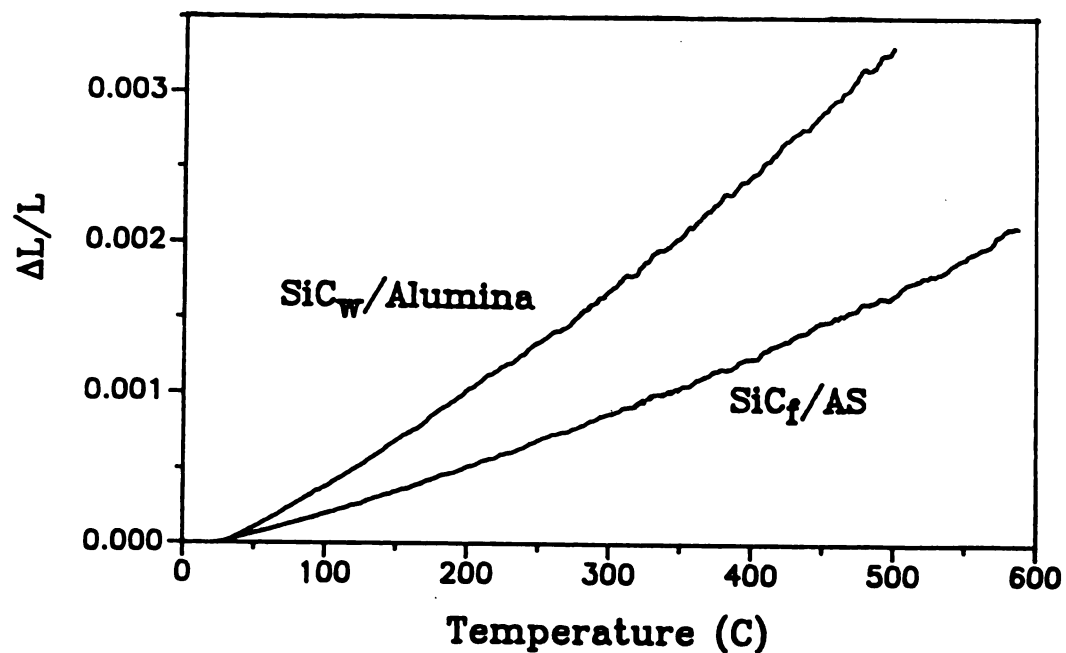


Figure E5b. Thermal expansion of SiC_f/AS as a function of temperature.

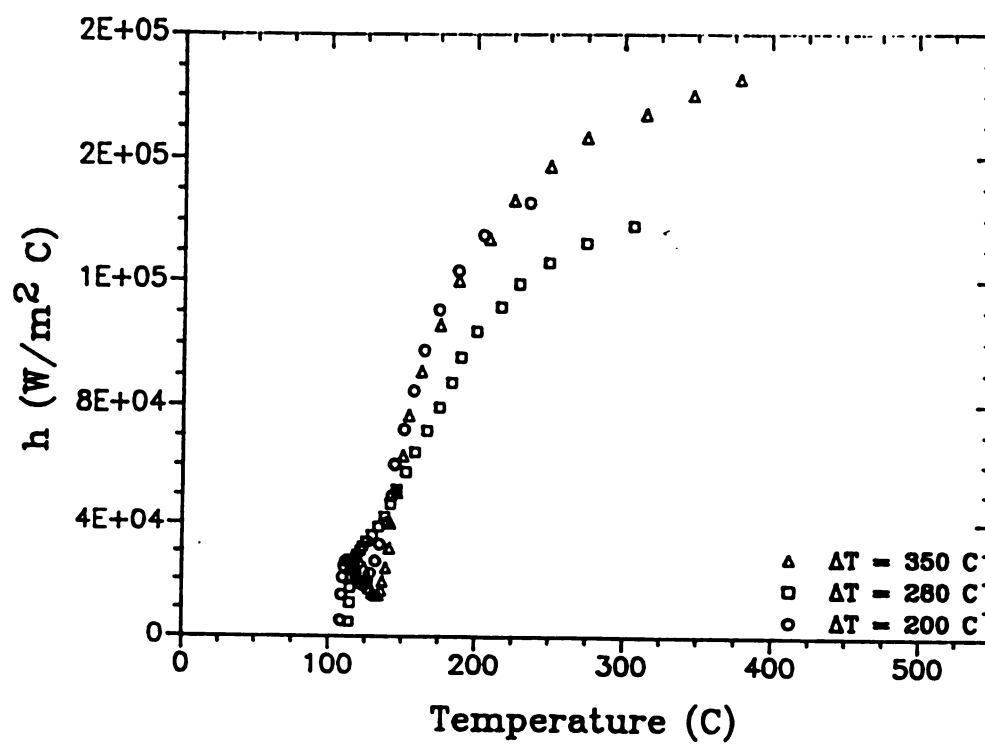


Figure E6a. The estimated surface heat transfer coefficient versus temperature calculated from the experimentally obtained thermocouple voltage versus cooling time for water quenched Macor ($\Delta T = 200, 280$ and 350°C).

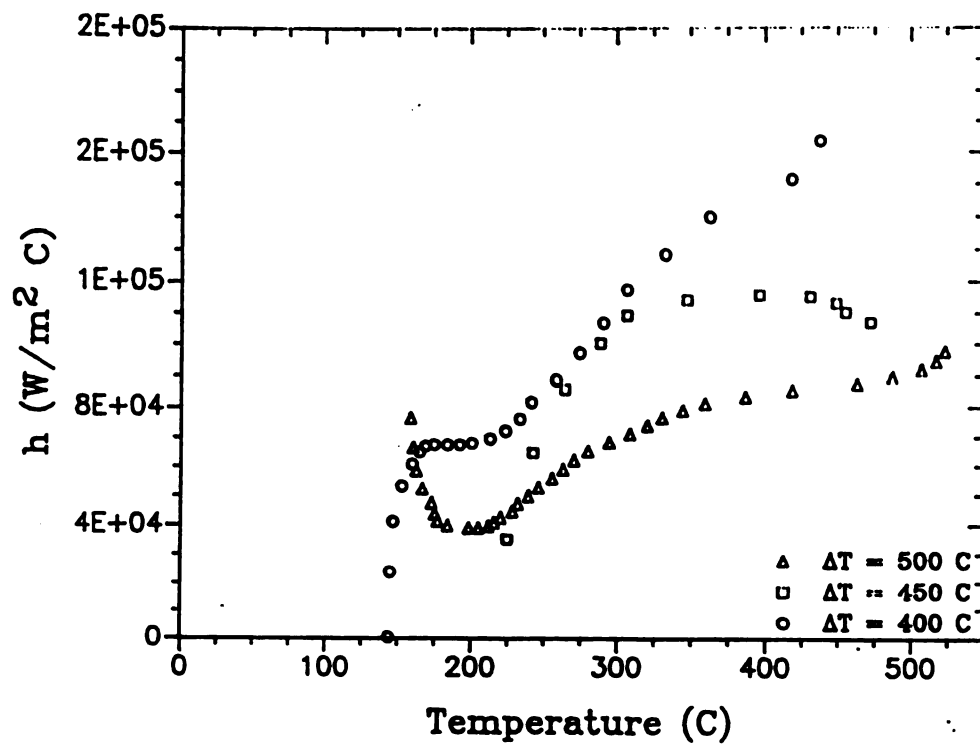


Figure E6b. The estimated surface heat transfer coefficient versus temperature calculated from the experimentally obtained thermocouple voltage versus cooling time for water quenched Macor ($\Delta T = 400, 450$ and 500 C).

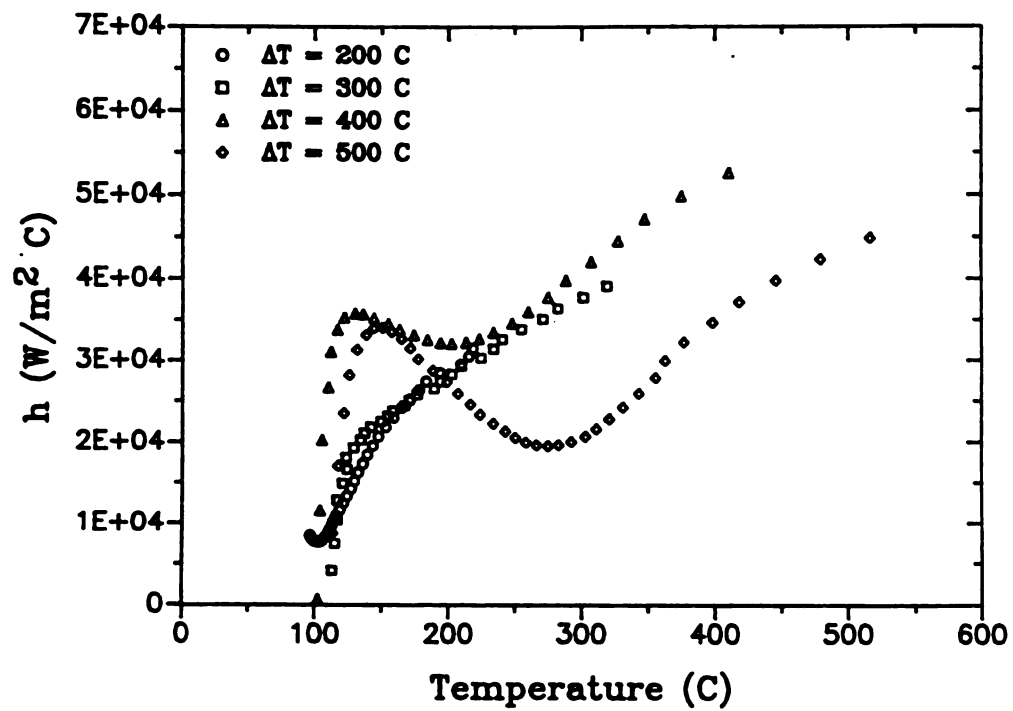


Figure E7. The estimated surface heat transfer coefficient versus temperature calculated from the experimentally obtained thermocouple voltage versus cooling time for water quenched TiB₂.

transfer coefficient of the SiC whisker reinforced alumina composites by the heat transfer coefficient of alumina.

