WHISKER-REINFORCED CERAMIC COMPOSITES FOR

HEAT ENGINE COMPONENTS*

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ABSTRACT

Much work has been undertaken to develop techniques of incorporating SiC whiskers into either a Si_3N_4 or SiC matrix. The result has been the fabrication of ceramic composites with ever-increasing fracture toughness and strength. To complement this research effort, the fracture behavior of whisker-reinforced ceramics is studied so as to develop methodologies for the analysis of structural components fabricated from this toughened material. The results, outlined herein, focus on the following areas: the use of micromechanics to predict thermoelastic properties, theoretical aspects of fracture behavior, and reliability analysis.

SCOPE

Current research activities are being focused on upgrading the performance of
heat engines by increasing operating temperatures. With this goal in mind,
whisker reinforced ceramics with improved strength and fracture toughness are
being investigated for use as structural components in engine hot sections.
Even though the fracture toughness of these composites is improved relative to
unreinforced ceramics, they remain brittle in nature. Work has been initiated
to identify and develop probabilistic methods required in the analysis of
whisker-reinforced ceramic components. In addition, approaches using micro-
mechanics to predict thermoelastic properties are under review.

- OBJECTIVES

- TOUGHENING MECHANISMS

- CRACK GROWTH MITIGATION PROCESSES IN WHISKER-REINFORCED CERAMICS

- IMPROVEMENTS IN STRENGTH AND TOUGHNESS

- MICROMECHANICS AND WHISKER ORIENTATION

- ELASTIC MATERIAL PROPERTIES

- WEIBULL DATA

- RELIABILITY ANALYSIS

- SUMMARY

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OBJECTIVE

The objective of this effort includes the identification of whisker reinforced ceramics for use in engine hot sections. Further, the technical thrust is directed towards developing and/or refining analytical methods and computer codes that adequately predict fast fracture and life. The effort complements concurrent research by other organizational groups within NASA, the federal government, academia, and industry.
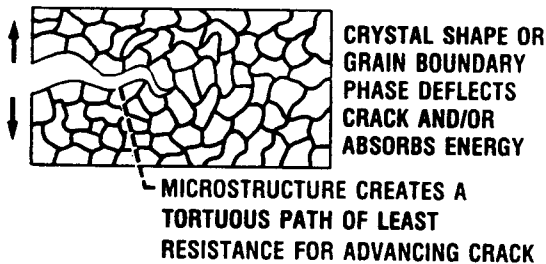
- IDENTIFY WHISKER REINFORCED CERAMICS FOR USE IN ENGINE HOT SECTIONS

- DEVELOP AND REFINE ANALYTICAL METHODS AND COMPUTER CODES FOR PREDICTING
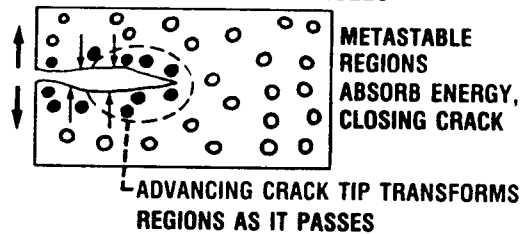   - FAST FRACTURE
    - LIFE

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TYPICAL TOUGHENING MECHANISMS FOR CERAMICS

Historically, three approaches have been taken to improve fracture toughness. One approach is to engineer the grain size and shape to provide a tortuous microstructure such that the path of the crack tip is deflected from the optimum orientation for crack growth. The second approach creates microstructures containing second-phase particles resulting in transformation toughening. Here, a zone surrounding the crack tip absorbs energy and shields the tip by reducing the near field stress. The third approach, including whiskers in the matrix, increases toughness by pinning, deflecting, and/or bridging the crack tip.
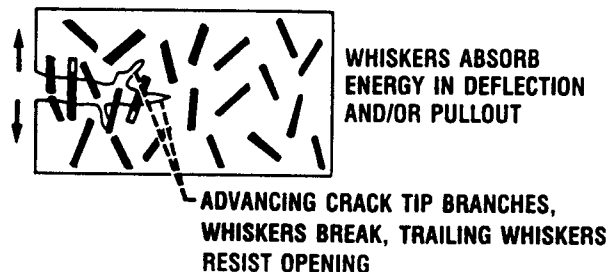
● ENGINEERING GRAIN SIZE AND SHAPE

