

**ANALYTICAL AND EXPERIMENTAL EVALUATION OF JOINING
CERAMIC OXIDES TO CERAMIC OXIDES AND
CERAMIC OXIDES TO METAL FOR
ADVANCED HEAT ENGINE APPLICATIONS**

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LIST OF SYMBOLS

α	coefficient of linear thermal expansion
β	constant appearing in Equation (25)
Γ	contour of integration
γ	constant appearing in Equation (25)
δ_{ij}	Kronecker delta
ΔT	difference between joining temperature and test temperature
ϵ	Bimaterial constant
ϵ	strain
η	logarithmic term in stress field
Θ	angle measured from disk-bond line to compressive loading direction
λ	measure of thermal stress singularity at the specimen edge
μ	shear modulus
ν	Poisson's ratio
σ	stress
σ_0	yield strength
σ_c	critical stress value
σ_R	uniform compressive residual stress
σ_R	residual stress
σ_t	nominal residual stress
σ_x	stress component in the x-direction
Φ	angle that assessment vector makes with the strength axis
χ	distance between center of interlayer and crack location
Ψ	phase angle measuring degree of mode I and mode II fracture toughness
	mixity
a	one half the total crack length
\bar{a}	dimensionless crack length, a/W
A	assessment vector magnitude
\bar{A}	assessment vector
B	specimen thickness
C	parameter in failure criterion on page 39
c	denotes ceramic when used as subscript
C	geometry dependent factor in asymptotic residual stress solution
D	coefficient in Ramberg-Osgood relation
E	Young's modulus
F_R	rational function of a/W used in calculating the residual stress load
	parameter P_R
f_s	shielding factor due to mismatches in Young's modulus
G	crack driving force
G	total (elastic-plastic) energy release rate
G_c	critical crack driving force
G_e	elastic crack tip energy release rate
G_r	dimensionless crack driving force parameter
i	denotes interlayer when used as subscript

LIST OF SYMBOLS (cont.)

K_I	mode I (opening) fracture toughness for monolithic system
K_{Ic}	critical value of fracture toughness, a material property
K_{II}	mode II (shear) fracture toughness for monolithic system
L	distance between outer loading points in four point bend tests
m	denotes metal when used as subscript
M	bending moment
n_j	outward normal to contour of integration Γ
P	total load on the joint
P_c	value of P at which joint failure occurs
P_{pr}	predicted failure load
P_R	residual stress load parameter
P_r	dimensionless strength parameter
q	distance at which σ_x passes through zero
Q_I	mode I (opening) fracture toughness for bimaterial system
Q_{II}	mode II (shear) fracture toughness for bimaterial system
R	total tensile residual stress acting over the length q
R	radius of disk specimens
r	distance from edge of specimen
S	distance between inner loading points in four point bend tests
T	temperature
t_i	interlayer thickness
u_i	displacement component in direction i
V	volume percent zirconia in MASZ compositions
W	stress work density
W	bend bar depth
Y	a function used in reduction of experimental fracture toughness data

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ABSTRACT

The problem of designing reliable, high strength zirconia-to-zirconia and zirconia-to-nodular cast iron joints is addressed by developing a general joint design and assessment methodology. A joint's load carrying capability is predicted in terms of its material strength and fracture toughness characteristics. The effects of joint constituent properties and joining process variables are included. The methodology is verified in a two step process by applying it first to notched bend bars and then to a notched disk specimen loaded in compression.

Key technical accomplishments in the program include the development of a joint design and assessment methodology which predicts failure based on a combination of strength and toughness, the development of a new method of hot forging magnesia partially stabilized zirconia to itself, and the development of a bimaterial disk-shaped specimen notched along the diametral bond line and compressively loaded to generate both shear and tensile loadings on the bond line.

Mechanical and thermal characterization of joints, adherents, and interlayer materials were performed to provide data for input to the design methodology. Results from over 150 room temperature tests and 30 high temperature tests are reported. Extensive comparisons of experimental results are made with model predictions of failure load.

The joint design and assessment model, as applied to the materials and test specimens of this program, has been programmed for a PC and is available to interested researchers.

EXECUTIVE SUMMARY

The research discussed in this report was funded by the U.S. Department of Energy under Subcontract No. 86X-SB046C to Martin Marietta Energy Systems. The program was one of three similar contracts dealing with developing a validated design methodology for improving the strength of joints made by joining tough ceramic oxides to either ceramic

oxides or to metals. This project deals with the development of procedures necessary for the design of reliable, high strength zirconia-to-zirconia and zirconia-to-nodular cast iron joints. The objectives were met by implementing a combined program of analysis and experimentation.

Figure 1 is a schematic of the program showing the relationships between the various tasks and milestones in the joint design methodology. It involved finite element analyses of the joint configurations based on joint geometry, properties of constituent materials (adherents and joint interlayers) and the joining temperature; reduction of the finite element analysis results to a simplified engineering joint assessment and design model; experimental evaluation of the failure load of notched and unnotched bend bars containing joints; using the preliminary validation results to fine tune the joint assessment and design model by incorporating factors which were previously not accounted for; and finally, testing cracked disk specimens and comparing measured and predicted fracture loads as part of the final validation of the methodology.

The joint assessment methodology, as it is currently formulated, can be used in two ways. First, it can be used in the machine design of components containing joints, where the processing conditions and interlayer constituents are already prescribed. Second, the methodology can be used for improving joint performance. In this latter mode, processing temperatures and constituent properties can be altered within limits and the model will provide a quantitative assessment of how those changes affect the load bearing capability of the joint relative to the load bearing capability of the monolithic ceramic of the same geometry.

ANALYTICAL DESIGN METHODOLOGY

An analytically based joint design methodology has been developed. The methodology has been applied to the particular materials and geometries studied in this program and the results incorporated into a PC code.

The essence of the methodology is that for a given specimen geometry and the manner in which it is loaded, a joint's load carrying capacity is determined by its strength and toughness properties. Which of the two properties dictates how and at what load failure occurs depends on their relative magnitude and residual stresses in the joint. Joint constituent properties influence the load carrying capacity of a joint through their inherent strength and toughness. Joining process variables influence the load bearing capacity through their effect on residual stresses, local stress-strain behavior and their effects on interlayer and joint constituent properties.

A key difference in this approach and other available formulations is the way failure depends on a combination of toughness and strength. The current approach represents a synergism of applied stress, crack length, toughness, residual stress, and strength in a self-consistent manner.

Stress analysis and fracture mechanics based models were developed for determining the stress field, σ , crack driving force, G , and residual stresses σ_R , for the test specimen geometries being tested in this program. These computations used linear and nonlinear finite element analyses which accounted for the physical properties of the joint constituents as well as the joining temperature and interlayer thickness. Other processing variables were

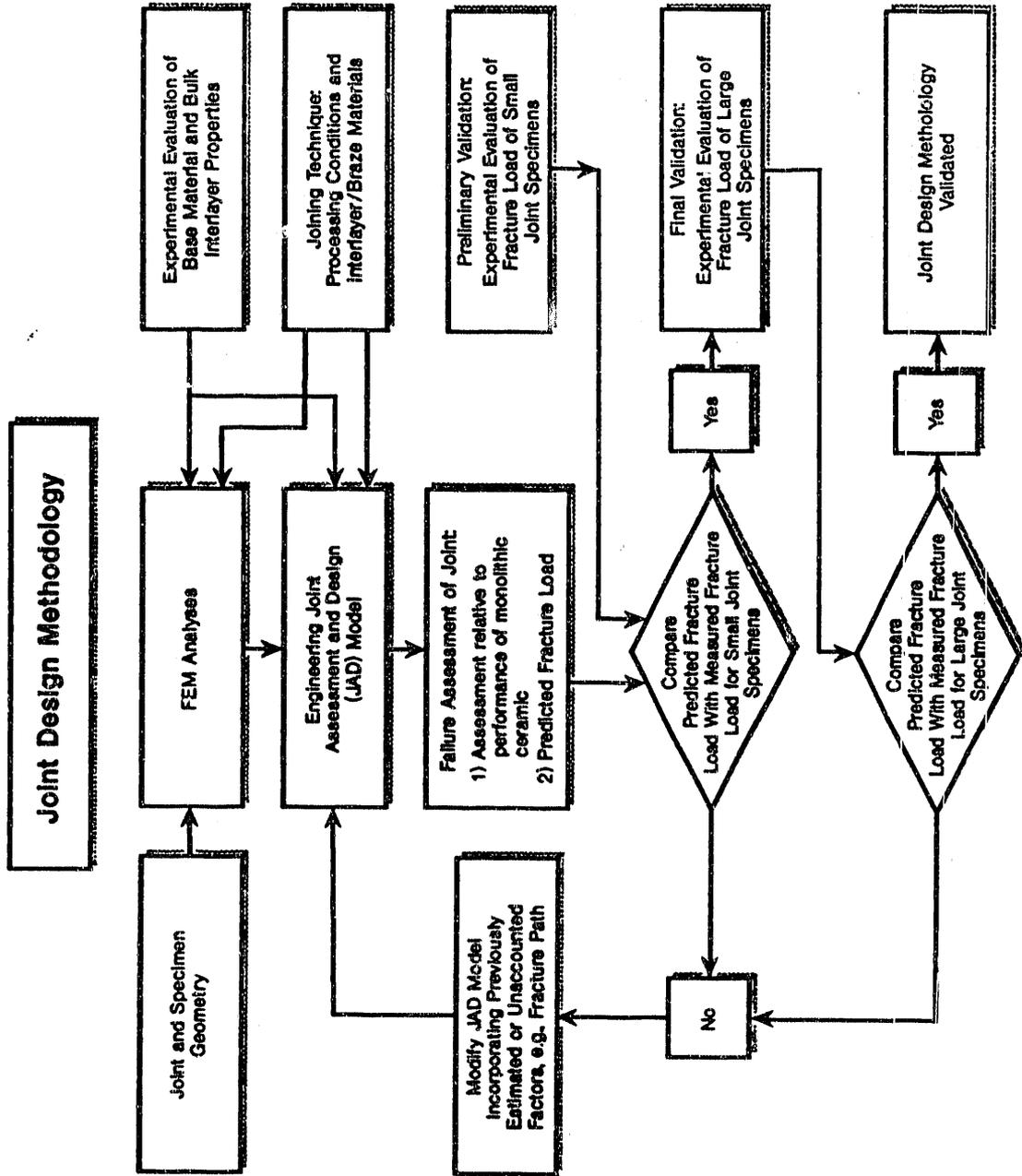


Figure 1. Schematic of joint design methodology development.

handled empirically. The results of the detailed analyses were used to develop simpler engineering models for estimating the factors which influence joint performance.