By applying the temperature dependent material properties (Table E1) and heat transfer coefficient (figure 29-31, E6 and E7), the maximum thermal tensile stress at the surface of the plate, $\sigma_{\max}$ can be assessed as a function of the quench temperature difference for four different material (figure E8-E11). The peak thermal stress for polycrystalline alumina and SiC whisker reinforced alumina composites as a function of the specimen surface temperature is fairly linear in the region $\Delta T > 200$ C when the specimens were quenched into a 20 degrees C water bath (figure E8a and E9). For Macor, the peak thermal stress is almost linear for the entire temperature range.

Appendix E References

- E1. K. Kokini, "Thermal Shock of Cracked Strip: Effect of Temperature Dependent Material Properties", J. Eng. Frac. Mec., 25 [2]: 167-176, 1986.
- E2. J. P. Singh, Y. Tree and D. P. H. Hasselman, "Effect of Bath and Specimen Temperature on Thermal Stress Resistance of Brittle Ceramics Subjected to Thermal Quenching", J. Mat. Sci., 16: 2109-2118, 1981.
- E3. G. Simmons and H. Wang, Single Crystal Elastic Constants and Calculated Aggregate Properties: A Handbook, The M. I. T. Press, Cambridge, MA, 1971.
- E4. W. H. Gitzen, p 65 in Alumina as a Ceramic Material, American Ceramic Society, Columbus, OH, 1970.
- E5. Z. Li and R. C. Bradt, "Thermal Expansion of Hexagonal Polytype of Silicon Carbide", J. Amer. Ceram. Soc., 69 [12]: 863-866, 1986.

- E6. P. H. McCluskey, R. K. Williams, R. S. Graves and T. N. Tiegs, "Thermal Diffusivity/Conductivity of Alumina-Silicon Carbide Composites", J. Amer. Ceram. Soc., 73 [2]: 461-464, 1990.
- E7. Y. S. Touloukian, editor, pp 236-249 in Thermophysical Properties of High Temperature Solid Materials, Vol. 6: Part I, Thermophysical Properties Research Center, Purdue University, 1967.
- E8. Astro Met Associates Inc. (A distributor of the Corning Glass products, Cincinnati, OH) provided technical data for Macor.

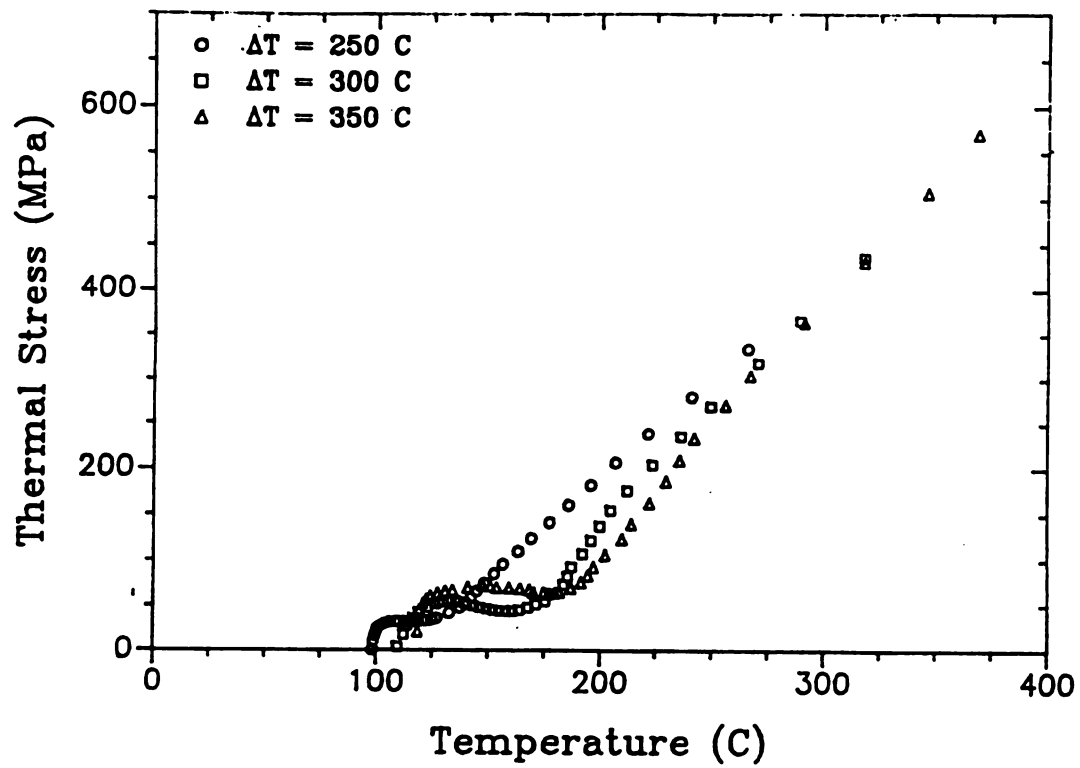


Figure E8a. Maximum surface tensile stress calculated from measured surface heat transfer coefficient and temperature dependent material properties for alumina (water quench).

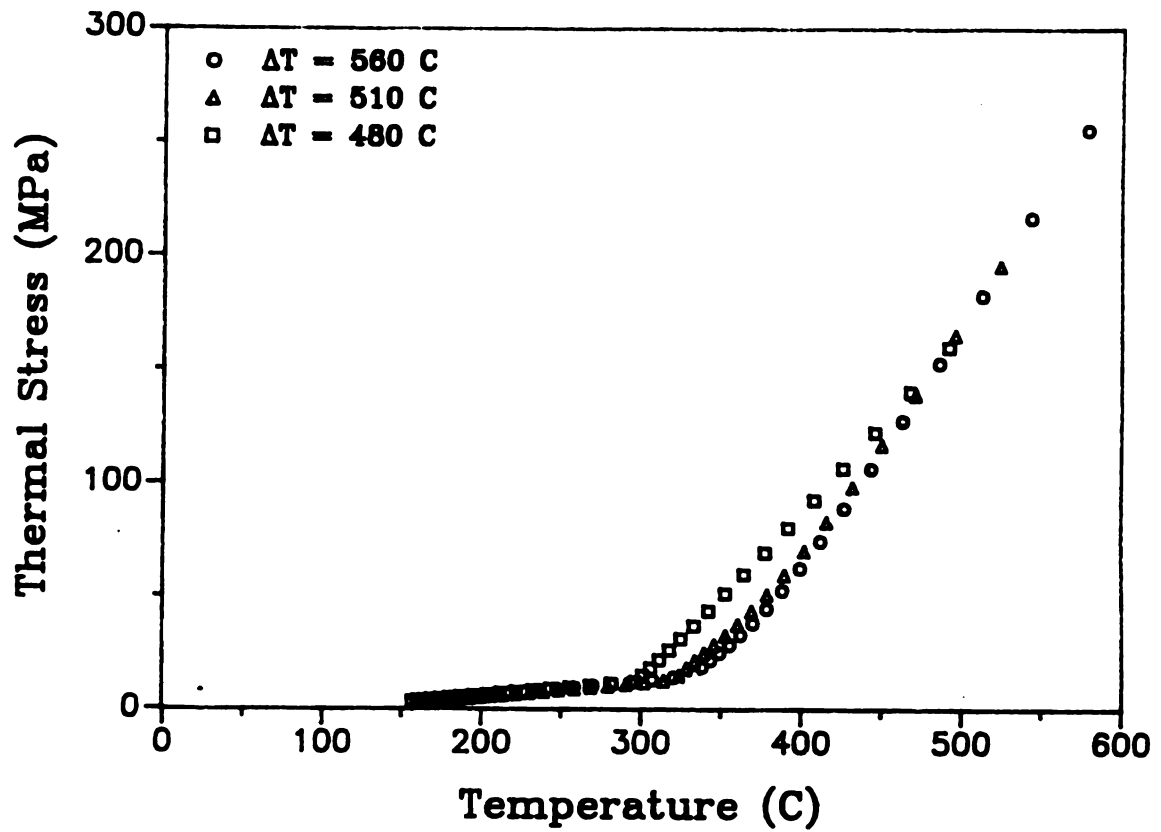


Figure E8b. Maximum surface tensile stress calculated from measured surface heat transfer coefficient and temperature dependent material properties for alumina (oil quench).