CRYSTAL SHAPE OR
GRAIN BOUNDARY
PHASE DEFLECTS
CRACK AND/OR
ABSORBS ENERGY

└ MICROSTRUCTURE CREATES A
TORTUOUS PATH OF LEAST
RESISTANCE FOR ADVANCING CRACK

● CREATING MICROSTRUCTURES WITH
SECOND-PHASE PARTICLES

METASTABLE
REGIONS
ABSORB ENERGY,
CLOSING CRACK

└ ADVANCING CRACK TIP TRANSFORMS
REGIONS AS IT PASSES

● ADDING HIGH-STRENGTH WHISKERS

WHISKERS ABSORB
ENERGY IN DEFLECTION
AND/OR PULLOUT

└ ADVANCING CRACK TIP BRANCHES,
WHISKERS BREAK, TRAILING WHISKERS
RESIST OPENING

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CRACK GROWTH MITIGATION PROCESSES - WHISKER REINFORCED CERAMICS

It has been demonstrated experimentally that dispersing whiskers in a brittle matrix will mitigate crack growth. The presence of whiskers at the crack tip modifies fracture behavior by effectively increasing the required crack driving force through several mechanisms. As noted previously, these mechanisms include crack deflection, crack pinning, and whisker bridging. Faber and Evans (1983) have studied crack deflection and provide a lucid discussion. Lange (1971) discussed the process of crack pinning, and Wetherhold (1987) provides a probabilistic treatment of the crack bridging phenomenon.

THREE PROCESSES INCREASE FRACTURE TOUGHNESS

- CRACK DEFLECTION
  - TILTING
  - TWISTING
- CRACK PINNING
- WHISKER BRIDGING

SEVERAL PROCESSES MAY OPERATE SIMULTANEOUSLY

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IMPROVEMENTS IN STRENGTH AND TOUGHNESS

The addition of SiC whiskers in a Si_3N_4 matrix offers potential for considerable improvement in fracture toughness and strength. Note that the raw materials necessary for fabricating these composites are nonstrategic and are inherently lightweight. Initial attempts to develop whisker composites with these materials met with varying degrees of success (e.g., increased fracture toughness and decreased strength). Recently, Buljan et al. (1987) at GTE Laboratories, Inc., reported improvements in both toughness and strength over the entire range of whisker contents tested.
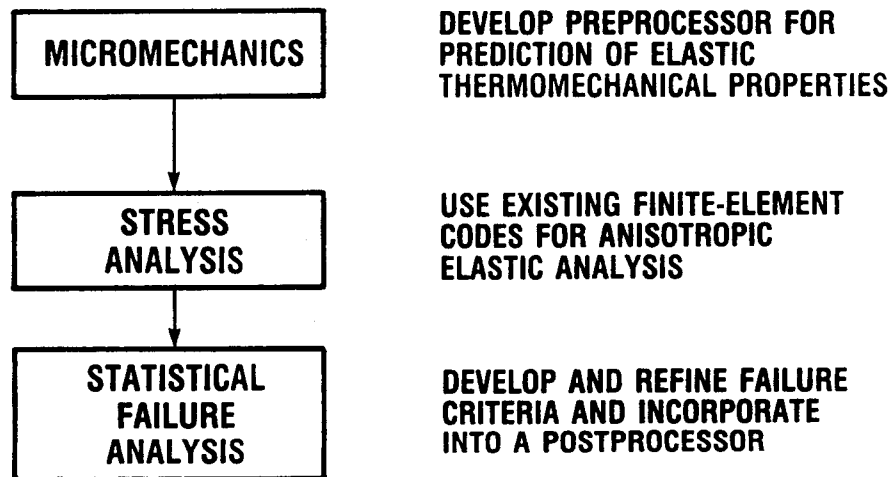
HOT PRESSED Si₃N₄—SiC WHISKER COMPOSITES

FRACTURE TOUGHNESS, K_{IC}, MPa•m½

MODULUS OF RUPTURE MPa•m½

SiC CONTENT, (vol %)