A joint assessment and design model (JAD model) which unifies the three primary factors (σ , G and σ_R) embodies the simplified analysis. The simplicity resides in the fact that the engineering model is fashioned after a homogeneous material. The complexity of the joint constituents and process conditions are handled by incorporating algebraic factors which account for thermal expansion mismatch effects (residual stresses) and shielding effects (arising from elastic modulus mismatch). A joint assessment vector \bar{A} with components depending on strength, crack driving force, residual stress, and load was derived. It has the property that the smaller its magnitude, the less the propensity for failure of the joint. Another aspect of the design model is a failure curve which depends on the joint type and geometry. The conditions for which the vector extends to the failure curve for a given joint type and geometry define failure.

The area enclosed by the failure curve (or a fraction thereof) represents a design space for the joint. So long as \bar{A} is within this space the joint is not predicted to fail under the prescribed load. For failure prediction, both the assessment vector and the failure curve are needed. A different application in which joint improvement is sought only requires the assessment vector. Here, at some specified reference or design load one would try to minimize the length of \bar{A} with respect to suitably constrained values of the constituent properties and process variables.

JOINT FABRICATION

To validate the methodology and provide input data to the analyses, joints were fabricated by bonding blocks of ceramic and iron materials. Specimens were machined from the resulting billets. All bonds were geometrically simple butt joints to facilitate both analysis and experiment. While smaller joints (typically 1.0-1.6 cm²) were produced for initial screening, the strength and fracture test specimens were cut from large (≥ 20 cm²) bonded areas.

Since bonded joints are likely to contain measurable flaws, it is essential to measure both strength and fracture toughness, either of which property can control the load-carrying capacity of a joint. Furthermore, because joints composed of dissimilar materials have an inherent mixed mode nature arising from elastic property mismatch between constituents, it is important to assess any design and predictive methodology by imposing various combinations of tensile and shear loads on the joints.

Two geometries were used for mechanical testing: bend bars and disks. The bend bar is the most common specimen for measuring ceramic strength in tension. It is also well established as a fracture-toughness specimen. The disk specimen, developed as part of this research, consists of two D-shaped pieces bonded together and loaded in compression. By varying the angle between the bond line and the loading line, desired proportions of tensile and shear stresses were exerted on the bond line. This design was used for both strength and toughness measurements.

MATERIALS SELECTION

Zirconia. Specification of zirconia involved first deciding whether to use one grade for both ceramic/ceramic and ceramic/metal bonds or to tailor the grade to the bond. The choice of a single grade for both bonds provided for the most efficient use of resources in that more effort was devoted to modeling and less to routine material characterization. The magnesia-stabilized grade (Zircoa Zycron L Mg-PSZ) was chosen over a yttria-stabilized grade because it is a more well-established material, a fully-dense ceramic can be more readily produced, and it is easier to machine. It also has good strength and fracture toughness as well as an acceptably low coefficient-of-thermal-expansion mismatch with cast iron.

Nodular Cast Iron. The nodular cast iron (NCI) chosen for this program had a ferritic microstructure, corresponding both to SAE D4018 and to ASTM A536, Grade 60-40-18. The main reason for this choice was that it was the most metallurgically stable grade. It may be noted that significant microstructural changes can occur when NCI is heated to around 760 C, which is only slightly above the temperature range in which brazing is normally carried out. As a result, the mechanical properties of the NCI after brazing can be significantly different from those of the as-received iron and can vary from bond-to-bond, masking the effects of bonding variables and complicating the application of the design model. The major drawback to the chosen grade was that it is weaker than the grade used previously in the Oak Ridge National Laboratories (ORNL) bonding studies (D5506) and much weaker than the one currently being used there (8003).

Zirconia/Zirconia Joint Fabrication. Zirconia/zirconia joints were fabricated using a ceramic interlayer that became molten at the joining temperature. Metallic brazes were rejected due to their limited high-temperature capabilities since the goal was to obtain acceptable strength and toughness of joints at temperatures up to 1000 C. Initially, two ternary eutectic oxides (calcia-titania-silica and calcia-alumina-silica) were evaluated because they become liquid at a bonding temperature of 1420 C where no phase changes were expected in the base zirconia. These eutectic materials proved to be too weak at room temperature, despite good wetting and minimal influence on residual stresses. The eventual choice of interlayer material was magnesia-alumina-silica-zirconia (MASZ) which had acceptable strength and toughness. In order to allow validation of the joint design and assessment methodology over a range of process variables, the composition of the MASZ interlayers was altered by changing the zirconia content.

In addition to fabricating joints with MASZ interlayers, bulk interlayer blocks were fabricated by sintering, and these allowed the determination of thermal expansion coefficients, deformation characteristics, and inherent strength and toughness of bulk MASZ materials.

Zirconia/NCI Joint Fabrication. Brazing of zirconia to NCI was investigated using both the active-substrate and active-filler-metal methods. Attempts to join these materials using the active-substrate brazing method were generally unsuccessful, except when the joint area was small. However, large-area strong bonds were obtained using Incusil-ABA, an active-filler-metal braze (Ag-27.5Cu-12In-1.2Ti), in which the relative amounts of silver, copper, and titanium were nearly the same as produced in the active-substrate brazing

method. In contrast to the active substrate process, no surface coatings were applied to either the zirconia or the NCI in the active filler metal process and no significant voids were observed in the resulting joint.

Joint Scale-up

Although initial fabrication using small joints (2 sq. cm.) with a goal of scaling up to 20 sq. cm. was envisioned, research during the course of the project showed that the larger joints could be fabricated without any additional difficulty. For this reason, the size objective for the joints was achieved.

CHARACTERIZATION AND ANALYSIS OF JOINTS AND MONOLITHIC MATERIALS

Mechanical and thermal characterization of joints, adherents, and interlayer materials were performed. Specifically, stress-versus-strain curves were measured for the zirconia, MASZ interlayer materials, Incusil-ABA, and the cast iron. In the case of the zirconia, stress-strain behavior for both as received material and material heat treated at 1350 C was measured. Thermal expansion curves were obtained over appropriate temperature ranges for each of these materials for use in residual stress calculations. Based on these results and the published literature, estimates were obtained for the flow stress, ultimate tensile strength, elastic modulus, and Poisson's ratio of the adherents as a function of temperature.

Five different specimen types were used to characterize the materials and joints. Unnotched bend specimens were loaded in 4-point bending to determine the bend strength and stress-strain behavior of monolithic materials and specimens containing joints. Notched bend specimens were loaded in 4-point bending to determine the fracture toughness of monolithic materials and specimens containing joints. The notched bend specimens containing joints were used in the preliminary validation procedure. Tensile specimens were used to evaluate the stress-strain behavior of nodular cast iron and Incusil ABA. Uncracked disk specimens were loaded in diametral compression to evaluate the shear strength of zirconia/zirconia joints. Cracked disk specimens were loaded in diametral compression for final validation of the joint design methodology. The disk specimens had bond areas that were approximately 20 times that of the bend bars used in the preliminary validation.

Approximately 160 room temperature tests and 30 high temperature tests were performed to generate material data and validation results. For the zirconia/zirconia joints high temperature tests were performed at 1000 C; for zirconia/NCI joints high temperature tests were performed at 400 C.

Fracture surfaces were examined optically and using a scanning electron microscope. Failure of zirconia/zirconia joints occurred primarily through the ceramic interlayer, although in a few cases the crack also propagated partly through the reaction zone of the base material. The propensity of the crack to propagate through the interlayer provided a good opportunity to predict the failure load of these joints based on the strength and toughness of the bulk interlayer; this was the approach used in failure load predictions. For the zirconia/NCI joints, however, failure occurred partly through the reaction zone of the

zirconia and partly through the Ti interface of the braze-metal/zirconia interface. In this system, the fracture toughness associated with the failure location had to be estimated indirectly.

VALIDATION OF JOINT ASSESSMENT AND DESIGN METHODOLOGY

Two kinds of validation experiments were conducted to compare with the JAD model analyses. Bend bars, both notched and unnotched, were used in preliminary validation experiments. These experiments were conducted to fine tune the model and provide some degree of assurance that the predictions made by the methodology were reasonably accurate. The second type of experiments were termed final validation experiments and were conducted on disk specimens. Because the geometry of the disk specimens was significantly different than that of the bend bars, these experiments provided a challenging validation of the design methodology developed on this program.

In all of the comparisons made between computations and experiments the parameter used for comparison was the failure load. During preliminary validation the model was used to calculate the failure load of notched bend bars fabricated from zirconia/zirconia joints and zirconia/nodular cast iron joints. Final validation computations were made on disk specimens of a similar variety; zirconia/zirconia disks and zirconia/nodular cast iron disks.

