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CRACK BARRIERS IMPROVE THE MECHANICAL AND THERMAL PROPERTIES
OF NON-METALLIC SINTER MATERIALS

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(NASA-TM-75426) CRACK BARRIERS IMPROVE THE
MECHANICAL AND THERMAL PROPERTIES OF
NON-METALLIC SINTER MATERIALS (National
Aeronautics and Space Administration) 16 p
HC A02/NF A01 CSCI 11D G3/24

N79-24065

Unclass
20912

Translation of "Rissbarrieren verbessern die mechanischen und thermischen Eigenschaften nichtmetallischer Sinterwerkstoffe", Plansee Seminar, 8th, Reutte, Austria, May 27-30, 1974, Preprints, Vol. 3, 18 pp.



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D. C. 20546 APRIL 1979

STANDARD TITLE PAGE

1. Report No. NASA TM-75426	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle CRACK BARRIERS IMPROVE THE MECHANICAL AND THERMAL PROPERTIES OF NON-METALLIC SINTER MATERIALS		5. Report Date April 1979	
		6. Performing Organization Code	
7. Author(s) K. H. Gruenthaler, W. Heinrich, S. Janes and J. Nixdorf		8. Performing Organization Report No.	
		10. Work Unit No.	
9. Performing Organization Name and Address SCITRAN Box 5456 Santa Barbara, CA 93108		11. Contract or Grant No. NASW-3198	
		13. Type of Report and Period Covered Translation	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		14. Sponsoring Agency Code	
		15. Supplementary Notes Translation of: "Rissbarrieren verbessern die mechanischen und thermischen Eigenschaften nichtmetallischer Sinterwerkstoffe", Plansee Seminar, 8th, Reutte, Austria, May 27-30, 1974, Preprints, Vol. 3, 18 pp. (A74-36098).	
16. Abstract Means of improving the tensile strength of ceramic composites by introducing ductile intermediate layers capable of absorbing the elastic energy at the rupture front are studied. Tests with an Al ₂ O ₃ laminate with niobium inclusions showed that crack propagation could be successfully precluded by dissipation of the energy by deformation and/or delamination at the inclusion/matrix interface.			
17. Key Words (Selected by Author(s))		18. Distribution Statement Unclassified - Unlimited	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 16	22.

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and J. Nixdorf*

1. Introduction

For high temperature applications, ceramic substances are superior to metals with regard to pressure resistance, creep resistance, and oxidation resistance. The low tensile, bending and shock resistance and the poor alternating temperature resistance due to deficient ductility limit the application potentials of ceramic substances. /1**

As for all metals, the tensile strength of polycrystalline ceramic substances is far below that value resulting from the atomic bonding forces in the crystal lattice. It is assumed in the theory of brittle fracture that micro-cracks are present in the material. The spontaneous crack propagation leading to fracture occurs when the stored elastic energy becomes greater than the energy needed to form the break surface. If σ is the effective tensile stress and E the elasticity modulus of the material, then the elastic energy per unit volume is:

$$u = \frac{\sigma^2}{2E} \quad (1)$$

If by γ_{eff} we mean the effective surface fracture energy of all

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**Numbers in margin indicate pagination in original foreign text.

mechanisms affecting crack propagation — primarily the formation of new surfaces (surface energy) and anelastic deformation — then for the newly formed surface, we have:

$$\Delta \leq \frac{U}{\gamma_{eff}} \quad (2)$$

If C is the length (at the surface) or half length (in the interior) of the crack, then according to Griffith [1] the critical tensile stress for crack propagation is:

$$\sigma_K = \sqrt{\frac{2 E \gamma_{eff}}{\pi c}} \quad (3)$$

Thus, the tensile strength of brittle materials increases for reductions in the size of existing cracks and for increases in surface fracture energy. If the critical tensile stress can be increased by appropriate means, then the shock resistance and alternating temperature resistance are improved, since the material is able to absorb a greater energy or endure a greater self-stress under rapid temperature change.