1000 °C   1200 °C
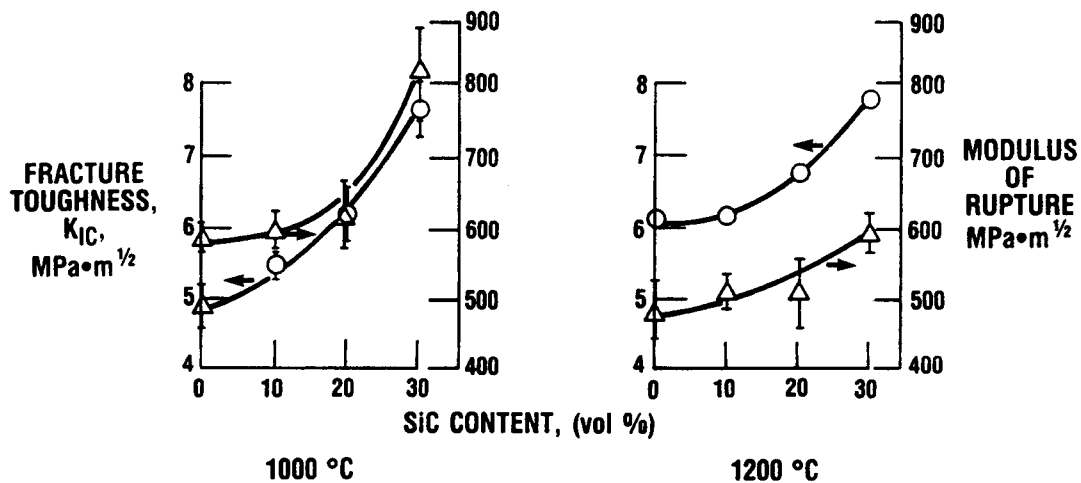
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ANALYTICAL APPROACH

The analytical approach taken involves the development of an integrated computer algorithm. The algorithm consists of (1) a micromechanics preprocessor, (2) a fintie element code capable of incorporating material anisotropy, and (3) a statistical failure postprocessor. Proposed use of MSC/Nastran as the finite element code is based on its anisotropic analysis capability and widespread user base. The preprocessors and postprocessors are currently under development.

| MICROMECHANICS | DEVELOP PREPROCESSOR FOR PREDICTION OF ELASTIC THERMOMECHANICAL PROPERTIES |

↓

| STRESS ANALYSIS | USE EXISTING FINITE-ELEMENT CODES FOR ANISOTROPIC ELASTIC ANALYSIS |

↓

| STATISTICAL FAILURE ANALYSIS | DEVELOP AND REFINE FAILURE CRITERIA AND INCORPORATE INTO A POSTPROCESSOR |

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MICROMECHANICS AND WHISKER ORIENTATION

The thermoelastic properties of ceramic whisker composites are determined by process-induced whisker orientation and the constituent properties. Possible material symmetries resulting from fabrication include isotropy, transverse isotropy, and orthotropy. To characterize the internal structure, a whisker orientation distribution function is adopted. This approach was suggested by Pipes et al. (1982). The function quantifies all states of orientation, from random to perfectly aligned.

MATERIAL SYMMETRIES

- **ISOTROPIC**
- **TRANSVERSELY ISOTROPIC**
- **ORTHOTROPIC**

DEFINE WHISKER ORIENTATION PROBABILITY FUNCTION $n(\phi)$ SUBJECT TO

$$\int_{-\pi}^{\pi} n(\phi) \, d\phi = 1$$

WHEN

$n(\phi) = $ CONSTANT   **RANDOM WHISKER ORIENTATION**

$n(\phi) = \delta(\eta - \phi)$ **ALIGNED WHISKER ORIENTATION**

HERE δ IS THE DIRAC DELTA FUNCTION AND η IS THE PRINCIPAL MATERIAL DIRECTION

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The micromechanics theory proposed by Wu and McCullough (1977) for short fiber polymer composites is being considered for adaptation to whisker-reinforced ceramics. The theory was developed using variational techniques and incorporates the degree of whisker orientation, constituent properties, and volume fractions. The method requires the properties of a reference orientation, usually taken as the random orientation state. The parameters f and g define the orientation distribution and are included in the calculation of volume averaged properties.