For several tests of zirconia/zirconia bend bars with a notch the measured failure loads had an average value of 211 N. The design model predicted a failure load of 260 N. For zirconia/cast iron bend bars an average failure load of 514 N was measured. This compares with a calculated value of 505 N. In an example of a zirconia/zirconia disk with a chevron notch and loaded in compression in line with the joint, the measured failure load was 1442 N while the calculated failure load was 1600 N. A final example is supplied by a metal-ceramic disk loaded in compression with the crack and bond line at an angle of 15 degrees to the load line. The specimen failed at 6631 N and was predicted to be able to withstand 7100 N. The degree of difference between experiment and theory cited in these examples was typical for all of the validation experiments. A master plot comparing measured and predicted failure loads is presented in Section 4.0 Validation.

1.0 CERAMIC OXIDE JOINT DESIGN METHODOLOGY

1.1 JOINT ASSESSMENT AND DESIGN MODEL FORMULATION

In this section a predictive model that can be used in design and structural integrity assessments of ceramic to ceramic and metal to ceramic joints for advanced gas turbine and internal combustion engine applications is developed.

In the modeling effort, the measure of joint performance was taken to be its ability to withstand monotonically applied load. A pragmatic, continuum mechanics-based approach was adopted which links joint performance with joining process variables and physical properties of the constituents. The approach was based on the notion that for a given joint configuration, its ability to withstand load is dictated by strength (σ_c) and toughness (G_c) properties at the potential failure location and the residual stress (σ_R) induced by the joining

process. Physical properties of the joint constituents (ceramic, metal and joining material) and joint process variables (e.g., interlayer thickness and joining temperature) influence joint performance indirectly by changing σ_c , G_c , and σ_R . Also, it was assumed that a micromechanical defect population and distribution associated with a joining process affects joint performance indirectly by changing the apparent strength and toughness properties. The influence of joining area on performance was dealt with empirically by testing specimens with significantly different joint dimensions.

With strength, toughness, and residual stress being the primary factors influencing performance, effort was focussed on developing stress analysis and fracture mechanics based models which would determine the stress field (σ), crack driving force (G) and residual stress (σ_R) for the test specimen geometries shown in Figure 2. Only cracked/notched specimens with crack lengths significantly larger than the interlayer thickness were included in the JAD model calculations. However, the same methodology can easily be extended to macroscopically uncracked geometries, where any crack would typically have dimensions less than the dimensions of the interlayer thickness. As discussed in Appendix A, this modeling effort involved detailed linear and nonlinear finite element analyses whose results were subsequently used to develop simpler and easier-to-use engineering models for estimating σ , G and σ_R . Input to these stress and fracture mechanics models included physical properties of the joint constituent materials as well as two joining process variables—joining temperature (T_j) and interlayer thickness (t_j). The affect of other joining process variables could only be included empirically. For ceramic/ceramic joints, this was done by developing an empirical relation between joining material (MASZ) composition and the interlayer's elastic modulus, yield strength and toughness.

Following the development of engineering models capable of estimating σ , G , and σ_R corresponding to all the loads, specimens and material combinations included in the experimental part of the project, the focus shifted to developing a joint assessment and design model. The objective was to formulate a methodology in which the three primary factors (σ_c , G_c , and σ_R) would be represented in a synergistic and unified manner. This objective was achieved by invoking concepts in engineering nonlinear fracture mechanics^[1,2] to derive an expression for a joint assessment vector (\bar{A}) as follows:

$$\bar{A} = \left(\frac{G_e}{G_c} \right)^{\frac{1}{2}} i + \left(\frac{P}{P_c} \right) j \quad (1)$$

in which i and j denote orthogonal unit vectors, P is a load parameter (related to joint stresses due to applied loads and residual stresses) and P_c is the critical load parameter (related to strength, σ_c). The quantity G_e is the elastic crack tip energy release rate for the cracked joint geometry of interest, corresponding to the combined action of residual stresses and applied load. The magnitude of the vector,

$$A = \left[\frac{G_e}{G_c} + \left(\frac{P}{P_c} \right)^2 \right]^{\frac{1}{2}} \quad (2)$$

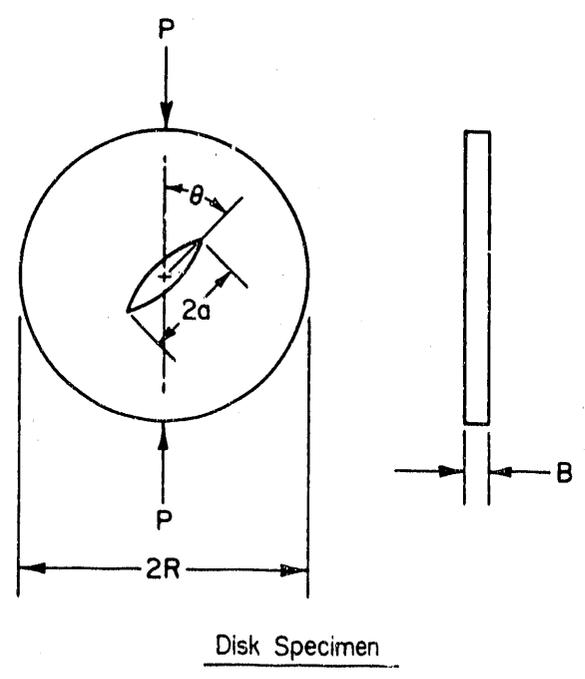
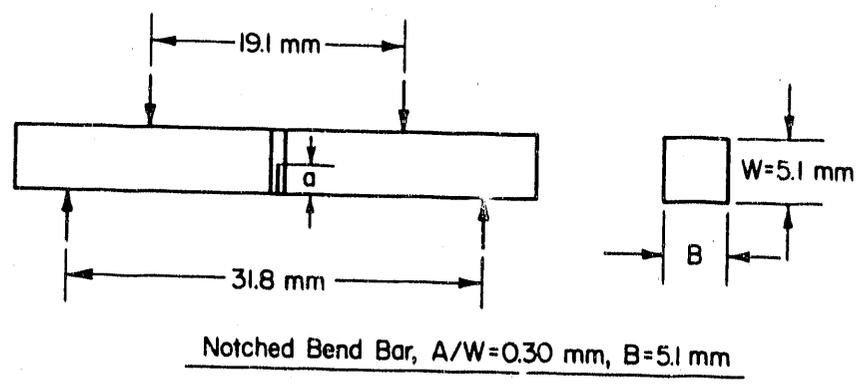
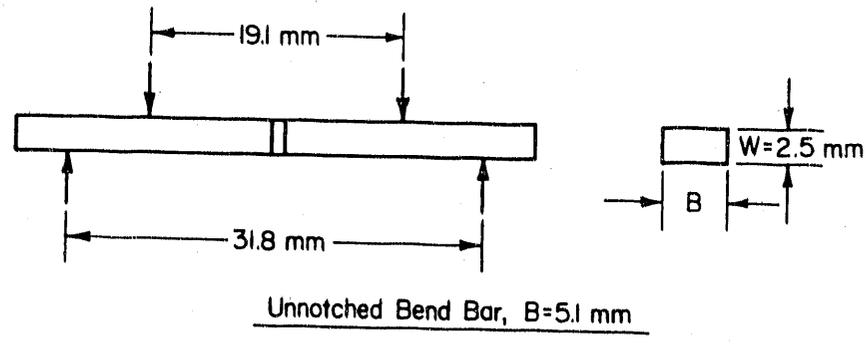


Figure 2. Test specimen geometries studied by finite element analysis.

represents the propensity for joint failure in the direction in G-P space given by

$$\phi = \tan^{-1} \left[\frac{\sqrt{G_e/G_c}}{(P/P_c)} \right] \quad (3)$$

The smaller the magnitude of A , the smaller is the propensity for joint failure. For a given joint type and geometry, failure would occur when the vector intersects the following failure curve

$$\sqrt{G_r} = \frac{\sqrt{G_e(a, P_r)}}{G(a, P_r)} \quad (4)$$

in which a is crack length dimension, $G_r = G_e/G_c$, $P_r = P/P_c$ and G represents the total (elastic-plastic) energy release rate. Note that equation (4) defines the failure curve and is not the definition of G_r . For $P \ll P_c$, the plastic deformation contribution to G is small, G approaches G_e , and the right hand side of Equation (4) approaches unity. For P approaching P_c , $G \gg G_e$, and the right hand side of Equation (4) approaches zero.

A graphical representation of the assessment vector [Equation (1)] and the failure curve [Equation (4)] is shown in Figure 3. Using the dimensionless quantities G_r and P_r , the assessment vector can be expressed as follows:

$$\bar{A} = (G_r)^{\frac{1}{2}} i + (P_r) j \quad (5)$$

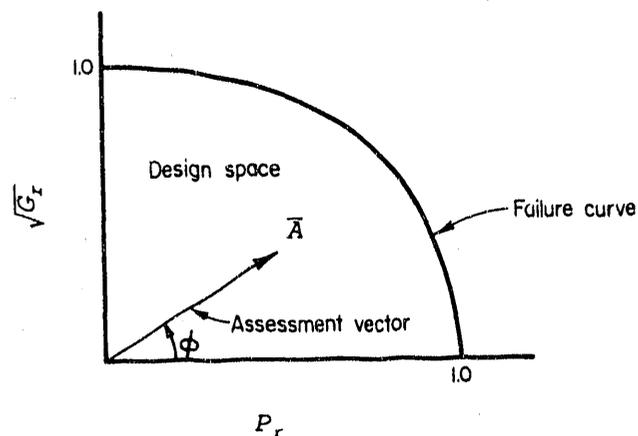


Figure 3. Graphical representation of assessment vector and failure curve.

In the graphical representation of the JAD model, the space enclosed by the failure curve (or a fraction thereof) may be defined as the design space within which the assessment vector \bar{A} would be required to reside. At a reference or anticipated service applied load (P_A), one would try to minimize A with respect to joint constituent properties and process variables to improve joint performance. To predict failure, one would find the applied load (P_f) such that the vector \bar{A} intersects the failure curve. Thus, for failure prediction one needs both the failure curve [Equation (4)] and the assessment vector [Equation (5)]. For improving design, only the assessment vector is needed.