The size of the existing lattice faults can be reduced by surface treatment, by reducing the granular size and decreasing the porosity [2]. The possibilities for increasing tensile strength by embedding are discussed below.

2. Improving mechanical properties of ceramic substances by inclusions

The reinforcement of oxide and special ceramics with embedded fibers in the actual sense through stress transfer encounters significant difficulties. The requirement for a reinforcement that the E-modulus of the fibers must exceed that of the matrix is met only by a few fibrous materials. In all other cases, reinforcement can only occur when the matrix is prestressed by the fibers which must be released upon tensile stress of the composite material [3]. The generation of a preliminary pressure stress on the matrix is possible when the fibers have a larger thermal elongation coefficient

than the matrix so that after cooling from the fabrication temperature, the fibers are under tensile stress.

The restrictive prerequisites are: sufficiently large values of flow limit and E-modulus of the fibers, sufficient thermodynamic stability of fiber and matrix material under the conditions of manufacture of the composite material and a good bonding of the components.

Embedding of ductile materials is very promising for crack restriction. A crack can be prevented from expansion if it encounters a material capable of dissipating the existing elastic energy through energy absorbing mechanisms. In addition to plastic deformation of the embedded materials, delaminations between the composite components will increase the surface rupture energy.

When embedding discontinuous fibers, energy is consumed by friction arising from "pull-out" effects [4]. If σ_{2Bf} is the fiber tensile strength, τ_{f-m} the shear strength between fiber and matrix, V_f/V_{fm} the fiber vol.-%, and d the fiber diameter, then the maximum rupture work per surface unit is [5]:

$$G = \frac{1}{2d} \frac{V_f}{V_{fm}} \frac{d \cdot \sigma_{2Bf}^2}{\tau_{f-m}} \quad (4)$$

Metallic inclusions improve properties by increasing the surface rupture energy and improve alternating temperature resistance as a result of their greater thermal conductance [6].

The development of Cermet (ceramic-metal composites) has shown that an improvement in ductility can be attained by metallic inclusions in ceramic substances [7].

At Battelle Institute, the effectiveness of crack barriers made of ductile material was studied on the Al_2O_3 -niobium system [8].
The composite materials are sufficiently compatible at temperatures up to 1500° C [9].

The following material values are valid for dense Al_2O_3 (index m) and niobium (index f) of 99.9/99.8% purity at a temperature of 500°C [10]:

a) Tensile strength

Elastic limit of f: $\sigma_{sf} = 19 \text{ kp/mm}^2$
Tensile strength of m: $\sigma_{zlm} = 26 \text{ kp/mm}^2$

b) Elastic modulus

$E_f = 11600 \text{ kp/mm}^2$
 $E_m = 36000 \text{ kp/mm}^2$

c) Transverse contraction ratio

$\nu_f^u = 0,38$
 $\nu_m^u = 0,32$

d) Linear thermal elongation coefficient

$\alpha_f = 7,9 \cdot 10^{-6}/^\circ\text{C}$ (between 0 and 1000°C)
 $\alpha_m = 8,3 \cdot 10^{-6}/^\circ\text{C}$ (between 0 and 1000°C)

e) Thermal conductivity

$k_f = 0,14 \text{ cal/cm s } ^\circ\text{C}$
 $k_m = 0,026 \text{ cal/cm s } ^\circ\text{C}$

Owing to the fact that the thermal elongation coefficient of Al_2O_3 and niobium are approximately equal, a shaped part of this bonded combination is generally free of eigenstresses.

For bending tests, we prepared plates of Al_2O_3 with layered embedded niobium foils. We use Al_2O_3 powder with an average granular size of $1 \mu\text{m}$ and $60\text{-}\mu\text{m}$ thick niobium foils. The compaction occurred through pressure sinterization at a temperature of 1500°C , a pressure of 250 kp/mm^2 , and a dwell time of 20 min.