EXPRESSING THE EFFECTIVE ELASTIC MATERIAL CONSTANTS AS

$$\langle C_{ijkl} \rangle = C_{ijkl}^{\circ} + F(C_{ijkl}^{\circ}, \overline{C}_{ijkl}) \, f + G(C_{ijkl}^{\circ}, \overline{C}_{ijkl}) \, g$$

WHERE

$\langle C_{ijkl} \rangle$ PREDICTED ELASTIC CONSTANTS OF THE COMPOSITE

C_{ijkl}° ELASTIC CONSTANTS FOR RANDOM WHISKER ORIENTATION

$\overline{C}_{ijkl}$ LINEAR COMBINATION OF THE CONSTITUENT ELASTIC CONSTANTS

THE PARAMETERS f AND g ARE FUNCTIONALLY DEPENDENT UPON $n(\phi)$ AND DESCRIBE THE ORIENTATION STATE. WHEN

$f = g = 0$ RANDOM WHISKER ORIENTATION

$f = g = 1$ ALIGNED WHISKER ORIENTATION

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The planar orientation of whiskers is often encountered in hot pressed composites. This special case was considered by Pipes et al. (1982). The orientation descriptors f_p and g_p along with the distribution function are defined below. Bozarth, et al. (1987) developed a Monte Carlo simulation that depicts the whisker orientation for specified values f_p.

APPLYING THE CONCEPT TO A PLANAR ORIENTATION OF WHISKERS

$$n(\phi) = K \cos (\lambda \phi)$$

$$f_p = 2 \langle \cos^2 \phi \rangle - 1$$

$$g_p = \frac{2f_p(7 - 2f_p)}{5(4 - 2f_p)}$$

WHERE

$$\langle \cos^2 \phi \rangle = \int_{-\pi/2}^{\pi/2} n(\phi) \cos^2 \phi \, d\phi$$

THEN

$f_p = 0, 1$ **TRANSVERSE ISOTROPY**

$0 < f_p < 1$ **ORTHOTROPY**

A MONTE CARLO SIMULATION ILLUSTRATES GRAPHICALLY VARIOUS f_p VALUES

$f_p = 0.0$ $f_p = 0.3$ $f_p = 0.6$ $f_p = 0.9$

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INFLUENCE OF WHISKER ORIENTATION ON MATERIAL PROPERTIES

The influence of orientation on material properties is show below (from Pipes et al., 1982). Both the elastic modulii and the coefficients of thermal expansion are plotted for a short glass fiber phenolic resin matrix composite. The constants are plotted over the full range of f_p. Note that for $f_p = 1$ and $f_p = 0$, the material is transversely isotropic. For all other values, the material is orthotropic.

PREDICTIONS USING THE APPROACH FOR A SHORT FIBER POLYMER COMPOSITE

### FIBER ASPECT RATIO ≃ 40-100; FIBER CONTENT BY WEIGHT = 58%
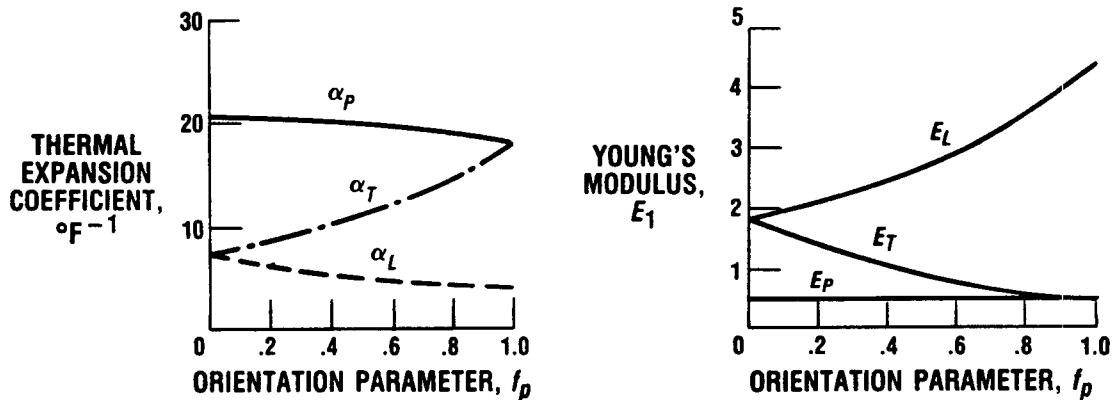
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The examples shown below of stress-strain curves corresponding to various whisker contents were reported by Shalek et al. (1986). Note that the curves exhibit a linear stress-strain response up to the point of fracture. This indicates that crack growth involves only brittle fracture and allows for the application of linear elastic fracture mechanics (LEFM).