For joint design one needs to specify a reference load (P_A) which is used to find the total load, P , on the joint

$$P = P_A + P_R \quad (6)$$

in the evaluation of A . In Equation (6), P_R represents a load parameter due to residual stress in the joint. From a practical viewpoint P_A should be chosen such that $P_R \ll P_A < (S_F \cdot P_f)$ where S_F is a safety factor and P_f is the failure load. For the present work, we choose P_A to be the minimum of the failure loads corresponding to a monolithic specimen of the materials being joined. For example, in designing a nodular cast iron/zirconia bend bar joint, P_A would be the failure load corresponding to a monolithic zirconia bend bar of the same dimensions and subjected to the same type of loading, knowing that the failure load corresponding to a cast iron bend bar would be higher. This particular choice of P_A is, of course, not mandatory. But it may have practical significance if it is argued that a component containing a joint need not withstand any more load than a monolithic component of either of the two materials being joined.

Based on the stress and fracture mechanics engineering models mentioned earlier, expressions for A and $\sqrt{G_f}$ for the four joint types and specimen geometry combinations are given in the following paragraphs. Details and justifications for the assumptions involved in their derivation are given in Appendix A.

1.2 ZIRCONIA/ZIRCONIA BEND SPECIMEN

For cracked zirconia/zirconia bend specimens, the failure curve is given by the following equation:

$$\sqrt{G_f} = \frac{\pi f g}{2 \sqrt{2}} P_f \left[\ln \left\{ \sec \left(\frac{\pi}{2} \cdot f \cdot g \cdot P_f \right) \right\} \right]^{-\frac{1}{2}} \quad (7)$$

where

$$g = 1 - \sqrt{P_f} + \frac{P_f}{f} \quad (8)$$

The assessment vector for this specimen type (for $P_R < P_C$) is given by the following equation:

$$\bar{A} = \left[\frac{3f(S-L)P}{2W^2t} \left(\frac{\pi a}{E'_1 G_{c1}} \right)^{\frac{1}{2}} \right] i + P_r j \quad (9)$$

In Equations (7), (8), and (9)

$$E'_1 = E_1 / (1 - \nu_1^2) \quad (10)$$

$$f = f_H \cdot f_s \quad (11)$$

$$f_H = 1.12 - 1.39 \bar{a} + 7.32 \bar{a}^2 - 13.1 \bar{a}^3 + 14.0 \bar{a}^4 \quad (12)$$

$$\bar{a} = a/W \quad (13)$$

$$f_s = 1.0 + \left(0.376 + 2.34 \frac{t_1}{2a} \right) \left(1 - \frac{\mu_1}{\mu_c} \right) - \left(2.504 + 3.87 \frac{t_1}{2a} \right) \left(1 - \frac{\mu_1}{\mu_c} \right)^2 \quad (14)$$

$$P_r = P_A \frac{1.5 (S-L) \bar{\sigma}}{W^2 t \sigma_{c1}} \quad (15)$$

$$P_A = \frac{W^2 t}{1.5 (S-L) f_H} \left(\frac{G_{cc} E'_c}{\pi a} \right)^{\frac{1}{2}} \quad (16)$$

$$P = P_A + \frac{2 W^2 t F_R \sigma_{RT}}{3 (S-L) f} \quad (17)$$

$$\bar{\sigma} = 1.0 + \left(\frac{t_i}{W} \right) \left(1 - \frac{\mu_i}{\mu_c} \right) \left[177 \frac{t_i}{W} - 10.9 \right] \quad (18)$$

$$F_R = \frac{f'_R}{\pi a} - f'_R \quad (19)$$

$$f_R = \frac{3.52}{(1-\bar{a})^{\frac{3}{2}}} - \frac{4.35}{(1-\bar{a})^{\frac{1}{2}}} + 2.13 (1-\bar{a}) \quad (20)$$

$$f'_R = 0.265 (1-\bar{a})^4 + \frac{0.857 + 0.265 (\bar{a})}{(1-\bar{a})^{\frac{3}{2}}} \quad (21)$$

The symbols ν , μ , E , and σ_0 represent Poisson's Ratio, the shear modulus, Young's Modulus and yield strength, respectively, with subscripts i and c denoting interlayer (MASZ) and ceramic (zirconia) materials. Geometric dimensions (a , W , t , S , L , and t_i) used in Equations (9) to (21) are shown in Figure 2. In Equations (11) and (14) the term f_a is a shielding factor and accounts for modulus mismatch effects on joint stresses. The word "shielding", in this context, has been coined because the interlayers used in this investigation (as well as in most other ceramic joining processes) had lower moduli than the adherents, and acted to reduce the local stresses and crack driving forces compared with corresponding values calculated based on a homogeneous material. The quantity σ_{RT} is related to the residual stress across the bond line which occurs close to the specimen edges. At the ceramic-interlayer interface,

$$\sigma_{RT} = \frac{2 t_i \sigma_t (\lambda - 1)}{\lambda (W - 2 t_i)} \left[\left(\frac{\sigma_t}{\sigma_Y + \sigma_t} \right)^{\frac{\lambda}{1-\lambda}} - 1 \right] \quad (22)$$

where

$$\sigma_t = \left| \frac{E'_i E'_c}{E'_i + E'_c} \left[(1 + \nu_i) \alpha_i - (1 + \nu_c) \alpha_c \right] \cdot \Delta T \right| \quad (23)$$

- ΔT = Difference between joining temperature (T_j) and test temperature (T_t)
- σ_y = minimum of σ_{oi} and σ_{oc}
- $\alpha_{i,c}$ = coefficients of linear thermal expansion corresponding to interlayer (i) and ceramic (c)
- λ = 1.0 + power of thermal stress singularity at the specimen edge. For a zirconia/zirconia joint with an MASZ interlayer, $\lambda = -0.97$.

For the parameters given in the third line of Table 1, a graphical representation of the JAD model application to a zirconia/zirconia bend bar joint is shown in Figure 4. The reference load, which is much higher than the predicted and measured failure load for the joint, is the load which would be needed to cause fracture in a geometrically identical monolithic specimen made of zirconia. The predicted failure load corresponds to the intersection of the vector with the curve at $P/P_o = 0.493$, and is somewhat lower than the measured value of 260N. To improve joint design one would need to increase G_{ci} and σ_{oi} (e.g. by increasing zirconia content in the interlayer material) which would reduce the assessment vector length. As discussed later in the report, empirical relations between σ_{oi} , G_{ci} , E_i and volume percent (V) of zirconia in the interlayer material (MASZ) are as follows:

σ_{oi}	=	$41.4 + 2.35 (V-50)$	MPa	
E_i	=	$109 + 0.1588 (V-50)$	GPa	$50 \leq V < 67$
G_{ci}	=	$[1.52 + 0.03 (V-50)]^2 / 10^3 E_i$	N/m	
σ_{oi}	=	$74.0 + 2.13 (V-67)$	MPa	
E_i	=	$111.7 + 2.2692 (V-67)$	GPa	$67 \leq V < 80$
G_{ci}	=	$[2.04 + 0.06 (V-67)]^2 / 10^3 E_i$	N/m	
σ_{oi}	=	$101.7 + 1.95 (V-80)$	MPa	
E_i	=	$141.2 + 3.04 (V-80)$	GPa	$80 \leq V$
G_{ci}	=	$[2.82 + 0.0125 (V-80)]^2 / 10^3 E_i$	N/m	

As an example, using 80 percent zirconia (instead of 67 percent) would reduce the assessment vector length by approximately 37 percent. Further reduction may also be effected by reducing interlayer thickness of the joint.

Table 1. Parameters used in assessment diagram calculations.

Joint Type	Joining Temp., C	Width Meters	Thickness Meters	Notch Depth Meters	Radius Meters	Theta Degrees	MASZ Percent
ZrO ₂ /Iron Bend Bar	700	.00508	.00508	.001600			
ZrO ₂ /Iron Disk	700		.00363	.000825	.00178	10.00	
ZrO ₂ /ZrO ₂ Bend Bar	1350	.00508	.00580	.001549			67
ZrO ₂ /ZrO ₂ Disk	1350		.00363	.008250	.01780	0.00	80

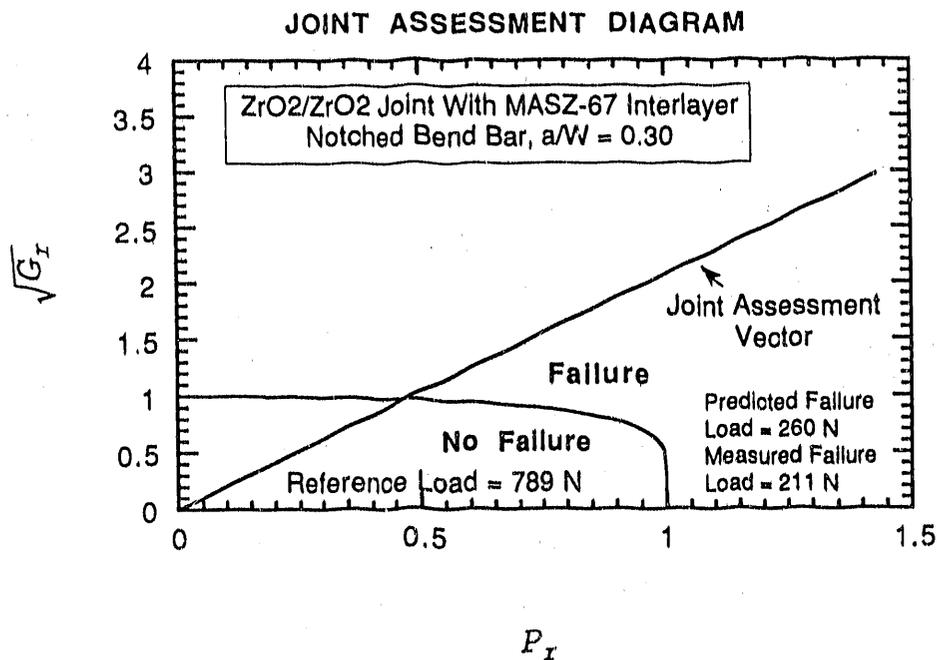


Figure 4. Assessment diagram for zirconia/zirconia bend specimen.