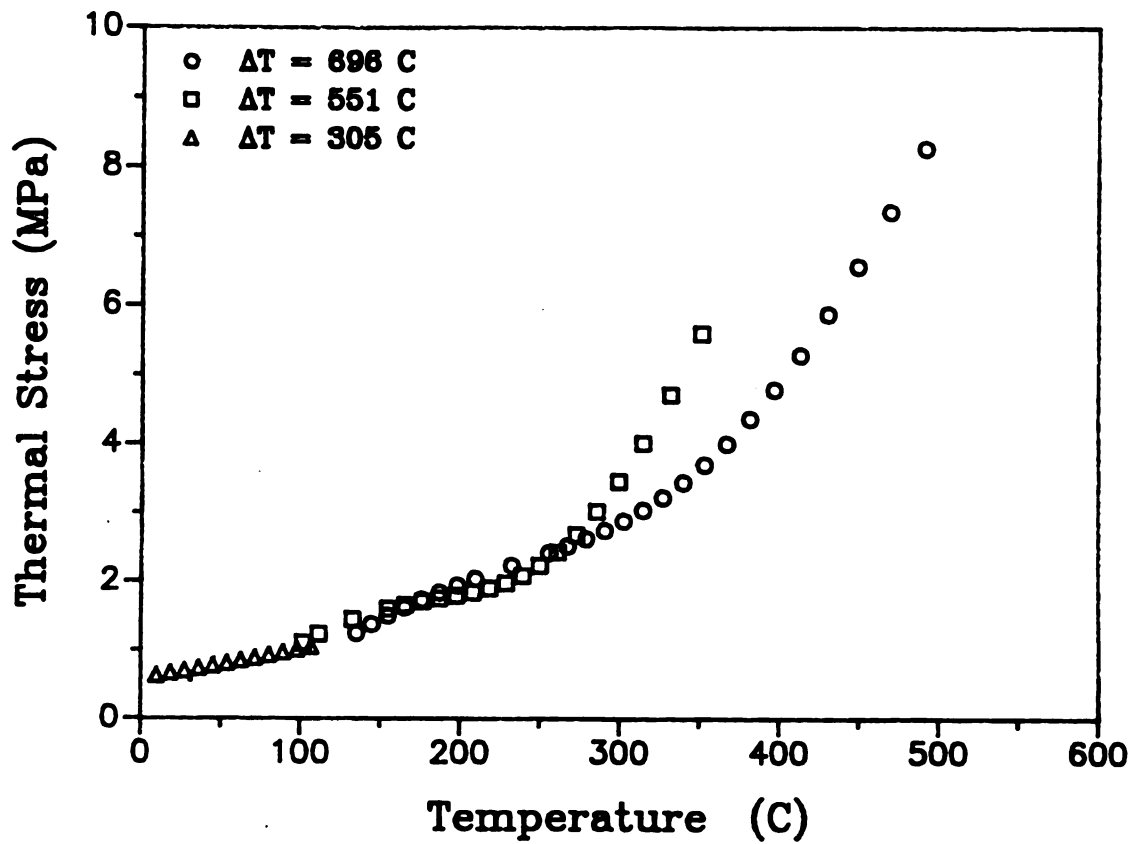


Figure E8c. Maximum surface tensile stress calculated from measured surface heat transfer coefficient and temperature dependent material properties for alumina (liquid nitrogen quench).

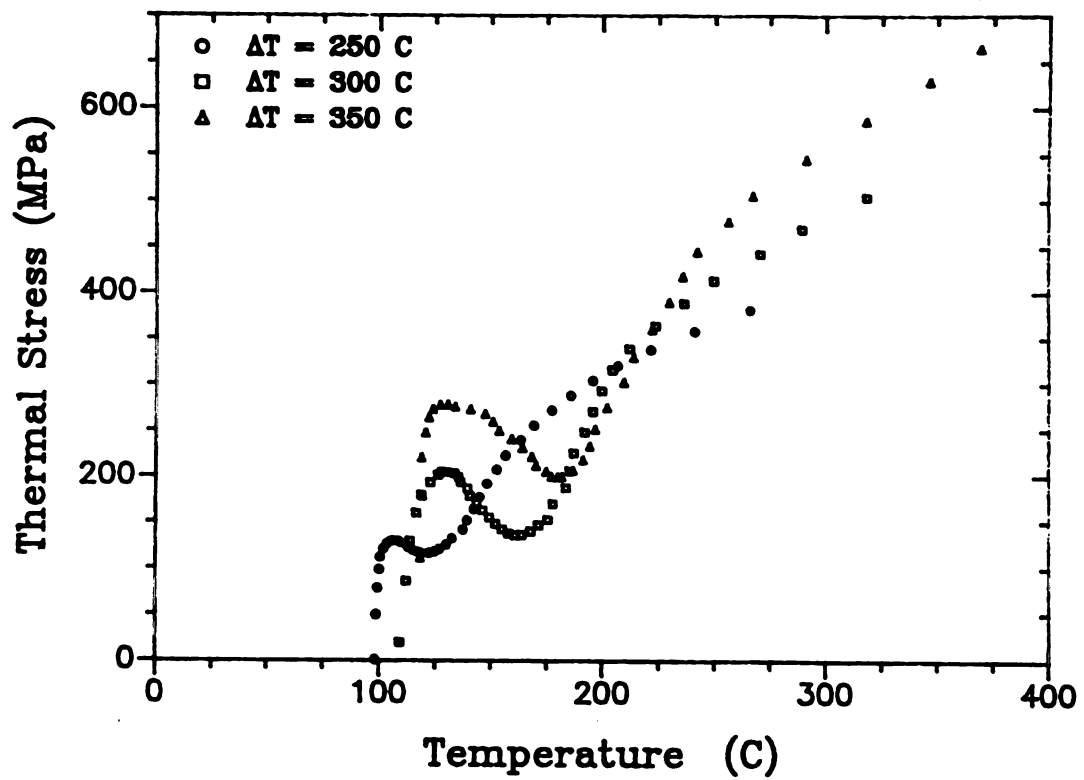


Figure E9. Maximum surface tensile stress calculated from measured surface heat transfer coefficient and temperature dependent material properties for SiC whisker reinforced alumina composites.

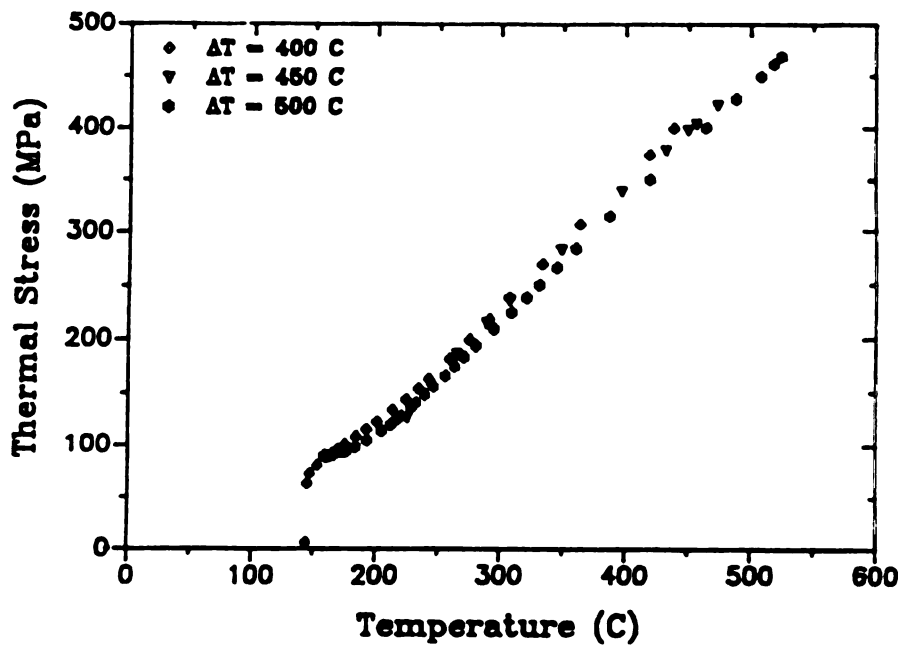
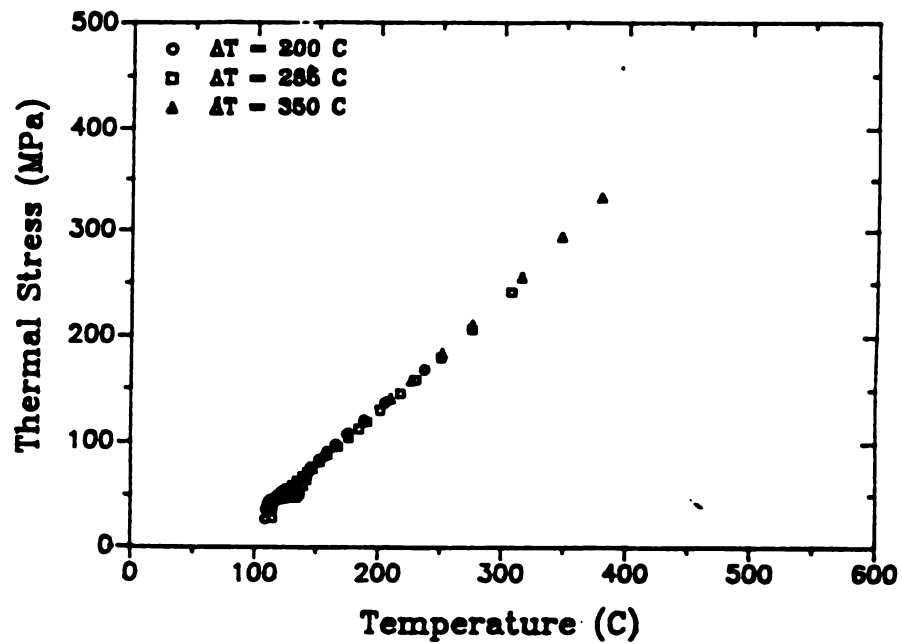


Figure E10. Maximum surface tensile stress calculated from measured surface heat transfer coefficient and temperature dependent material properties for Macor.