SiC WISKER-HOT-PRESSED Si₃N₄

**FOUR-POINT BEND STRESS-STRAIN CURVES**

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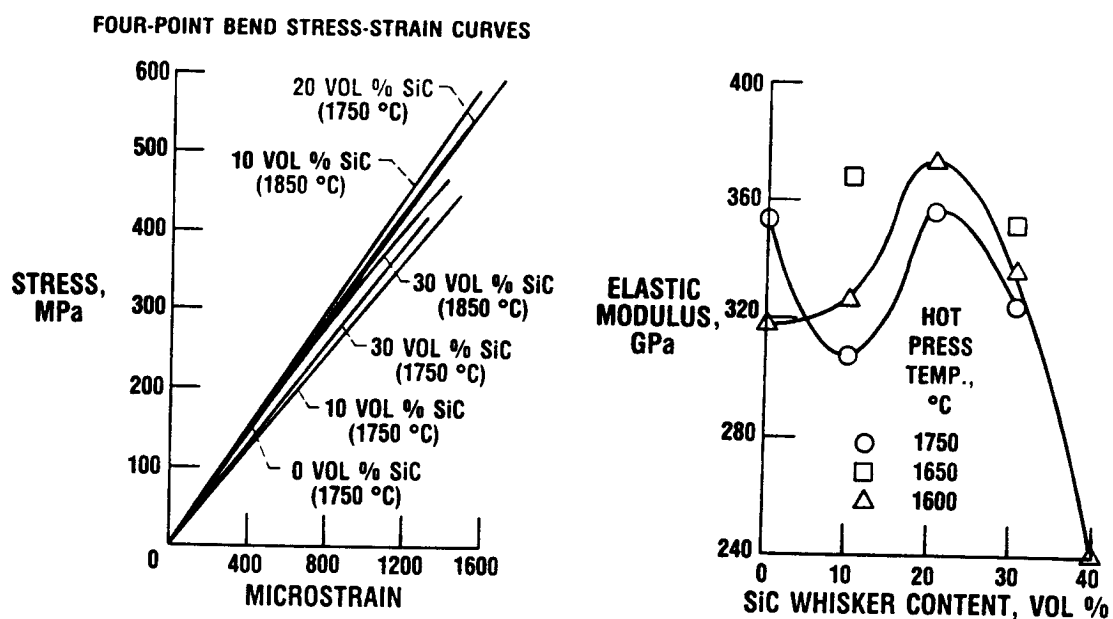
Greatly improved reliability has been cited by a number of authors, including Claussen and Petzow (1985), who have reported the highest Weibull modulus (m = 24) in the open literature. Improved processing techniques have resulted in the reduction of inhomogeneities, uniform whisker distribution, and a dense matrix. However, the variability of strength is still too high for the application of deterministic fracture theories.

30% SiC-WHISKER-Si_3N_4-MATRIX COMPOSITE

WHISKER REINFORCED CERAMICS EXHIBIT A VARIABILITY IN STRENGTH; HENCE, PROBABILISTIC METHODS OF ANALYSIS MUST BE APPLIED

PROBABILITY OF FAILURE

m = 24

FRACTURE STRENGTH, MPa
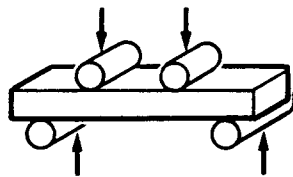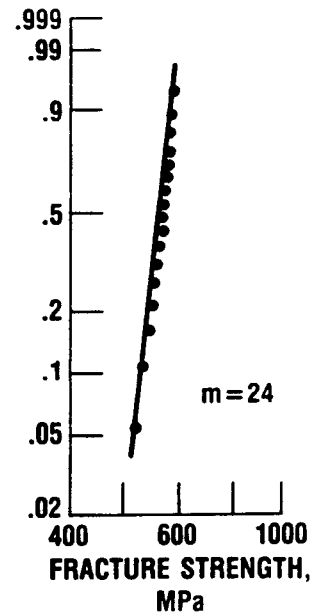
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As noted previously, depending on fabrication, a whisker composite may have isotropic, transversely isotopic, or orthotropic material symmetry. It is anticipated that the probabilistic methods used in the analysis of monolithic ceramics will be appropriate for isotropic whisker composites. However, for transversely isotropic whisker composites, the probability of failure P_f must also reflect the preferred direction of the material d_i. This direction is defined as the normal to the plane of isotropy. A simple but rigorous method of including material symmetry in the functional dependence of P_f for transverse isotropy is presented. A similar approach can be developed for orthotropy.