1.3 ZIRCONIA/NODULAR CAST IRON BEND SPECIMEN

For ceramic/metal bend specimens (with appropriate material property changes) all the equations from (7) to (23), with the exception of Equations (14) and (18), still apply. The dimensionless quantities f_s and $\bar{\sigma}$ are now redefined as:

$$f_s = 0.4766 + 1.0468 \left(\frac{\mu_f}{\mu_m + \mu_c} \right) \quad (24)$$

$$\bar{\sigma} = \left\{ 1.0 + \frac{t_f}{W} \left(1 - \frac{2 \mu_f}{\mu_m + \mu_c} \right) \left[177 \frac{t_f}{W} - 10.9 \right] \right\} \cdot \left[0.98 + \beta \left(\frac{2x}{t_f} \right) + \gamma \left(\frac{2x}{t_f} \right)^2 \right] \quad (25)$$

where $\beta = 0.0389$, $\gamma = 0.0949$, and x denotes the distance from the center of the interlayer to the crack location. For the cast iron/zirconia joints in the present program, the crack location was at the ceramic-interlayer interface. Thus, $x = t_f/2$. As before μ represents shear modulus and subscript m represents metal (cast iron).

For the parameters given in Table 1, a graphical representation of the JAD model application to a metal-ceramic bend specimen geometry is shown in Figure 5.

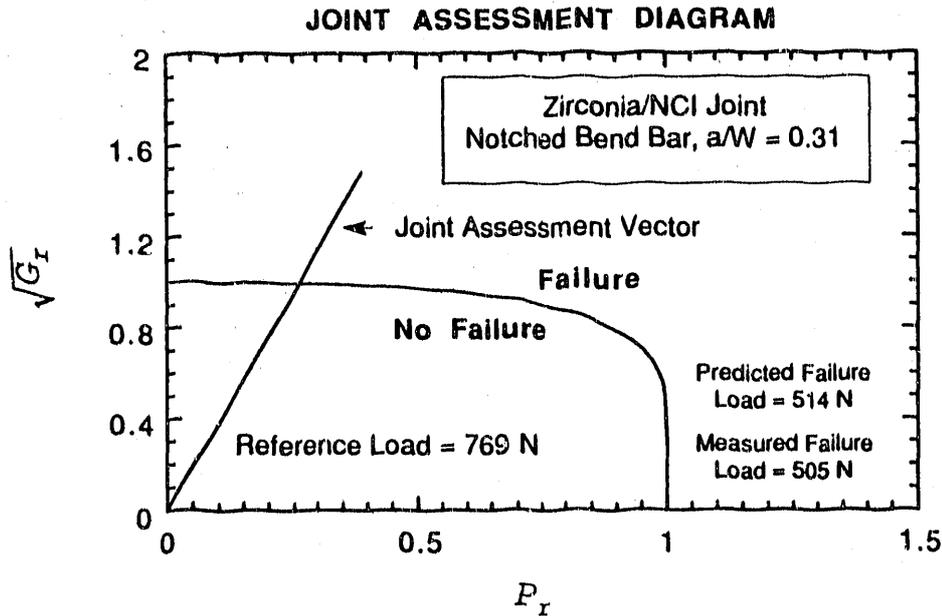


Figure 5. Assessment diagram for zirconia/cast iron bend specimen.

1.4 ZIRCONIA/ZIRCONIA DISK SPECIMEN

For a zirconia/zirconia disk specimen with load angle θ between the bond line and load line in the range $0.0 \leq \theta \leq 22.5$ degrees and $a/R = 0.5$ the failure curve is given by the following equation

$$\sqrt{G_r} = \frac{\pi f g}{2\sqrt{2}} P_r \left[\ln \left\{ \sec \left(\frac{\pi}{2} f g P_r \right) \right\} \right]^{\frac{1}{2}} \quad (26)$$

where

$$g = 1 - \sqrt{P_r} + \frac{P_r}{f} \quad (27)$$

The assessment vector for this specimen type (for $P_R \ll P_C$) is given by the following equation

$$\bar{A} = \left[\frac{f P}{Rt} \left(\frac{a}{\pi E'_1 G_{c1}} \right)^{\frac{1}{2}} \right] i + P_r j \quad (29)$$

In Equations (26), (27), and (29)

$$f = f_H \cdot f_s \quad (30)$$

$$f_H = 62.755 - 43.316 \sin \theta - 65.257 \cos \theta + 21.883 \sin^2 \theta + 4.485 \cos^2 \theta \quad (31)$$

$$f_s = (\cos^2 \theta) \cdot \left[\frac{\mu_i (1 - \nu_c)}{\mu_c (1 - \nu_i)} \left\{ 1.0 - 0.224 \left(1 - \frac{\mu_i}{\mu_c} \right) + 0.375 \left(1 - \frac{\mu_i}{\mu_c} \right)^2 \right\} \right]^{\frac{1}{2}} \quad (32)$$

$$P_r = P_A \bar{\sigma} / (\pi R t \sigma_{o1}) \quad (33)$$

$$P_A = \frac{Rt}{f_H} \left[\frac{\pi E_c G_{cc}}{(1 - \nu_c^2) a} \right]^{\frac{1}{2}} \quad (34)$$

$$P = P_A - \pi R t \sigma_{RT} \quad (35)$$

The quantity σ_{RT} is the same as defined in Equations (23) and (24), and for the disk specimen, the dimensionless quantity $\bar{\sigma}$ appearing in Equation (33) is approximately unity.

For the parameters given in Table 1, a graphical representation of the JAD model application to a zirconia/zirconia disk specimen geometry is shown in Figure 6.

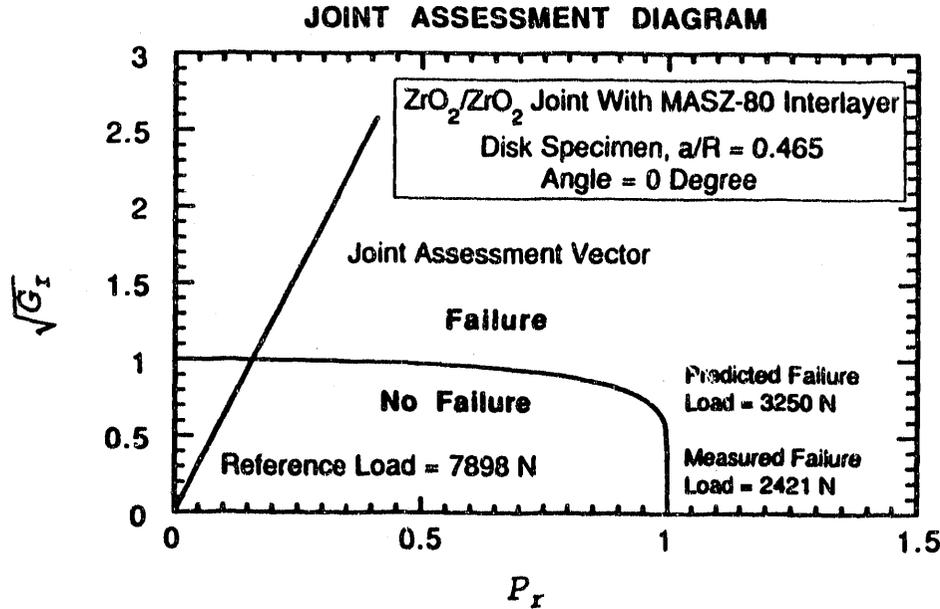


Figure 6. Assessment diagram for zirconia/zirconia disk specimen.

1.5 ZIRCONIA/NODULAR CAST IRON DISK SPECIMEN

For a zirconia/nodular cast iron disk specimen with load angle θ in the range $0.0 \leq \theta \leq 22.5$ degrees and $a/R = 0.5$, the failure curve and assessment vector are given by Equations (26) to (30). The dimensionless quantities f_H and f_s are for this case defined as follows:

$$f_H = [62.493 - 42.935 \sin\theta - 65.112 \cos\theta + 21.765 \sin 2\theta + 4.587 \cos 2\theta]^{1/2} \quad (36)$$

$$f_s = \left[0.4766 + 1.0468 \left(\frac{\mu_d}{\mu_m + \mu_c} \right) \right] \cos^2\theta \quad (37)$$

Equations (33), (34), and (35) remain applicable to metal-ceramic disk specimens with $\bar{\sigma} = 1.0$ and σ_{RT} defined by Equations (23) and (24).

For the parameters given in Table 1, a graphical representation of the JAD model application to a cast iron/zirconia disk specimen geometry is shown in Figure 7.