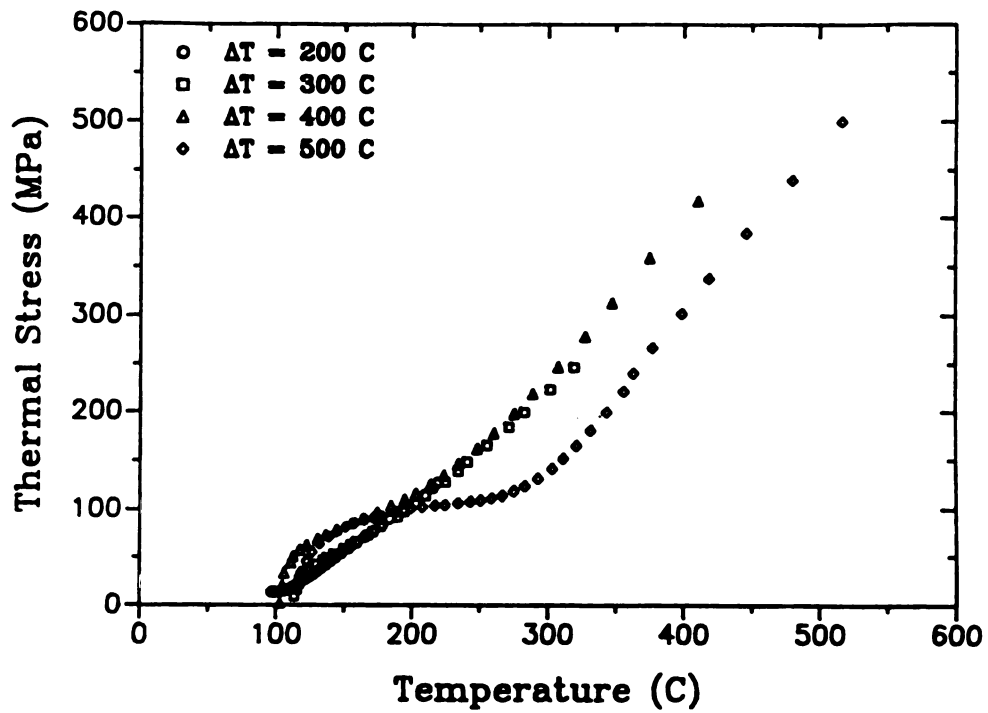


Figure E11. Maximum surface tensile stress calculated from measured surface heat transfer coefficient and temperature dependent material properties for titanium diboride.

Appendix F. Calculation of Heat Transfer Coefficient

The surface heat transfer coefficient calculation program evaluate the surface heat transfer coefficient using the measured specimen surface temperature and the temperature dependent specific heat.

```

10 REM ** Estimation of h (Inverse Parameter Estimation-Beck) **
20 CLS:KEY OFF
40 NDATA=31 : REM Data point taken from experimentat result
100 OPEN "BI250.DAT" FOR OUTPUT AS #2
140 READ WID,LENGTH,THICKNESS,DENSITY,TAMB
150 REM
155 REM
160 FOR KK = 0 TO NDATA
170   TIME = KK
180   GOSUB 1000 : REM ** Calculation of Volume and Total Surface Area
200   GOSUB 2000 : REM ** Calculation of h from measured temp-time
      data **
500   GOSUB 5000 : REM PRINT RESULTS TO CRT
700 NEXT KK
900 END
950 REM
970 REM
1000 REM **Calculation of Volume and Total Surface Area **
1140 VOLUME = WID*LENGTH*THICKNESS
1160 SURFACE = 2*( WID*THICKNESS + THICKNESS*LENGTH + LENGTH*WID)
1900 RETURN
1950 REM
1970 REM
2000 REM **calculation of h **
2020 TEMP = 258.6- 18.876*TIME + 1.094*TIME^2 - .031*TIME^3
      +.000324*TIME^4
2040 TEMPTIME = (-18.88+ 2*1.094*TIME - 3*.031*TIME^2 +
      4*.000324*TIME^3) / .0025
2050 K = 20.5 - .0319*TEMP + .000019*TEMP^2
2060 CP = 791.4 + 1.407*TEMP - .001634*TEMP^2 + 7.029E-07*TEMP^3
2080 HCOEFF = (DENSITY*CP*VOLUME*TEMPTIME)/(SURFACE*(TAMB-TEMP))
2090 BI = HCOEFF*.5*THICKNESS/K
2900 RETURN
2950 REM
2980 REM
5000 REM Print results to crt
5100 A$ = "  #.#####  #####.##  #####.####  ###.####  "

```

```
5105 IF KK = 0 THEN PRINT " " : PRINT " "  
5110 IF KK = 0 THEN PRINT " Timesteps(t/delta t) TEMP h (W/m2-C) Biot"  
5130 PRINT USING A$;TIME, TEMP, HCOEFF, BI  
5200 WRITE #2, TEMP, HCOEFF  
5900 RETURN  
20000 DATA 0.01200,0.06970,0.001,3700,20
```


Appendix G. Calculation of Maximum Thermal Stress

This program calculates maximum tensile stress at surface developed during quenching. The temperature dependent functions of Young's modulus, thermal expansion coefficient, Poisson's ratio and Biot modulus were used.

```

100 REM
500 REM CALCULATION of maximum surface thermal stress of the plate
1000 REM Equations are based on the Manson's work.
1020 OPEN "250.dat" FOR OUTPUT AS #1
1100 INPUT "Material, Alumina(1) or SiC/Alumina(2)"; MATERIAL
1120 N = 37
1124 IF MATERIAL = 1, THEN TH = .05
1128 REM TH is half thickness of the specimen in cm.
1130 IF MATERIAL = 2, THEN TH = .15
1140 I2 = N
1160 DIM H(I2), T(I2), ST(I2), F(I2)
1180 DIM E(I2), A(I2), K(I2), BI(I2), V(I2)
1200 FOR J = 1 TO I2
1220   READ T(J), H(J)
1240 NEXT J
1250 IF MATERIAL = 2 THEN GOTO 2300
1280 REM Material properties of alumina
1300 DEF FNE(I) = 335.49 - .027*T(I) : REM Young's modulus (GPa)
1320 DEF FNA(I) = 6.05 + .019 *T(I) - .000019 *T(I)^2 :
      REM Thermal expansion coeff. (e-06)
1340 DEF FNV(I) = .233 - .0000149*T(I) : REM Poisson's ratio
1350 DEF FNK(I) = .205 - .000319*T(I) + 1.9E-07*T(I)^2 :
      REM Thermal conductivity (W/cm C)
1360 DEF FNBI(I) = H(I)*TH / (.205 - .000319*T(I) + 1.9E-07*T(I)^2) :
      REM Biot modulus
1380 DEF FNF(I) = 1 / ( 1.5 + (3.25/FNBI(I)) - .5*EXP(-16/FNBI(I))) :
      REM Bi factor from Manson
1400 DEF FNST(I) = FNE(I)*FNA(I)*FNF(I)*(T(1)-23)*.001 / (1-FNV(I))
      REM max. thermal stress (MPa)
1500 FOR L = 1 TO I2
1520   PRINT T(L), FNBI(L), FNST(L)
1530   WRITE #1, T(L)
1540 NEXT L
1550 END
2000 REM
2100 REM Material properties of SiC/alumina composite
2300 DEF FNE(I) = 409.5 - .047*T(I) : REM Young's modulus (GPa)