TRANSVERSELY ISOTROPIC WHISKER COMPOSITES

HOT PRESSED   INJECTION MOLDED
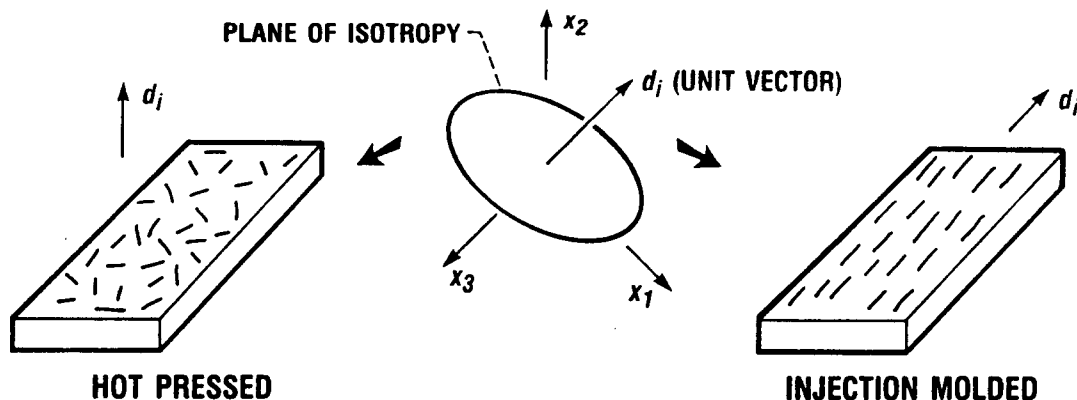
DEPENDENCE OF P_f MUST REFLECT σ_{ij}, d_i, VOLUME AS WELL AS THE WEIBULL PARAMETERS

$$P_f = P_f\ (\sigma_{ij},\ d_i d_j,\ V,\ ....)$$

ASSUMING WEAKEST LINK THEORY IS APPROPRIATE, FORMULATE P_f IN A WAY THAT ACCOUNTS FOR THE ABOVE DEPENDENCE

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As P_f is a scalar function, it must remain form invariant under arbitrary proper orthogonal transformations. Form invariance is ensured if dependence is taken on invariants that constitute an integrity basis or any subset thereof. Tensorial invariant theory (see Spencer (1971)) serves as the basic mathematical tool in the development of the integrity basis. A subsequent geometric argument is made in constructing a slightly different set of invariants that corresponds to physical mechanisms related to fracture.

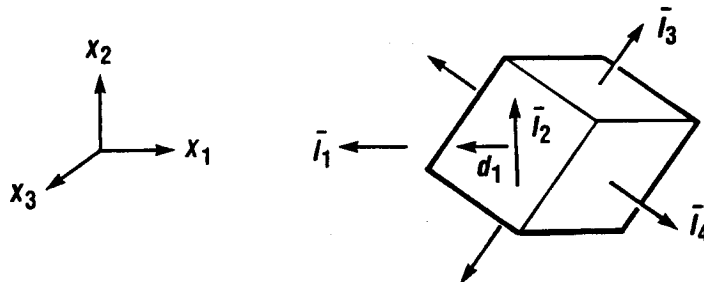**ADOPT THE FOLLOWING INTEGRITY BASIS:**

$$I_1 = \sigma_{ii} \qquad\qquad I_4 = d_i\, d_j\, \sigma_{ij}$$

$$I_2 = \sigma_{ij}\, \sigma_{ji} \qquad\qquad I_5 = d_i\, \sigma_{ij}\, \sigma_{jk}\, d_k$$

$$I_3 = \sigma_{ij}\, \sigma_{jk}\, \sigma_{ki}$$

IDENTIFY DAMAGING STRESS TRACTION VECTORS:

$\bar{I}_3,\ \bar{I}_4$ **MAXIMUM AND MINIMUM NORMAL STRESSES IN PLANE OF ISOTROPY**

$\bar{I}_1$ **NORMAL STRESS IN DIRECTION OF d_i**

$\bar{I}_2$ **SHEAR STRESS ACTING ACROSS d_i**

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The dependence of P_f is subsequently taken on the invariants $\bar{I}_1$, $\bar{I}_2$, $\bar{I}_3$, and $\bar{I}_4$. This new set of invariants corresponds to the magnitudes of the stress traction vectors assumed to be the primary causes of fracture. These invariants incorporate both the stress tensor σ_{ij} and the direction vector d_i. Adopting the simplest of probabilistic failure theories, that is, a noninteractive theory, results in the form of P_f shown in the figure.