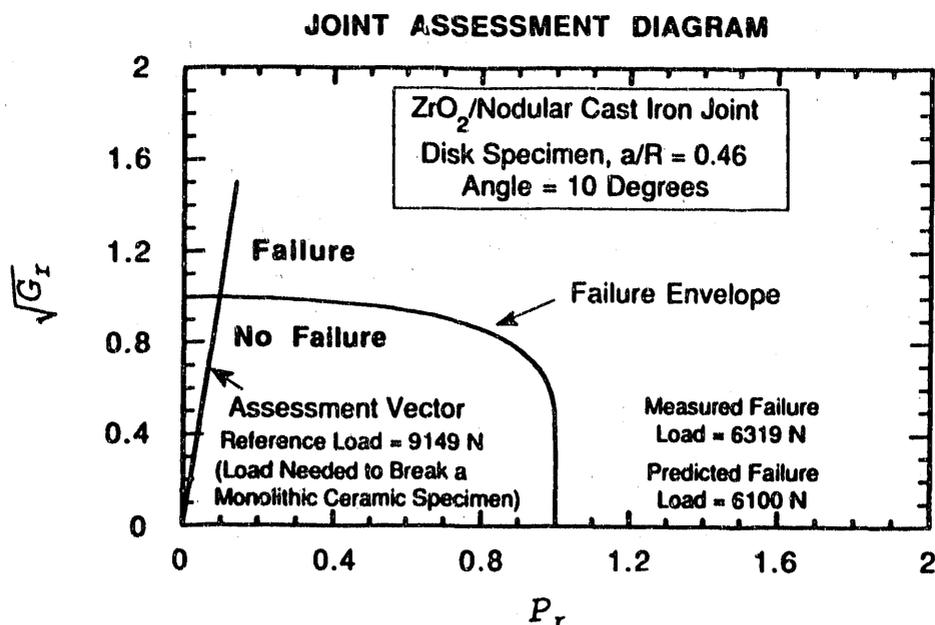


Figure 7. Assessment diagram for zirconia/cast iron disk specimen.

1.6 DISCUSSION OF THE JAD MODEL

The JAD model developed in the present work represents an attempt to provide a link between the structural performance of ceramic/ceramic and metal/ceramic joints with the fracture, mechanical and thermal expansion properties of the joint constituents (ceramic, metal and interlayer). For the materials and joint geometries selected for material property data generation and model validation, the JAD model was found to provide reasonably accurate results. The bend bar and disk specimens tested in the project represent significantly different geometries, giving rise to widely different stress distributions across the joint. The fact that the model provided reasonably good results for both geometries and for both material bonds, suggests that the modeling approach may provide a useful tool for design and assessment of joints.

While model validation was performed on only bend bar and disk (with varying load angles) specimens, the modeling approach adopted in the present work is quite general. It can be readily extended to other joint configurations as well as other material combinations. To do so, one would need to find (probably numerically) appropriate expressions for G_e , G , and P_r appearing in Equations (4) and (5) for the failure curve and assessment vector. If it is found (by numerical analysis of other material and geometry combinations) that the equation for the failure curve [see Equations (7) and (26)] is a reasonably good representation for other geometries as well, one would need to find only the dimensionless factors f and g for the new geometry and material. Figure 8 shows the variation in the failure curve shape using the extreme values of f [with g as defined by Equation (8)] encountered in the present work. The smaller value of f corresponds to a bend specimen and the larger value

corresponds to a disk specimen with load angle (θ) of 22.5 degrees. The figure suggests that the failure curve shape does not change drastically by varying f . Thus, for other geometries a fixed value of f (say, $f = 1.0$) may be appropriate. If both f and g are taken to be unity, the curve shape, also shown in Figure 8, becomes specimen geometry independent.

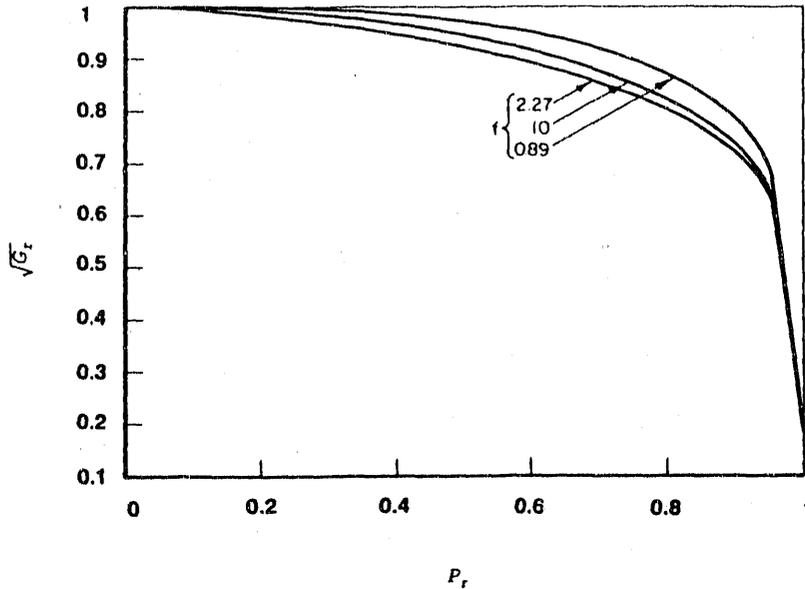


Figure 8. Variation in failure curve shape.

From a design view point, it is desirable that joining process variables (and not only constituent material properties) can also be linked to joint performance. Two of the process variables (joining temperature and interlayer thickness) are directly represented in the JAD model. For ceramic/ceramic joints with an MASZ interlayer, the zirconia content in the MASZ is also included in the model using empirical relations between percent zirconia and E_p , σ_{op} , and G_{cp} , as discussed in Section 3.0 Characterization and Analysis of Joints and Monolithic Materials.

The JAD model has been programmed in BASIC for use on an IBM-compatible personal computer. Instructions for obtaining a copy of the code can be found in Appendix D. The code (which contains the material property data base generated in the present project) represents a first step toward a potentially much more general and useful design tool for ceramic/ceramic and ceramic metal joints. A more general code would encompass a wider range of geometry configurations, material combinations, joining methods and service load and environmental conditions.

2.0 JOINT FABRICATION

Figure 9 summarizes the joint types and fabrication techniques used in this investigation. The adherents were magnesia partially stabilized zirconia and nodular cast iron. Small scale joints with bond areas approximately 12.5 mm x 12.5 mm were used for preliminary screening of the joining techniques. Later, large scale joints were fabricated using disks 51 mm in diameter and 19 mm thick and joining them along their flat faces. Mechanical test specimens used for preliminary and final validation were machined from these large scale joints. As illustrated in Figure 9 a number of techniques were tried for fabricating zirconia/zirconia and zirconia/NCI joint. Among these, the magnesia-alumina-silica-zirconia system was selected for fabricating zirconia/zirconia joints for validation testing. The active filler metal approach was finally selected for joining zirconia and cast iron. The details of material selection and fabrication techniques are discussed in the paragraphs that follow.

2.1 MATERIALS SELECTION

2.1.1 Zirconia

Type Zycron-L magnesia partially stabilized zirconia (PSZ) was purchased from Zircoa Products, Inc.. This grade was selected to obtain optimal strength and toughness up to temperatures of 400 C. It is chemically inert to molten metals, minimizing hot corrosion problems during brazing. Finally, a good thermal-expansion match to nodular cast iron minimizes residual stresses and a good elastic-modulus match minimizes interface stresses under load. Two other types of zirconia were used in the development stages of the joining process. These included an yttria zirconia which was fabricated by vacuum hot pressing at Ohio State and a magnesia zirconia, Nielsen grade MS, with an electron-beam PVC titanium coating supplied by ORNL. However, all of the joints used in validation testing were fabricated using the Zircoa Zycron-L material.

2.1.2 Nodular Cast Iron

A piece of nodular cast iron measuring 203 mm x 203 mm x 25 mm was supplied by Sandia National Laboratory and used in metal/ceramic joints. The material is of ferritic microstructure and close to SAE D4018 (equivalent to ASTM A536, Grade 60-40-18).^(a) However, it is weaker than the grade used previously in the ORNL bonding studies (D5506) and much weaker than the one currently being used there (8003).

(a) The SAE designation code for nodular iron is DXXOO, where XX = Yield Strength and oo = Elongation.

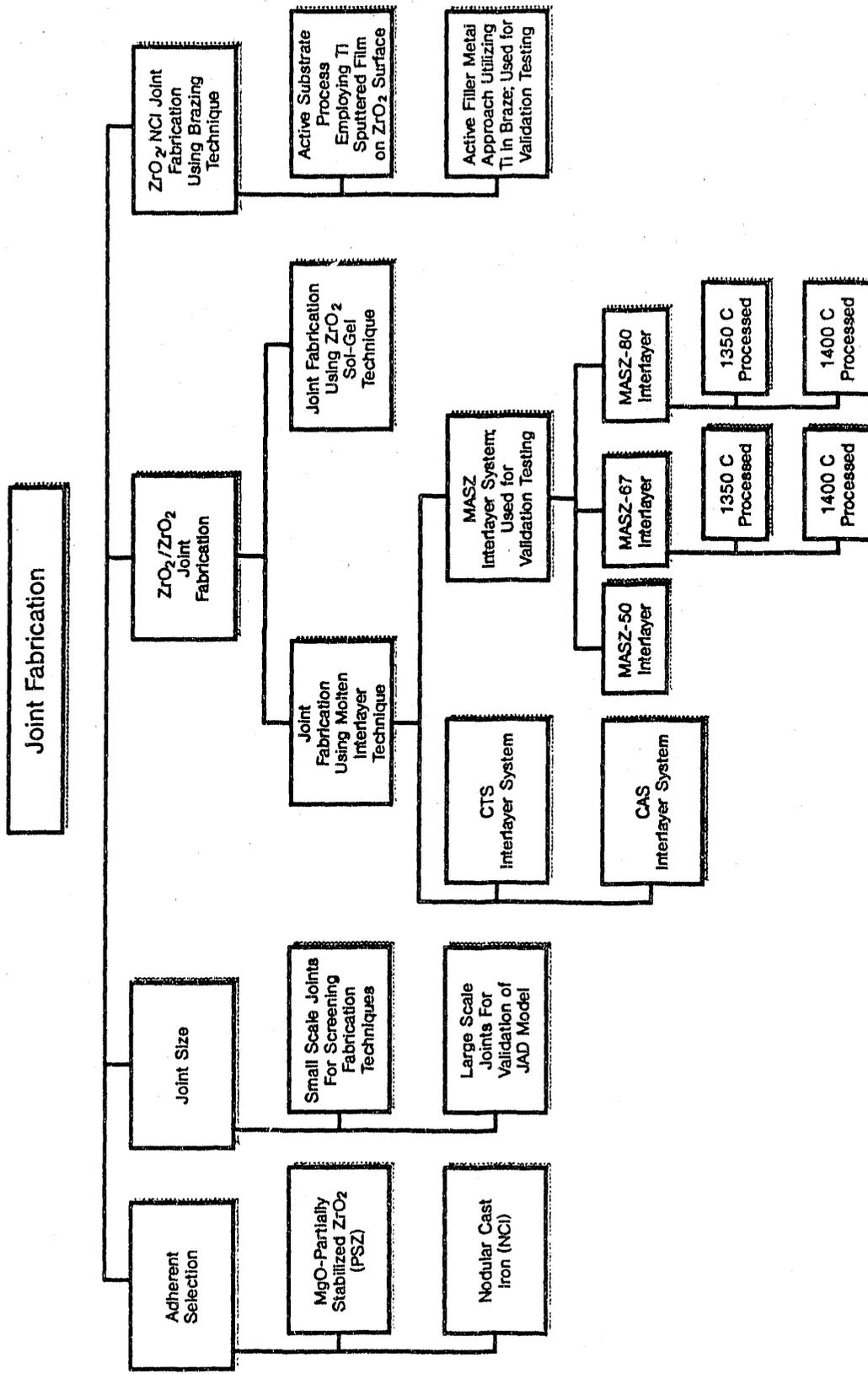


Figure 9. Summary of joint types and fabrication techniques.