```

```

2320 DEF FNA(I) = 5.13 + .0057*T(I) + .0000045*T(I)^2 :
                        REM Thermal expansion coefficient (e-06)
2340 DEF FNV(I) = .22 :REM Poisson's ratio
2350 DEF FNK(I) = .35-.000682*T(I)+5.1E-07*T(I)^2 :
                        REM Thermal conductivity (W/cm C)
2360 DEF FNBI(I) = H(I)*TH/FNK(I) :REM Biot modulus
2380 DEF FNF(I) = 1/( 1.5+(3.25/FNBI(I))-.5*EXP(-16/FNBI(I))) :
                        REM Bi factor from Manson
2400 DEF FNST(I) = FNE(I)*FNA(I)*FNF(I)*(T(1)-23)*.001/(1-FNV(I))
                        :REM max. thermal stress (MPa)

2500 REM
3000 FOR L = 1 TO I2
3020 PRINT T(L), FNBI(L), FNST(L)
3030 WRITE #1, T(L), FNST(L)
3040 NEXT L
3050 END

5000 DATA 266, 5.71481
5001 DATA 241, 5.428731
5002 DATA 221.5, 5.124645
5003 DATA 206.7, 4.805566
5004 DATA 195.7, 4.475244
5005 DATA 185.75, 4.138125
5006 DATA 177.25, 3.799249
5007 DATA 169.2, 3.464065
5008 DATA 163.25, 3.138183
5009 DATA 156.55, 2.827082
5010 DATA 152.6, 2.535827
5011 DATA 148.25, 2.268804
5012 DATA 144.95, 2.029522
5013 DATA 142.3, 1.820487
5014 DATA 139.3, 1.64316
5015 DATA 137.4, 1.497979
5016 DATA 132.5, 1.384444
5017 DATA 129.8, 1.301233
5018 DATA 126.85, 1.246325
5019 DATA 124.6, 1.217134
5020 DATA 122, 1.210601
5021 DATA 118.75, 1.223277
5022 DATA 117.1, 1.251366
5023 DATA 114.82, 1.290734
5024 DATA 112.9, 1.336884
5025 DATA 112.25, 1.384904
5026 DATA 109.7, 1.429395
5027 DATA 108.35, 1.464378
5028 DATA 106.07, 1.483195
5029 DATA 104.95, 1.478467
5030 DATA 103.4, 1.442029
5031 DATA 101.87, 1.365054
5032 DATA 100.4, 1.238226
5033 DATA 100.1, 1.052188

```

5034 DATA 99.3, 0.7982244
5035 DATA 98.7, 0.4692003
5036 DATA 98.2, 0.0060804

Appendix H. Calculation of the Product between the Transient Thermal Stress and Nondimensional Time

This program calculates the product (area) between the time dependent surface thermal stress (equation 80 in Section 4.4.6.) and duration time which exceeds the tensile strength of the material.

```

100 REM Calculation of area of transient thermal stress versus time
120 REM   Assume Biot's modulus is constant
140 REM
150 INPUT "MATERIAL (AL203 (1) SiC/AL203 (2) TiB2 (3))" ; MATERIAL
200 INPUT "OUTPUT FILE NAME" ; B$
300 INPUT "DELTA TEMPERATURE (Tspecimen - Tbath)"; DT
400 INPUT "BIOT MODULUS" ; BI
440 REM
480 REM
500 REM OPEN B$ + ".dat" FOR OUTPUT AS #2
700 A$ = "###.####^^^#.#####  ##.#####^  ##.#####  ##.####^^^ "
900 CLS
1000 PI = 3.14159
1020 R = 1/BI      : REM R factor in Cheung
1050 IF MATERIAL = 1 THEN GOSUB 5000 :
      REM Function definitions for material parameters of alumina
1070 IF MATERIAL = 2 THEN GOSUB 6000 :
      REM Function definitions for material parameters of SiC/alumina
1080 IF MATERIAL = 3 THEN GOSUB 6500 :
      REM Function definitions for material parameters of TiB2
1120   FOR I = 0 TO 60
1130     TIME = I/1000 :REM total time=500/10000 sec,
                        subdiv in time = 0.1 msec
1135     GOSUB 4000 : REM Temperature changes as a function of
                        time for each delta T
1140     F = (FNK(T)*TIME/(P*TH^2*FNC(T))) : REM nondimensional time
1145     DF = F - FPRE :REM subdivision in f
1150     X = (F^.5)*BI
1170     GOSUB 7000 : REM Error function calculation
1190     GOSUB 7500 : REM Stress calculation, based on Cheng
1195     FPRE = F
1420     IF STRESS < TFS THEN GOTO 1500
1440     GOSUB 8000 : REM NUMERICAL INTEGRATION
1500   NEXT I
1700 PRINT TFS, SUMAREA
2000 END
2500 REM
3000 REM

```

```

4000 REM Temp. changes as a function of time for each delta T
4100 REM *****
4110 REM *** WATER QUENCH of ALUMINA ***
4120 IF DT = 230 THEN T = 238.6-17.63*TIME/.0025+.997*(TIME/.0025)^2
      -.028*(TIME/.0025)^3+.000297*(TIME/.0025)^4 :REM DT = 230
4130 IF DT = 250 THEN T = 258.6-18.88*TIME/.0025+1.09*(TIME/.0025)^2
      -.031*(TIME/.0025)^3+.000326*(TIME/.0025)^4 :REM DT = 250
4140 IF DT = 270 THEN T = 278.6-20.12*TIME/.0025+1.19*(TIME/.0025)^2
      -.034*(TIME/.0025)^3+.000351*(TIME/.0025)^4 :REM DT = 270
4150 IF DT = 290 THEN T = 298.5-21.35*TIME/.0025+1.28*(TIME/.0025)^2
      -.037*(TIME/.0025)^3+.000378*(TIME/.0025)^4 :REM DT = 290
4160 IF DT = 310 THEN T = 320.62-23.19*TIME/.0025+1.42*(TIME/.0025)^2
      -.041*(TIME/.0025)^3+.000423*(TIME/.0025)^4 :REM DT = 310
4220 IF DT = 210 THEN T = 228.6-17.19*TIME/.0025+.953*(TIME/.0025)^2
      -.027*(TIME/.0025)^3+.00028*(TIME/.0025)^4 :REM DT = 210
4300 REM *****
4310 REM *** OIL QUENCH of ALUMINA *****
4320 IF DT = 575 THEN T = 602.9-37.835*TIME/.02+.4148*(TIME/.02)^2
      +.3137*(TIME/.02)^3-.018969*(TIME/.02)^4 :REM DT = 575
4330 IF DT = 553 THEN T = 583.05-35.46*TIME/.02+.3574*(TIME/.02)^2
      +.157*(TIME/.02)^3-.00447*(TIME/.02)^4 :REM DT = 565
4340 IF DT = 542 THEN T = 573.2-35.47*TIME/.02+.6122*(TIME/.02)^2
      +.126*(TIME/.02)^3-.00444*(TIME/.02)^4 :REM DT = 545
4500 REM *****
4510 REM *** WATER QUENCH of TiB2 ***
4520 IF DT = 500 THEN T = 505.66-31.383*TIME/.005+1.751*(TIME/.005)^2
      -.055859*(TIME/.005)^3+.006403*(TIME/.005)^4 :REM DT = 500
4530 IF DT = 400 THEN T = 404.91-30.1685*TIME/.005+1.772*(TIME/.005)^2
      -.0689*(TIME/.005)^3+.00108*(TIME/.005)^4 :REM DT = 400
4540 IF DT = 300 THEN T = 318.135-18.175*TIME/.005+.7648*(TIME/.005)^2
      -.0216*(TIME/.005)^3+.00029*(TIME/.005)^4 :REM DT = 300
4900 RETURN
4950 REM
4970 REM
5000 REM Material Parameters for polycrystalline alumina
5020 TFS = 193! : REM tfs = thermal fracture stress
5040 DEF FNE(T) = (335.49 - .027*T)*1000 :
      REM Young's modulus [MPa]
5050 DEF FNA(T) = (6.05 + .019*T - .000019*(T^2))*0.000001 :
      REM Thermal expansion [/C]
5060 DEF FNV(T) = .233 - .0000149*T : REM Poisson's ratio
5070 DEF FNK(T) = .205 - .000319*T + 1.9E-07*T^2 :
      REM Thermal conductivity [W/cm C]
5080 TH = .05: IF DT = 210 THEN TH = .1 :
      REM Specimen half thickness in cm
5090 P = 3.75 : REM Mass density [g/cm3]
5100 DEF FNC(T) = .7914 + .001407*T - 1.63E-06*T^2 + 7.029E-10*T^3 :
      REM Heat capacity [J/g C]
5300 RETURN
5350 REM