FROM THE INTEGRITY BASIS CONSTRUCT INVARIANTS WITH PHYSICAL INTERPRETATIONS CORRESPONDING TO THE MAGNITUDES OF THE DAMAGING STRESS TRACTION VECTORS

$\bar{I}_1 = I_4$

= COMPONENT OF S_i PROJECTED ON DIRECTION d_i,

$\bar{I}_2 = (I_5 - I_4{}^2)^{1/2}$

= COMPONENT OF S_i PROJECTED ON THE PLANE OF ISOTROPY

$\bar{I}_3 = \frac{1}{2}(I_1 - I_4) + [(\frac{1}{2})I_2 - I_5 + \frac{1}{4}(I_4{}^2 - I_1{}^2) + (\frac{1}{2})I_1 I_4]^{1/2}$

= MAXIMUM NORMAL STRESS IN PLANE OF ISOTROPY

$S_i = \sigma_{ij} d_i$

$\bar{I}_4 = \frac{1}{2}(I_1 - I_4) - [\frac{1}{2}I_2 - I_5 + \frac{1}{4}(I_4^2 - I_1^2) + \frac{1}{2}I_1 I_4]^{1/2}$

= MAXIMUM NORMAL STRESS IN PLANE OF ISOTROPY

TAKING

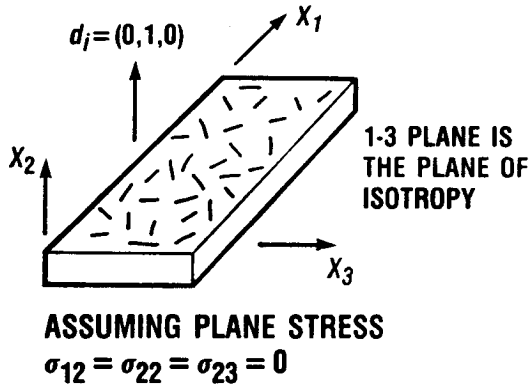$$P_f = P_f(\bar{I}_1, \bar{I}_2, \bar{I}_3, \bar{I}_4, V, ...)$$

ENSURES P_f IS FORM INVARIANT. ASSUMING THE INVARIANTS ACT SEPARATELY IN PRODUCING FAILURE

$$P_f = 1 - \exp\left\{-\int_V\left[\left(\frac{\bar{I}_1}{\beta_1}\right)^{\alpha_1} + \left(\frac{\bar{I}_2}{\beta_2}\right)^{\alpha_2} + \left(\frac{\bar{I}_3}{\beta_3}\right)^{\alpha_3} + \left(\frac{\bar{I}_4}{\beta_3}\right)^{\alpha_3}\right]dV\right\}$$

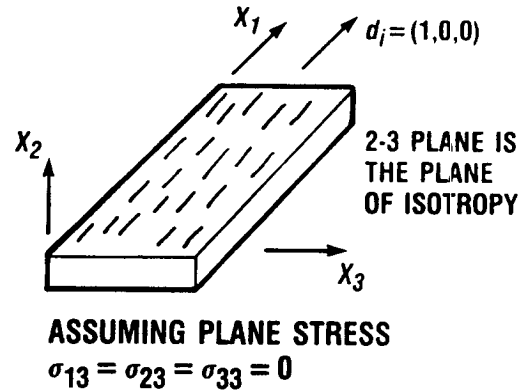CD-88-32526

PLANE STRESS PROBABILISTIC ANALYSIS

The form of P_f is simplified for plane stress conditions considering two planar orientations of the whiskers. In both cases it is assumed that the whiskers are confined to the 1-3 plane due to fabrication. A random orientation of whiskers in the 1-3 plane reduces P_f to the isotropic formulation. Alternatively, perfect alignment of the whiskers in the 1-3 plane reduces P_f to a formulation proposed by Sun and Yamada (1978), Wetherhold and Pipes (1984), and Cassenti (1984).