The reasons for choosing this material were:

1. It has the most stable microstructure in this alloy system. Significant metallurgical changes can occur when these alloys are heated to around 760 C, which is only slightly above the temperature range in which brazing is normally carried out. As a result, there is concern that the mechanical properties of the iron after brazing will not only be significantly different from those of the as-received material, but also will vary from bond-to-bond, which would mask the effects of bonding variables. To compound the problem, some of the physical properties become erratic when these changes occur, complicating the model verification. Therefore, use of the ferritic material was expected to minimize problems associated with possible phase and microstructural changes.
2. The physical and mechanical properties of this grade of nodular cast iron have been characterized in great detail. For example, Sandia is doing extensive work on the same block that was used in this investigation.

2.2 ZIRCONIA/ZIRCONIA JOINTS

A literature survey indicated that there was no established technique that provided zirconia/zirconia joints of acceptable strengths at temperatures up to 1000 C. A brazing technique was ruled out because the braze metal is molten below that temperature. Accordingly, various ceramic interlayers were evaluated for joining zirconia to itself. Results on experiments performed with various interlayer materials are discussed below and also in the papers^[3, 4, 5] provided in Appendix B.

2.2.1 Sol-gel Zirconia Interlayer

The use of a zirconia interlayer would be ideal because the thermal expansion match would be good and because of the inherent strength of zirconia ceramics. However, a bond could be formed with a zirconia interlayer only through solid-state diffusion. It was felt that this would be possible with a highly reactive, fine-particle-size zirconia powder produced by sol-gel processing. A sol-gel method for the preparation of an MgO-doped zirconia powder was developed. This powder was used as an interlayer for a joining experiment with 12.5 mm diameter zirconia disks. The zirconia powder was dispersed in methanol and applied to the mating faces. Sintering of the zirconia sandwich in air at a temperature of 1500 C resulted in a bond that survived several drop-tests before failing. Observation of the fracture surface indicated that the bond was porous and that the interlayer exhibited lateral shrinkage during sintering. It was clear that the application of pressure during bonding would be required for a sol-gel zirconia interlayer to be effective for joining.

An air-ambient hot-forging apparatus was built at Battelle for this program, and two joining experiments were performed with the sol-gel zirconia interlayer. In the first experiment, two 12.5 mm diameter zirconia disks were joined by the application of 13.8 MPa at a temperature of 1600 C. A good bond was formed although the specimen size was too small for a bond strength measurement. Optical micrographs (Figure 10) showed that the as-formed bond region was very dense, although some porous regions were present. A bonding experiment was then repeated using 51 mm diameter zirconia billets.

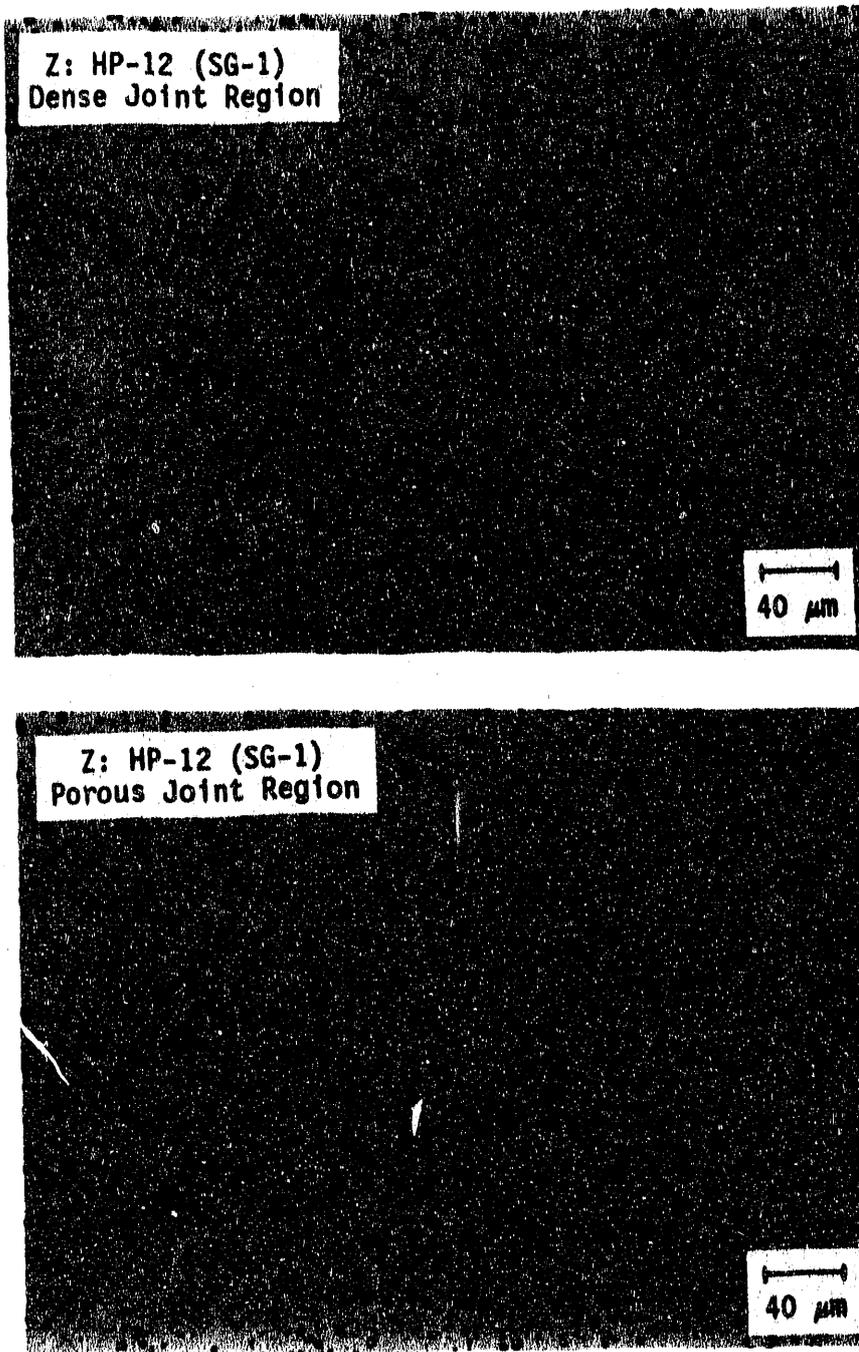


Figure 10. Optical micrographs of zirconia joint produced by hot-forging at 1600 C with a sol-gel zirconia interlayer. Both dense (top) and porous (bottom) joint regions are shown.

Unfortunately, this experiment ended in catastrophic failure of the alumina forging rams. It became clear that hot forging was a promising approach but that lower joining temperatures were required.

2.2.2 CTS and CAS Interlayers

The use of Battelle's air-ambient hot-forging apparatus for joining zirconia ceramics was limited to below about 1450 C. Since solid-state diffusional bonding with zirconia interlayers was not feasible at these low temperatures, a molten interlayer technique was considered to reduce the bonding temperatures for hot-forging. A temperature of 1420 C was selected as the bonding temperature, because it was thought that this temperature would minimize degradation of the base zirconia material (although this assumption was not evaluated). Two possible molten-interlayer systems were identified by searching through various phase diagrams. These were the CaO-TiO₂-SiO₂ (CTS) and CaO-Al₂O₃-SiO₂ (CAS) systems, each of which has eutectic compositions below 1400 C.

Interlayer powders of three CTS compositions and three CAS compositions were prepared by milling the oxide/carbonate raw materials, followed by calcination at 1100 C, and sieving to below 200 mesh. Compositions of CTS and CAS interlayer powders are given in Tables 2 and 3, respectively. Both small-scale (15 mm diameter disks) and large-scale (51 mm diameter disks) joining experiments were performed. Interlayers were applied to the zirconia disks through a methanol slurry, and disks were joined by hot-forging at 1420 C under pressures of 3.4 to 13.8 MPa. Both CTS and CAS interlayers provided successful small-scale bonds. Optical micrographs of small-scale zirconia joints produced with CTS and CAS interlayers are provided in Figures 11 and 12, respectively. Scale-up problems were encountered, related to thermal expansion mismatch and to "squeeze-out" of the interlayer material during hot forging. This was addressed by adding 30 wt% zirconia powder to the CAS and CTS interlayer material. The zirconia powder increased the viscosity of the interlayer during hot-forging, thus preventing "squeeze out", and also provided for a better expansion match between the interlayer and the zirconia base material. The best large-scale joint was produced by hot-forging with the CTS-3 interlayer composition, containing 30 weight percent zirconia. The average joint strength (four-point bending) was 67 MPa, with a Weibull modulus of 6.1. An optical micrograph of this joint, and an SEM micrograph of the fractured surface of a bend-test specimen, are shown in Figure 13.