```

```

5400 REM
6000 REM Material Parameters for SiC whisker reinforced
      polycrystalline alumina
6020 TFS = 193 :REM TSF = thermal fracture stress
      (assume that the matrix is same as the unreinforced alumina.)
6040 DEF FNE(T) = (409.5 - .047*T) * 1000 :
      REM Young's modulus [MPa]
6050 DEF FNA(T) = (5.13 + .0057*T + .0000045*(T^2))*0.000001 :
      REM Thermal expansion [/C]
6060 DEF FNV(T) = .22 : REM Poisson's ratio
6070 DEF FNK(T) = .35 - .000682*T + 5.1E-07*T^2 :
      REM Thermal conductivity [W/cm C]
6080 TH = .15 : REM Specimen half thickness [cm]
6090 P = 3.82 : REM Mass density [g/cm3]
6100 DEF FNC(T) = .7620 + .001560*T - 1.95E-06*T^2 + 0.919E-09*T^3 :
      REM Heat capacity [J/g C]

6300 RETURN
6350 REM
6400 REM Material property of titanium diboride
6500 DEF FNC(T) = .6084 + .001616*T - 2.007E-06*T^2 + 9.2E-10*T^3 :
      REM Heat capacity [J/ g C]
6520 TFS = 308 :REM TSF = thermal fracture stress
      (probably bend strength)
6540 DEF FNE(T) = (496.7 - .038*T)*1000 :
      REM Young's modulus [MPa]
6550 DEF FNA(T) = (4.101+ .01013*T - .0000054*(T^2))*0.000001 :
      REM Thermal expansion [/C]
6560 DEF FNV(T) = .00987 + 1.1375E-05*T : REM Poisson's ratio
6570 DEF FNK(T) = .322- 8.273E-06*T + 6.51E-08*T^2 -8.783001E-12*T^3 :
      REM Thermal cond. [W/cm C]
6580 TH = .08 : REM Specimen half thickness [cm]
6590 P = 4.45 : REM Mass density [g/cm3]
6800 RETURN
6840 REM
6880 REM
7000 REM Error function calculation
7100 REM **THIS SERIES IS VALID WHEN 0 < X < 3
7160 ERFA = 1! - X^2/3
7180 ERFB = (X^4/2) * (1/5 - X^2/21)
7200 ERFC = (X^8/24)* (1/9 - X^2/(5*11))
7220 ERFD = (X^12/(6*5*4*3*2))*(1/13 - X^2/(7*15 ))
7240 ERFE = (X^16/(8*7*6*5*4*3*2))*(1/17 - X^2/(9*19))
7260 ERFF = (X^20/(90*56*30*24))*(1/21 - X^2/(11*23))
7265 ERFG = (X^24/(90*56*30*24*132))*(1/25 -X^2/(13*27))
7270 ERFH = (X^28/87178291200#)*(1/29 -X^2/(15*31))
7275 ERFI = (X^32/20922789890000#)*(1/33 -X^2/(17*35))
7280 ERFJ = (X^36/6402373705000000#)*(1/37 -X^2/(19*39))
7285 ERFK = (X^40/2.432902008D+18)*(1/41 -X^2/(21*43))
7290 ERFL = (X^44/1.124000728D+21)*(1/45 -X^2/(23*47))
7300 ERFM = (X^48/6.204484016D+23)*(1/49 -X^2/(25*51))

```

```

7305      ERFN = (X^52/4.03291461D+26)*(1/53 -X^2/(27*55))
7310      ERFO = (X^56/3.048883446D+29)*(1/57 -X^2/(29*59))
7320      ERFP = (X^60/2.652528598D+32)*(1/61 -X^2/(31*63))
7330      ERFQ = (X^64/2.631308368D+35)*(1/65 -X^2/(33*67))
7370      ERF = (2*X/PI^.5)*(ERFA + ERFB + ERFC + ERFD + ERFE
          + ERFF + ERFG + ERFH + ERFI + ERFJ + ERFK + ERFL +
          ERFM + ERFN + ERFO + ERFP + ERFQ)

7400 RETURN
7420 REM
7440 REM
7500 REM Stress calculation, according to Cheng
7580      SUBSTRESS = - (2*X*R/PI^.5 +(1 + R)*((1 - ERF)*EXP(X^2) - 1))
7590      STRESS = FNE(T)*FNA(T)*DT*SUBSTRESS/(1 - FNV(T))
7600      PRINT USING A$; F, X, ERF, SUBSTRESS, STRESS
7650      REM WRITE #2, F, STRESS
7800 RETURN
7820 REM
7840 REM
8000 REM Numerical integration
8100 REM increase subdivision untill no changes was found in 1/100
      digit
8140      SUMAREA = SUMAREA +(STRESS*DF)
8150      REM PRINT "sumstress = ";SUMSTRESS
8900 RETURN

```

LIST OF REFERENCES

REFERENCES

1. H. Ohira and R. C. Bradt, "Strength Distribution of a Quench-Strengthened Aluminosilicate Ceramic", J. Amer. Ceram. Soc., 71 [1]: 35-41, 1988.
2. W. P. Rogers, A. F. Emery, R. C. Bradt and A. S. Kobayashi, "Statistical Study of Thermal Fracture of Ceramic Materials in the Water Quench Test", J. Amer. Ceram. Soc., 70 [6]: 406-412, 1987.
3. M. Ashizuka, T. E. Easler and R. C. Bradt, "Statistical Study of Thermal Shock Damage of a Borosilicate Glass", J. Amer. Ceram. Soc., 66 [8]: 542-550, 1983.
4. W. J. Lee, "Characterization of Composites by Internal Friction and Elastic Modulus Measurements", M. S. Thesis, Michigan State University, East Lansing, Michigan, 1988.
5. C. C. Chiu and E. D. Case, "Influence of Quenching on Fracture Strength, Elastic Modulus, and Internal Friction of Glass Plates", Accepted for publication J. Mat. Sci.
6. H. M. Chou, "Studies on Thermal Shock and Crack Healing Behavior for Polycrystalline Yttrium iron Garnet (YIG)", Ph. D Dissertation, Michigan State University, East Lansing, Michigan, 1988.
7. C. E. Semler, Jr., T. H. Hawisher and R. C. Bradt, "Thermal Shock of Alumina Refractories: Damage-Resistance Parameter and the Ribbon Test", Amer. Ceram. Soc. Bull., 60 [7]: 724-729, 1981.
8. K. Matsushida, S. Kuratani, T. Okamoto and M. Shimada, "Young's Modulus and Internal Friction in Alumina Subjected to Thermal Shock", J. Mater. Sci. Lett., 3 [4]: 345-348, 1984.
9. S. Nishijima, K. Matsushita, T. Okada, T. Okamoto and T. Hagiwara, "Dynamic Young's Modulus and Internal Friction in Composite Materials", pp 143-151 in Nonmetallic Materials and Composites at Low Temperature 3, edited by G. Hartwig and D. Evans, Plenum Press, New York and London, 1986.
10. J. C. Coppola and R. C. Bradt, "Thermal-Shock Damage in SiC", J. Amer. Ceram. Soc., 56 [4]: 214-18, 1973.

11. Y. Kim, W. J. Lee, and E. D. Case, "Thermal Fatigue in SiC Fiber Reinforced Aluminosilicate Glass Ceramic Composite", pp 479-486, in Metal and Ceramic Matrix Composites: Processing, Modeling and Mechanical Behavior, Edited by R. B. Bhagat, A. H. Clauer, P. Kumar and A. M. Ritter, The Minerals, Metals and Materials Society, 1990.
12. Y. Kim, W. J. Lee and E. D. Case, "Thermal Fatigue Behavior of Ceramic Matrix Composites: A Comparison Among Fiber Reinforced, Whisker Reinforced, and Monolithic Ceramics", pp 871-881 in Proceedings of the American Society for Composites, 5th Technical Conference, 1990.
13. W. J. Lee and E. D. Case, "Cyclic Thermal Shock in SiC Whisker/Alumina Composites", Mater. Sci. and Eng., A119: 113-126, 1989.
14. W. J. Lee and E. D. Case, "Thermal Fatigue in Polycrystalline Alumina", J. Mater. Sci., 25: 5043-5054, 1990.
15. J. P. Singh, Y. Tree and D. P. H. Hasselman, "Effect of Bath and Specimen Temperature on Thermal Stress Resistance of Brittle Ceramics Subjected to Thermal Quenching", J. Mat. Sci., 16: 2109-2118, 1981.
16. A. G. Evans and E. A. Charles, "Structural Integrity in Severe Thermal Environments", J. Am. Ceram. Soc., 60[1-2]: 22-28, 1977.
17. H. P. Kirchner, R. E. Walker and D. R. Platts, "Strengthening Alumina by Quenching in Various Media", J. Appl. Phys., 42[10]: 3685-3692, 1971.
18. D. B. Marshall, M. D. Drory, R. L. Loh and A. G. Evans, "The Thermal Fracture of Alumina", in Fracture in Ceramic Materials pp 336-363, edited by A. G. Evans, Noyes Publication, NJ, 1984.
19. N. Kamiya and O. Kamigato, "Thermal Fatigue Life of Glass Subjected to Air Blast Quenching", J. Mat. Sci., 24: 2461-2466, 1989.
20. K. Nihara, J. P. Singh and D. P. H. Hasselman, "Observations on the Characteristics of a Fluidized Bed for the Thermal Shock Testing of Brittle Ceramics", *ibid*, 17: 2553-2559, 1982.
21. C. M. Cheng, "Resistance to Thermal Shock", J. Am. Rocket Soc., 21[6]: 147-153, 1951.
22. R. W. Hertzberg, pp. 521-523 in Deformation and Fracture Mechanics of Engineering Materials, Second Edition, John Wiley and Sons, NY, 1983.