$d_i = (0,1,0)$ X_1

X_2 X_3

1-3 PLANE IS THE PLANE OF ISOTROPY

ASSUMING PLANE STRESS

$\sigma_{12} = \sigma_{22} = \sigma_{23} = 0$

THEN

$$P_f = 1 - \exp\left\{ -\int_V \left[\left(\frac{\sigma_1}{\beta_3}\right)^{\alpha_3} + \left(\frac{\sigma_2}{\beta_3}\right)^{\alpha_3} \right] dV \right\}$$

WHERE σ_1 AND σ_2 ARE THE PRINCIPLE STRESSES IN THE PLANE OF ISOTROPY

X_1 $d_i = (1,0,0)$

X_2 X_3

2-3 PLANE IS THE PLANE OF ISOTROPY

ASSUMING PLANE STRESS

$\sigma_{13} = \sigma_{23} = \sigma_{33} = 0$

THEN

$$P_f = 1 - \exp\left\{ -\int_V \left[\left(\frac{\sigma_{11}}{\beta_1}\right)^{\alpha_1} + \left(\frac{\sigma_{12}}{\beta_2}\right)^{\alpha_2} + \left(\frac{\sigma_{22}}{\beta_3}\right)^{\alpha_3} \right] dV \right\}$$

Attempts have been made to develop statistical models that account for the microstructural events leading to crack propagation. Wetherhold (1987) derived a model assuming that fracture behavior is dominated by whiskers bridging a critical damage zone. The damage zone is analogous to a microcrack which is expected to grow and coalesce with other microcracks during progressive fracture. A distribution function for composite strength is developed based on the incorporation of random whisker strength into a bundle fracture theory.

THE FOLLOWING STATISTICAL APPROACH ACCOUNTS FOR THE WHISKER BRIDGING MECHANISM

SPECIMEN OF UNIT VOLUME
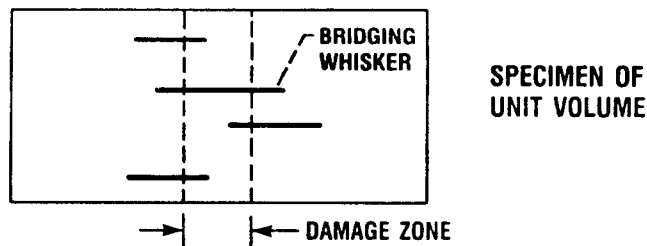
DEFINE

[A] = THE EVENT WHERE ULTIMATE COMPOSITE STRENGTH IS GREATER THAN THE APPLIED LOAD

THEN

$$P_S = P[A]$$

$$= \sum_{i=0}^{N} P[A|n=i]\, P[n=i]$$

WHERE

n **NUMBER OF WHISKERS BRIDGING THE DAMAGE ZONE**
N **NUMBER OF FIBERS PER UNIT VOLUME**

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SUMMARY

Enough experimental data exist that strongly suggests whisker-reinforced ceramics have promise, especially in the automotive industry where low-cost, high-volume fabrication is a necessity. The Structural Integrity Branch is identifying and refining existing design methodologies and, where necessary, assisting the research community in developing new methodologies. At present, there exists a need for analytical methods that capture the microstructural events that lead to increased fracture toughness and strength. Finally, work has begun on an integrated computer program capable of predicting elastic material properties through the application of micromechanics, the state of stress within a structural component, and the reliability of the component given the state of stress.

- EXPERIMENTAL DATA SHOW SIGNIFICANT INCREASES OF STRENGTH AND FRACTURE TOUGHNESS DUE TO THE ADDITION OF CERAMIC WHISKERS IN A CERAMIC MATRIX

- WHISKER COMPOSITES HAVE THE ATTRACTIVE FEATURE OF USING CONVENTIONAL POWDER PROCESSING TECHNIQUES IN HIGH-VOLUME, LOW-COST FABRICATION

- AN INTEGRATED STRUCTURAL ANALYSIS CODE IS BEING DEVELOPED WITH THE FOLLOWING COMPONENTS:
  - MICROMECHANICS PREPROCESSOR
  - FINITE-ELEMENT PROGRAM
  - STATISTICAL FAILURE ANALYSIS POSTPROCESSOR

- RESEARCH IS UNDERWAY TO DEVELOP AND REFINE STATISTICAL FAILURE THEORIES TO ACCOUNT FOR MATERIAL ANISOTROPY ALONG WITH THE MICROSTRUCTURAL EVENTS LEADING TO FAILURE (I.E., CRACK DEFLECTION AND WHISKER BRIDGING)

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