In order to gain information about the adhesion of the bonded components, the interlaminar shear strength was determined in a

3-point bending test with shortened support separation. At a sample thickness to support a separation ratio of 1:3, this value was about 4 kp/mm².

The bending strength of samples with 25 vol.-% embedded niobium foils was about 50 kp/mm². Al₂O₃ samples without embedded material exhibit a value of only about 30 kp/mm².

The increase in bending strength found for Al₂O₃ samples with embedded niobium foils is explained by an increase in fracture surface energy.

From Figure 1 we see the arrangement of a layered composite material comprised of Al₂O₃/niobium. Figure 2 shows the ground image of such a material after applied bending stress.

We see the typical crack profile which is distinguished by the appearance of the crack at the embedded material; energy is consumed through deformation or through delamination at the embedded material/matrix interface, and this prevents crack propagation. The long-running crack branch perpendicular to the direction of stress is visible in Figure 3 — showing the ground image of a break zone. Figures 4 and 5 — scanning electron microscopic images of break surfaces — illustrate that, in contrast to material without embedding, the layered composite material has stepwise formation of the break surfaces, since the crack is diverted at the embedded material.

3. Estimation of alternating temperature resistance of the Al₂O₃-niobium layered composite

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An estimation is given below for the influence of embedding on niobium into Al₂O₃ on alternating temperature resistance. The calculations are based on use of a plate cooled symmetrically from both sides.



Figure 1. Layered composite material niobium- Al_2O_3 (cross section: 50 x magnification)



Figure 3. Ground cross-section through break zone



Figure 2. Crack profile in a layered composite material (ground cross section: 100 x magnification)



Figure 4. Scanning electron microscope photograph of the break surface

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3.1. Alternating temperature resistance of plates made of brittle material

When cooling a plate, the greatest eignestress occurs in the surface layers. If the thermal transfer coefficient is large in comparison to the internal thermal conductance, then the surfaces of the plate immediately assume the temperature of the coolant, whereas the interior is still approximately at the initial temperature. For a small heat transfer coefficient, the maximum tensile stress is attained at that time when the temperature in the middle plain begins to drop. The lower the thermal transfer coefficient, the smaller is the maximum occurring tensile stress. For crack formation in the surface layers, the breaking point must be exceeded at the lattice faults.

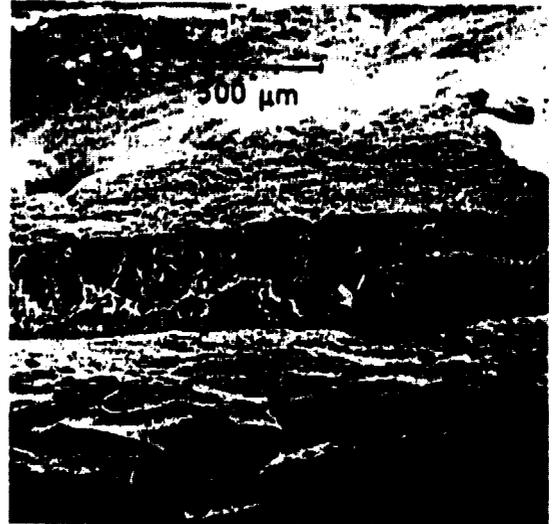


Figure 5. Scanning electron microscope photograph of the break surface

If we assume as an approximation that the material is elastically deformed in an ideal manner until fracture, and its property values are constant over the temperature range under consideration, then for the maximum temperature difference which can be endured without crack formation we have [11]:

$$\Delta T_{\max} = \sigma_{zB} \frac{1 - \mu}{E \alpha} \text{ for } \frac{k}{b h} \rightarrow 0 \quad (5)$$

$$\Delta T_{\max} = \sigma_{zB} \frac{1 - \mu}{E \alpha} (1.5 - 0.5 \exp(-\frac{16 k}{b h})) \text{ for } \frac{1.25 k}{b h} \text{ for } 0 < \frac{k}{b h} < 0.2 \quad (6)$$

$$\Delta T_{\max} = \sigma_{zB} \frac{1 - \mu}{E \alpha} (1.5 + \frac{1.25 k}{b h}) \text{ for } 0.2 < \frac{k}{b h} < \infty \quad (7)$$