23. B. R. Lawn, B. J. Hockey, and S. M. Wiederhorn, "Atomically Sharp Cracks in Brittle Solids: an Electron Microscopy Study", J. Mater. Sci., 15: 1207-1223, 1980.
24. M. J. Reece, F. Guiu and M. F. R. Sammur, "Cyclic Fatigue Crack Propagation in Alumina under Direct Tension-Compression Loading", J. Amer. Ceram. Soc., 72 [2]: 348-352, 1989.
25. T. Hoshida, T. Ohara and T. Yamada, "Fatigue Crack Growth from Indentation Flaw in Ceramics", Int. J. Frac., 37: 47-59, 1988.
26. A. Grossmuller, V. Zelizko and M. V. Swain, "Fatigue Crack Growth in Ceramics Using a Compressive loading geometry", J. Mat. Sci. Lett., 8 [1]: 29-31, 1989.
27. R. H. Dauskardt, W. Yu and R. O. Ritchie, "Fatigue Crack Propagation in Transformation-Toughened Zirconia Ceramic", J. Amer. Ceram. Soc., 70 [10]: C-248-C-252, 1987.
28. K. Kim and A. Mubeen, "Relationship Between Differential Stress Intensity Factor and Crack Growth Rate in Cyclic in Westerly Granite", in Fracture Mechanics Methods for Ceramics, Rocks, and Concrete, ASTM STP 745, pp. 157-168, edited by S. W. Freiman and E. R. Fuller, Jr., American Society for Testing and Materials, 1981.
29. A. G. Evans, "Fatigue in Ceramics", Lawrence Berkeley Laboratory Report LBL-9529, 1979.
30. A. G. Evans and R. M. Cannon, "Toughening of Brittle Solids by Martensitic Transformation", Acta Metall., 34 [5]: 761-800, 1986.
31. D. B. Marshall, M. D. Drory and A. G. Evans, "Transformation Toughening in Ceramics" in Fracture Mechanics of Ceramics, Vol. 6, pp. 289-307, edited by R. C. Bradt, A. G. Evans, D. P. H. Hasselman and F. F. Lange, Plenum Press, NY, 1983.
32. K. T. Faber and A. G. Evans, "Crack Deflection Processes I, Theory", Acta Metall., 31 [4]: 565-576, 1983.
33. K. T. Faber and A. G. Evans, "Crack Deflection Processes II, Experiment", *ibid.*, 31 [4]: 577-584, 1983.
34. P. L. Swanson, C. J. Fairbanks, B. R. Lawn, Y-M Mai and B. J. Hockey, "Crack-Interface Grain Bridging as a Fracture Resistance Mechanism in Ceramics", J. Amer. Ceram. Soc., 70 [4]: 279-289, 1987.

35. J. R. Brockenbrough and S. Suresh, "Constitutive Behavior of a Microcracking Brittle Solid in Cyclic Compression", J. Mech. Phys. Solids, 35 [6]: 721-742, 1987.
36. L. Ewart and S. Suresh, "Dynamic Fatigue Crack Growth in Polycrystalline Alumina in Cyclic Compression", J. Mater. Sci. Lett., 5 [8]: 774-778, 1986.
37. L. Ewart and S. Suresh, "Crack Propagation in Ceramic under Cyclic Loads", J. Mater. Sci., 22 [4]: 1173-1192, 1987.
38. E. K. Tschegg and S. Suresh, "Torsional Fracture of Fatigue Pre-Cracked Ceramic Rods", *ibid.*, 22 [8]: 2927-2932, 1987.
39. S. Suresh and L. X. Han, "Fracture of Si_3N_4 -SiC Whisker Composites under Cyclic Loads", J. Amer. Ceram. Soc., 71 [3]: C-158-C-161, 1988.
40. A. A. Morrone, S. R. Nutt and S. Suresh, "Fracture Toughness and Fatigue Crack Growth Behavior of an Al_2O_3 -SiC Composite", J. Mater. Sci., 23 [9]: 3206-3213, 1988.
41. S. Suresh and L. A. Sylva, "Room Temperature Fatigue Crack Growth in Cemented Carbide", Mater. Sci. Eng., 83 [1]: L7-L10, 1986.
42. F. A. McClintock and J. B. Walsh, in Proceedings of 4th US National Congress on Applied Mechanics, pp 1015, American Society of Mechanical Engineers, New York, 1962.
43. V. Tvergaard and J. W. Hutchinson, "Microcracking in Ceramics Induced by Thermal Expansion or Elastic Anisotropy", J. Amer. Ceram. Soc., 71 [3]: 157-166, 1988.
44. D. Lewis, "Cyclic Mechanical Fatigue in Ceramic-Ceramic Composites--An Update", Ceram. Eng. Sci. Proc., 4 [9-10]: 874-881, 1983.
45. D. Lewis and R. W. Lewis, "Thermal Shock Fatigue of Monolithic Ceramics and Ceramic-Ceramic Composites", *Ibid.*, 2 [7-8]: 712-718, 1981.
46. D. Lewis and R. W. Rice, "Comparison of Static, Cyclic, and Thermal Shock Fatigue in Ceramic Composites", *Ibid.*, 5 [9-10]: 714-721, 1982.
47. M. E. Lu and A. G. Evans, "Influence of Cyclic Tangential Loads on Indentation Fracture", J. Am. Ceramic Soc., 68 [9]: 505-510, 1985.

48. N. Brugger, "Verhalten von Rissen in keramischen Materialien unter statischer, langsam ansteigender und schwellender Belastung", Inst. fuer Material und Festkoeperforschung, 143 pages, June 1987.
49. R. L. Fullman, "Measurement of Particle Sizes in Opaque bodies", Trans. AIME, 197 [3]: 447-452, 1953.
50. F. Forster, "Ein neues Messverfahren zur Bestimmung des Elastizitätsmoduls und der Dämpfung" (A New Method for Determination of Modulus of Elasticity and Damping), Zeitschrift fur Metallkunde, 29[4]: 109-115, 1937.
51. E. Schreiber, O. L. Anderson, and N. Soga, Elastic Constants and Their Measurements, Chap. 4, McGraw-Hill, New York, 1974.
52. S. Spinner and W. E. Tefft, "A Method for Determining Mechanical Resonance Frequencies and for Calculating Elastic Moduli from These Frequencies", ASTM Proc., 61: 1221-1238, 1961.
53. G. Pickett, "Equations for Computing Elastic Constants from Flexural and Torsional Resonant Frequencies of Vibration of Prism and Cylinder", ASTM Proc., 45: 846-865, 1945.
54. D. P. H. Hasselman, Tables for the Computation of Shear Modulus and Young's Modulus of Elasticity from Resonant Frequencies of Rectangular Prisms, Carborundum Co., Niagara Falls, NY, 1961.
55. J. B. Wachtman, Jr. and W. E. Tefft, "Effect of Suspension on Apparent Values of Internal Friction Determined by Forster's Method", Rev. Sci. Instr., 29[6]: 517-520, 1959.
56. L. Rayleigh, pp. 253-83 in The Theory of Sound, vol.1, Dover Publication, New York, 1976.
57. T. N. Tiegs and P. F. Becher, "Thermal Shock Behavior of an Alumina-SiC Whisker Composite", J. Amer. Ceram. Soc., 70 [5]: C-109-C-111, 1987.
58. W. H. Gitzen, p 65 in Alumina as a Ceramic Material, American Ceramic Society, Columbus, OH, 1970.
59. B. R. Lawn and D. B. Marshall, "Hardness, Toughness, and Brittleness: An Indentation Analysis", J. Amer. Ceram. Soc., 62 [7-8]: 347-350, 1979.
60. G. R. Anstis, P. Chantikul, B. R. Lawn and D. B. Marshall, "A Critical Evaluation of Indentation Techniques for Measuring Fracture Toughness: I, Direct Crack Measurements", J. Am. Ceram. Soc., 64[9]: 533-538, 1981.

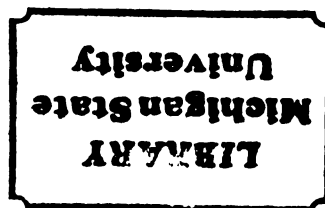
61. J. V. Beck and K. J. Arnold, pp 234-247 in Parameter Estimation in Engineering and Science, Wiley Series in Probability and Mathematical Statistics, John Wiley & Sons, New York, 1977.
62. J. V. Beck, B. Blackwell and C. R. St. Clair, Jr., pp 290-294 in Inverse Heat Conduction: Ill-Posed Problems, Wiley-Interscience, New York, 1985.
63. E. D. Case, J. R. Smyth, and O. Hunter, Jr., pp. 507-530 in Fracture Mechanics of Ceramics, Vol. 5, edited by R. C. Bradt, A. G. Evans, D. P. H. Hasselman, and F. F. Lange, Plenum Press, NY, 1983.
64. W. R. Manning and O. Hunter, Jr., "Elastic Properties of Polycrystalline Yttrium Oxide, Holmium Oxide, and Erbium Oxide; High-Temperature Measurements", J. Amer. Ceram. Soc., 52 [9]: 492-496, 1969.
65. J. A. Haglund and O. Hunter, Jr., "Elastic Properties of Polycrystalline Monoclinic Gadolinia", Ibid., 55 [6]: 327-330, 1973.
66. S. L. Dole, O. Hunter, Jr., F. W. Calderwood, and D. J. Bray, "Microcracking of Monoclinic Hafnia", J. Amer. Ceram. Soc., 61: 472-486, 1978.
67. E. Bonetti, E. Evangelista, and P. Gondi, pp. 401-405 in "High Temperature Damping in Silicon Nitride Depending on the Sintering Conditions", Internal Friction and Ultrasonic Attenuation in Solids, Pergamon Press, NY, 1980.
68. S. L. Dole, O. Hunter, Jr., and C. J. Wooge, "Elastic Properties of Monoclinic Hafnium Oxide at Room Temperature", J. Amer. Ceram. Soc., 60 [11-12]: 488-490, 1977.
69. C. T. Herakovich and M. W. Hyer, "Damage-Induced Property Changes in Composites Subjected to Cyclic Thermal Loading", Eng. Frac. Mec., 25 [5-6]: 779-791, 1986.
70. J. H. Ainsworth and R. H. Herron, "Thermal Shock Damage Resistance of Refractories", Amer. Ceram. Soc. Bull., 53 [7]: 533-538, 1974.
71. Youngman Kim, "Cyclic Thermal Shock in Ceramics and Ceramic Matrix Composites", Ph. D Dissertation, Michigan State University, East Lansing, MI, 1991.
72. C. E. Semler and T. H. Hawisher, "Evaluation of the Thermal Shock Resistance of Refractories Using the Ribbon Test Method", Amer. Ceram. Soc. Bull., 59 [7]: 732-738, 1980.

73. J. W. Lemmens, "Impulse Excitation: A Technique for Dynamic Modulus Measurement" pp. 90-99 in Dynamic Elastic Modulus Measurement in Materials, ASTM STP 1045 edited by A. Wolfenden, American Society for Testing and Materials, 1989.
74. C. Sheu and R. Scrutton, "Quenching, Polishing, and Grinding Reaction-Bonded Silicon Nitride", Amer. Ceram. Soc. Bull., 69 [7]: 1148-1151, 1990.
75. S. Liu and J. A. Nairn, "Fracture Mechanics Analysis of Composite Microcracking: Experimental Results in Fatigue", pp 287-295 in Proceeding of the American Society for Composites, 5th Technical Conference, 1990.
76. R. L. Salganik, "Mechanics of Bodies with Many Cracks", Mech. Solids, 8 [4]: 135, 1973.
77. B. Budiansky and R. J. O'Connell, "Elastic Moduli of a Cracked Solid ", Int. J. Solids Structures, 12: 81-97, 1975.
78. S. L. Ogin, P. A. Smith, and P. W. R. Beaumont, "Matrix Cracking and Stiffness Reduction of (0/90)s GFRP Laminate", Com. Sci. & Tech., 22: 23-31, 1985.
79. W. D. Kingery, H. K. Bowen and D. R. Uhlmann, Chap. 12 and 16 in Introduction to Ceramics, Second Edition, John Wiley and Sons, NY, 1976.
80. H. S. Carslaw and J. C. Jaeger, Conduction of Heat in Solids, Clarendon Press, Oxford, England, 1959.
81. D. B. Marshall, B. N. Cox, and A. G. Evans, "The Mechanics of Matrix Cracking in Brittle-Matrix Fiber Composites", Acta Metall., 33 [11]: 2013-2021, 1985.
82. D. B. Marshall and A. G. Evans, "The Tensile Strength of Uniaxially Reinforced Ceramic Fiber Composites", pp. 1-15 in Fracture Mechanics of Ceramics, vol. 7, edited by R. C. Bradt, A. G. Evans, D. P. H. Hasselman, and F. F. Lange, Plenum Press, NY, 1985.
83. G. Simmons and H. Wang, Single Crystal Elastic Constants and Calculated Aggregate Properties: A Handbook, The M. I. T. Press, Cambridge, MA, 1971.
84. J. C. Glandus and P. Boch, "Energy and Ceramics", pp. 661 in Proceeding of the 4th International Meeting on Modern Ceramic Technologies, edited by P. Vincenzini, 1979.

85. D. Cawley and R. D. Adams, "Vibration Techniques", pp 151-200 in Non-Destructive Testing of Fiber Reinforced Plastic Composites, Vol. 1, edited by J. Summerscales, Elsevier Applied Science, NY, 1987.
86. S. M. Ross, pp. 44-51 in Introduction of Probability Models, Academic Press, New York, 1972.
87. E. Parzen, pp. 11-19 in Stochastic Processes, Holden-Day, San Francisco, 1962.
88. B. A. Boley and J. H. Weiner, pp. 400-406 Theory of Thermal Stresses, John Wiley and Sons, New York, 1960.
89. S. L. Dole, O. Hunter, Jr. and C. J. Wooge, "Elastic Properties of Monoclinic Hafnium Oxide at Room Temperature", J. Amer. Ceram. Soc. 60 [11-12]: 488-90, 1977.
90. D. F. Porter, J. S. Reed, and D. Lewis III, "Elastic Moduli of Refractory Spinel", *ibid.*, 60 [7-8]: 345-349, 1977.
91. S. L. Dole, O. Hunter, Jr. and F. W. Calderwood, "Elastic Properties of Polycrystalline Scandium and Thulium Sesquioxides", *ibid.*, 60 [3-4]: 167-168, 1977.
92. B. R. Powell, Jr., O. Hunter, Jr. and W. R. Manning, "Elastic Properties of Polycrystalline Ytterbium Oxide", *ibid.*, 54 [10]: 488-490, 1971.
93. V. D. Krstic and W. H. Erickson, "A model for the Porosity Dependence of Young's Modulus in Brittle Solids Based on Crack Opening Displacement", J. Mater. Sci., 22 [8]: 2881-2886, 1987.
94. S. S. Manson, Chap. 7 in Thermal Stress and Low-Cycle Fatigue, McGraw-Hill, New York, 1966.
95. K. M. Prewo, "Fatigue and stress rupture of silicon carbide fiber-reinforced glass-ceramic", J. Mat. Sci., 22: 2695-2701, 1987.
96. J. J. Brennan and K. M. Prewo, "Silicon Carbide Fiber Reinforced Glass-Ceramic Matrix Composites exhibiting High Strength and Toughness", J. Mat. Sci., 17: 2371-2383, 1982.
97. Provided Data by Manufacturer (Specialty Materials Division, Eagle-Picher Industries, Inc.).
98. M. K. Ferber, P. F. Becher and C. B. Finch, "Effect of Microstructure on the Properties of TiB_2 Creamics", J. Am. Ceram. Soc., 66 [1]: C2-C4, 1982.

99. Z. Strnad, pp 226-230, in Glass-Ceramic Materials (Glass Science and Technology: Vol. 8), Elsevier, New York, 1986.
100. P. F. Becher, C. Hsueh, P. Angelini and T. N. Tiegs, "Toughening Behavior in Whisker-reinforced Ceramic Matrix Composites", J. Am. Ceram. Soc., 71 [12]: 1050-1061, 1988.
101. P. F. Becher and G. C. Wei, "Toughening Behavior in SiC Whisker-reinforced Alumina", *ibid*, 67 [12]: C267-C269, 1984.
102. D. R. Biswas and V. K. Pujari, "Verification of the Double-Torsion Equation by using Different Thickness Samples of a Machinable Glass-Ceramic", *ibid*, 64 [7]: C98-C99, 1981.
103. P. F. Becher, "Effect of Water Bath Temperature on the Thermal Shock of Al_2O_3 ", J. Am. Ceram. Soc. Comm., 64[1]: c17-c18, 1981.
104. P. F. Becher, D. Lewis III, K. R. Carman and A. C. Gonzales, "Thermal Shock Resistance of Ceramics: Size and Geometry Effects in Quench Tests", Bull. Am. Ceram. Soc., 59[5]: 542-548, 1980.
105. M. N. Ozisik, Heat Transfer, Chap. 10, McGraw-Hill, New York, 1985.
106. J. P. Holman, Heat Transfer, Fourth Edition, Chap. 9, McGraw-Hill, New York, 1976.
107. G. Stolz, Jr., V. Paschkis, C. F. Bonilla and G. Acevedo, "Thermal Considerations in Oil Quenching", J. Iron and Steel Institute, 193: 116-123, 1959.
108. T. Hachisu, T. Sakai and K. Taguchi, "Transient Temperature Distribution in Circular Cylinders During Quenching", Heat Transfer; Japanase Research, 10[1]: 52-64, 1981.
109. H. J. Sauer, Jr., and K. M. Ragsdell, "Film Pool Boiling of Nitrogen from Flat Surface", pp. 412-415 in Advances in Cryogenic Engineering Vol 16, edited by K. D. Timmerhaus, Plenum Press, New York, 1971.
110. F. P. Incropera and D. P. Dewitt, p. 431 in Introduction to Heat Transfer, John Wiley & Sons, New York, 1985.
111. Y. Kim, W. J. Lee, and E. D. Case, "The Measurement of the Surface Heat Transfer Coefficient for Ceramics Quenched into a Water Bath", Accepted for the publication in Mat. Sci. Eng.

112. M. Hoch and A. Silberstein, "Thermal Conductivity of Aluminum Oxide", pp. 509-526 in Proceedings of the Sixth Conference on Thermal Conductivity, edited by M. L. Minges and G. L. Denman, Air Force Materials Laboratory, Dayton, Ohio, 1966.
113. C. D. Hodgmand, R. C. Weast and S. M. Selby, pp. 3156 in Handbook of Chemistry and Physics, 37th ed., Chemical Rubber Publishing Co., Cleveland, Ohio, 1955.
114. Provided data by Manufacturer (Structural Division, Coors Ceramics Co.)
115. E. D. Case and A. G. Evans, "Length of Maximal Impact Damage Cracks as a Function of Impact Velocity" pp. 404-415 in Fracture in Ceramic Material, edited by A. G. Evans, Noyes Publication, NJ, 1984.
116. D. P. H. Hasselman, "Unified Theory of Thermal Shock Fracture Initiation and Crack Propagation in Brittle Ceramics", J. Am. Ceram. Soc., 52 [11]: 600-604, 1969.



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