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Here, b is half the plate thickness; h is the heat transfer coefficient and k is the thermal conductance; σ_{zB} is the tensile strength; E is the elasticity modulus; α is the linear thermal elongation coefficient; μ is the transverse contraction ratio of the material. /10

From the above equations the following thermal stress parameters are defined for resistance to crack formation:

a) for rapid cooling

$$R_1 = \sigma_{zB} \frac{1 - \mu}{E \alpha} \quad (h \rightarrow \infty) \quad (8)$$

b) for slow cooling

$$R_2 = \sigma_{zB} \frac{1 - \mu}{E \alpha} \cdot K \quad (h \rightarrow 0) \quad (9)$$

In general, surface cracks do not mean the failure of an alternating-temperature stressed body. Rather, we are interested in those factors on which resistance to crack propagation leading to complete destruction will depend.

If K is a geometric factor, then the energy stored upon fracture of a body stressed by thermal shock is [12]:

$$U = K \frac{\sigma_{zB}^2 (1 - \mu)}{E} \quad (10)$$

In accordance with Equations (1) and (2), the newly formed energy upon crack propagation is:

$$A = K \frac{\sigma_{zB}^2 (1 - \mu)}{E \gamma_{ch}} \quad (11)$$

If the entire surface A consists of N individual surfaces of size A_0 , i.e.,

$$A = A_0 \cdot N$$

then we have:

$$A_0 = \frac{K}{N} \frac{\sigma_{zB}^2 (1 - \mu)}{E \gamma_{ch}} \quad (12)$$

If A_0 becomes as large as the cross-sectional surface of the body, then total break occurs. Resistance to break due to thermal shock is greater as A_0 becomes smaller. Accordingly, we define a thermal stress parameter opposing crack expansion as:

$$R_3 = \frac{E \chi_m}{\sigma_{zB}^2 (1 - \nu)} \quad (13)$$

From a comparison of thermal stress parameters R_1 and R_3 , we know that opposing requirements arise for an increase in resistance to crack formation and expansion. With regard to alternating temperature resistance, there is thus an optimum value for the tensile strength of the material.

The level of surface fracture energy γ_{eff} is of decisive importance to the resistance against crack propagation. This can be determined experimentally from the load-bending diagram in the 3-point bending test with notched initial crack [13, 14].

3.2. Material values of the layered composite Al₂O₃-niobium

For the calculation it is assumed that niobium foils (index f) comprising a thickness fraction of $a = 0.4$ are embedded in Al₂O₃ (index m). The layers are oriented parallel to the surface. The material values of the composite are calculated from the values for the components given in section 2.

a) Tensile strength

Since crack formation for alternating temperature stress begins at the brittle matrix, it is assumed for the calculation that the critical tensile stress is determined by the tensile strength of the matrix. Accordingly,

$$\sigma_{zBm} = \sigma_{zBn} = 26 \text{ kp/mm}^2.$$

b) Elasticity modulus

$$E_{fm} = a E_f + (1-a) E_m$$

$$E_{fm} = 26200 \text{ kp/mm}^2$$

c) Transverse contraction ratio

$$\begin{aligned} \nu_{fm} &\approx a \nu_f + (1-a) \nu_m \\ \nu_{fm} &= 0.34 \end{aligned}$$

d) Linear thermal elongation coefficient

$$\begin{aligned} \alpha_{fm} &\approx a \alpha_f + (1-a) \alpha_m \\ \alpha_{fm} &= 8.2 \cdot 10^{-6} / ^\circ\text{C} \end{aligned}$$

e) Thermal conductivity

As equivalent thermal conductivity of a plate composed of thin layers of thickness d_i and thermal conductivity k_i , we have for the case when the heat flow runs normal to the layer [15]:

$$k = \frac{\sum d_i}{\sum \frac{d_i}{k_i}} \quad (14)$$

If layers of thermal conductivities k_f and k_m alternate, then these can be added together to form layers of thickness d_f and d_m . If a and $(1-a)$ are the corresponding thickness fractions in the plate, then we have:

$$k_{fm} = \frac{k_f \cdot k_m}{(1-a) k_f + a k_m} \quad (15)$$

A significant increase in thermal conductivity of the composite over the Al_2O_3 matrix is not attained until a high thickness fraction of niobium foil is present (Figure 6). For $a = 0.4$, an increase of 50% yields:

$$k_{fm} = 0.039 \text{ cal/cm s } ^\circ\text{C.}$$

3.3. Alternating temperature resistance of the Al_2O_3 niobium composite with regard to crack formation

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Calculation of the alternating temperature resistance proceeds from Equations (5) to (7) using material data in section 3.2. In Figure 7, we see the maximum temperature difference which is endured

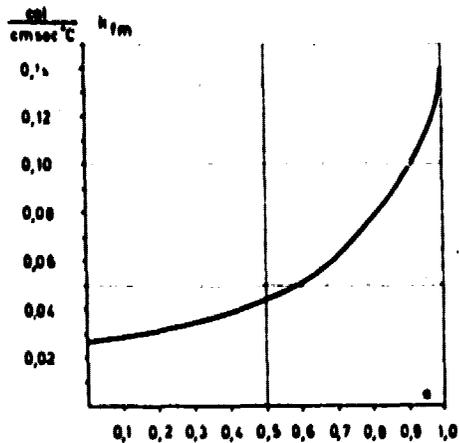


Figure 6. Thermal conductivity of the layered composite Al_2O_3 -niobium (at $500^\circ C$) as a function of the thickness fraction of niobium for thermal flow normal to the layers

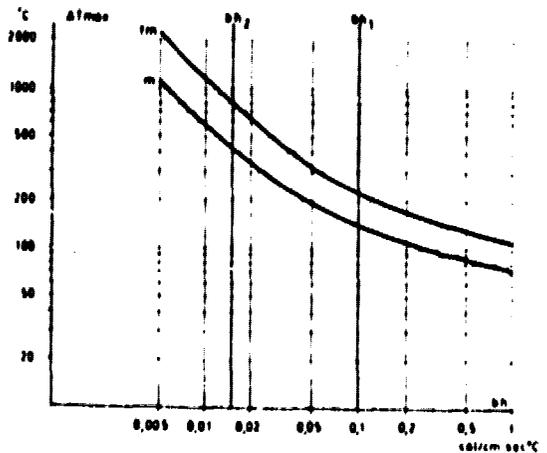


Figure 7. Calculated alternating temperature resistance of a plate of thickness $2b$, made of Al_2O_3 -niobium layered composite (Ni thickness-% = 0.4) or of Al_2O_3 as a function of the thermal heat transfer coefficient

without crack formation for Al_2O_3 (m) and the layered composite niobium- Al_2O_3 (fm) as a function of the product bh .

The following cases are considered for the thermal transfer coefficient h :

- $h_1 = 0.2$ cal/cm² s °C for immersion in water
- $h_2 = 0.03$ cal/cm² s °C for air moving at high speed
- $h_3 = 0.0002$ cal/cm² s °C for calm air

At a plate thickness of $2b = 1$ cm, the product bh assumes the following values:

- $bh_1 = 0.1$ cal/cm s °C
- $bh_2 = 0.015$ cal/cm s °C
- $bh_3 = 0.0001$ cal/cm s °C

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The thermal stress parameter for fast or slow cooling results from Equations (8) and (9) as:

$$\begin{aligned}R_{1m} &= 59 \text{ }^{\circ}\text{C} \\R_{1fm} &= 79 \text{ }^{\circ}\text{C} \\R_{2m} &= 1,53 \text{ cal/cm } \cdot \\R_{2fm} &= 3,1 \text{ cal/cm } \cdot\end{aligned}$$

The drop in E-modulus and the increase in thermal conductivity are important to the increase in resistance to crack formation arising from the embedding. We proceeded from the assumption that the critical tensile stress for crack formation is determined by the tensile strength of the matrix. At a niobium foil thickness-fraction of 0.4, we calculate an increase in endurable temperature difference upon immersion in water of a 1-cm-thick plate (bh_1) of 57% and for exposure to flowing air (bh_2) of 90%.

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100
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Literature

- 1) Griffith, A.A.: The Phenomena of Rupture in Solids, Phil. Trans. Roy. Soc. A 221 (1920) p. 163-198.
- 2) Claussen, N.; Exner H.E.: Potentials for Reinforcing Ceramic Materials Keram. Z. 21 (1969) no. 12, p. 800-802 and 22 (1970) no. 1, p. 35-38.
- 3) Gruenthaler, K.H.; Harkort, D.; Nixdorf, J.: Potentials for Improving Properties of Glass, Enamel and Ceramic by the Application of the Composite Principle Glas-Email-Keramo-Technik 21 (1970), no. 9, p. 313-324.
- 4) Hasselman, D.P.H.: Elastic Energy of Fracture and Surface Energy as Design Criteria for Thermal Shock. J. Am. Ceram. Soc. 46 (1963) p. 535-540.
- 5) Tetelman, A.S.; McEvily, J.: Fracture of Structural Materials, John Wiley & Sons, New York, 1967, 13 Fracture of Composite Materials, p. 635-661.
- 6) Arias, A.: Thermal Shock Resistance of Zirconia with 15 Mol-% Titanium, J. Am. Ceram. Soc. 49 (1966) p. 334-341.
- 7) Petzow, G.; Claussen, N.; Exner, H.E.: Structure and Properties of Cermet, Z. Metallkunde 59 (1968) no. 3, p. 170-179.
- 8) Janes, S.; Nixdorf, J.; Rochow, H.: Potentials for Improving Shock Resistance and Alternating Temperature Resistance of Brittle Materials, Battelle-Information 5, June 1969, p. 44-46.
- 9) Eissner, G.: Outlook for the Development of Technically Useful Fiber-reinforced High-Temperature Materials, Z. f. Werkstofftechnik 2 (1971) no. 7, p. 337-345.
- 10) Dechema-Werkstofftabellen: Physical Properties, 1963
Landolt-Boernstein: Numerical values and Functions IV 2, 1964
Samsonov. G.V.: The Oxide Handbook, IFI/Plenum, New York, Washington, London, 1973
Schack A.: The Industrial Heat Transfer, Stahleisen mbh Pub., Duesseldorf, 1962
- 11) Buessum, W.R.: Alternating temperature Resistance of Ceramic Masses, Sprechsaal fuer Keramik-Glas-Email 93 (1960), no. 6, p. 137-141.
- 12) Hennicke, H.W.; Kersting, R.: Alternating Temperature Resistance of Ceramic Materials, Handbook of Ceramics, Group IV B 21, p. 1-8, Schmid GmbH Pub., Freiburg, 1970.
- 13) Tattersall, H.G.; Tappin, G.: The Work of Fracture and its Measurement in Metals, Ceramics and other Materials, J. Mater. Sci. 1 (1966) p. 296-301.
- 14) Nakayama, J.: Direct Measurement of Fracture Energies of Brittle Heterogeneous Materials, J. Am. Ceram. Soc. 48 (1965) p. 583-587.

- 15) Eckert, E.R.G.: Introduction to Thermal and Material Exchange, Berlin, Heidelberg, New York: Springer Pub. 1966.