Electron microprobe results obtained during initial CTS joining experiments were published^[3]. Additional work describing zirconia joints formed using interlayers in both CTS and CAS systems was also published^[4]. These two papers are reproduced in Appendix B.

Table 2. CaO-TiO₂-SiO₂ (CTS) interlayer compositions.

	Composition (wt%)				Comments
	CaO	TiO ₂	SiO ₂	ZrO ₂	
CTS-1	35.0	50.0	15.0		T _m = 1372 C (DTA)
CTS-2	25.0	57.0	18.0		eutectic composition T _m = 1356 C (DTA)
CTS-3	17.5	39.9	12.6	30.0	70 wt% CTS-2 + 30 wt% Zro2 T _m = 1357 C (DTA)

Table 3. CaO-Al₂O₃-SiO₂ (CAS) Interlayer compositions.

	Composition (wt%)				Comments
	CaO	Al ₂ O ₃	SiO ₂	ZrO ₂	
CAS-4	49.7	43.4	7.0		eutectic composition T _m = 1323 C (DTA) α = 8.4 ppm/C
CAS-5	41.0	11.8	47.2		eutectic composition T _m = 1308 C (DTA) α = 8.9 ppm/C
CAS-6	28.7	8.3	33.0	30.0	70 wt% CAS-5 + 30 wt% Zro2

2.2.3 MASZ Interlayers

The low joint strengths achieved in the CTS and CAS systems required consideration of alternative molten-interlayer materials. One possibility was the use of a glass-ceramic material that was molten during joining but would crystallize, providing high strength to the joint, during cooling. Glass-ceramics in the MgO-Al₂O₃-SiO₂ (MAS) system are used for certain structural applications, and Battelle's experience with these materials suggested reasonable high-temperature (1000 C) strength. One such glass-ceramic material is Zircoa 9606 glass-ceramic, which contains MgO, Al₂O₃, SiO₂, and TiO₂ (MAST). An initial, rather

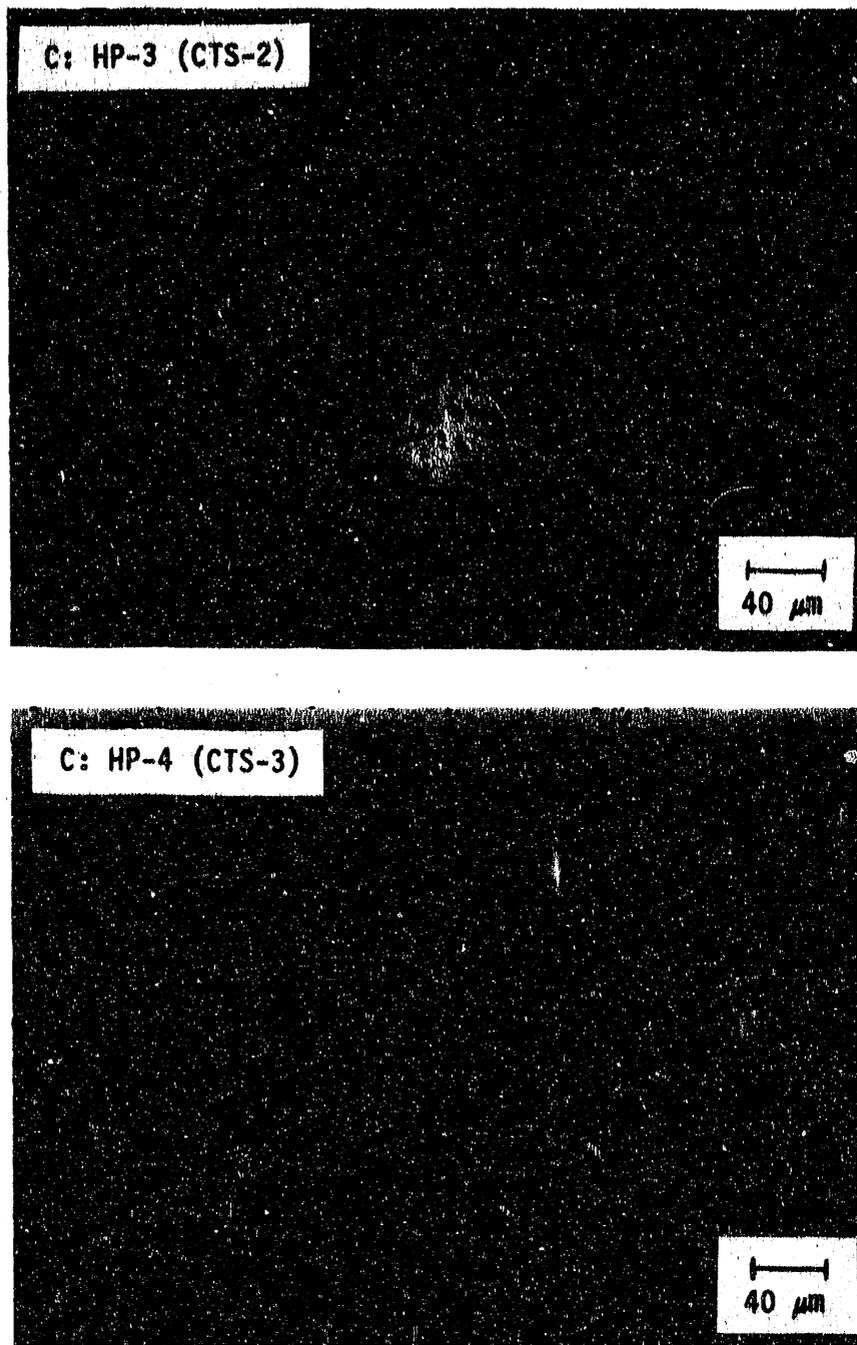


Figure 11. Optical micrographs of small scale zirconia joints produced by hot-forging with calcia-titania-silica CTS interlayers.

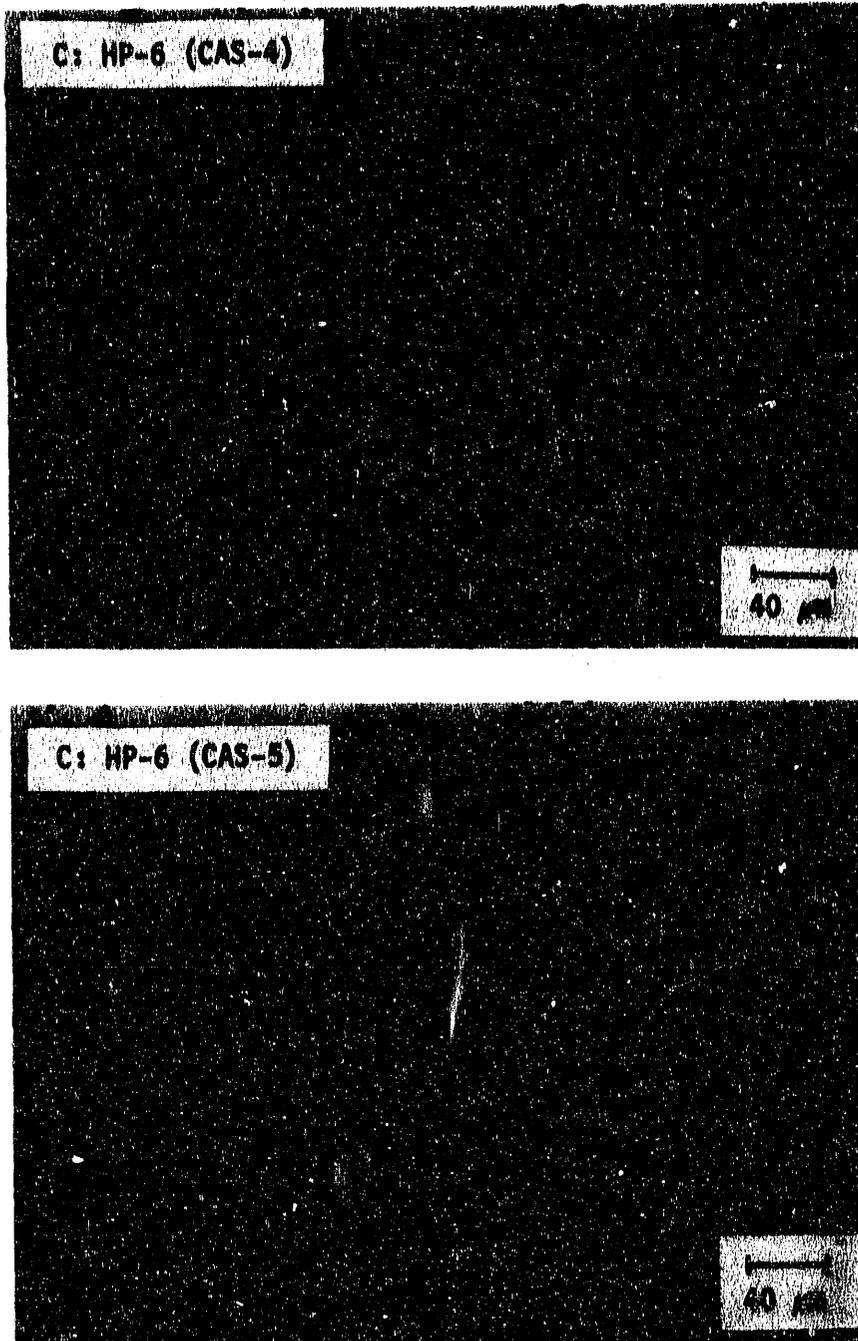


Figure 12. Optical micrographs of small scale zirconia joints produced by hot-forging with calcia-alumina-silica (CAS) interlayers.

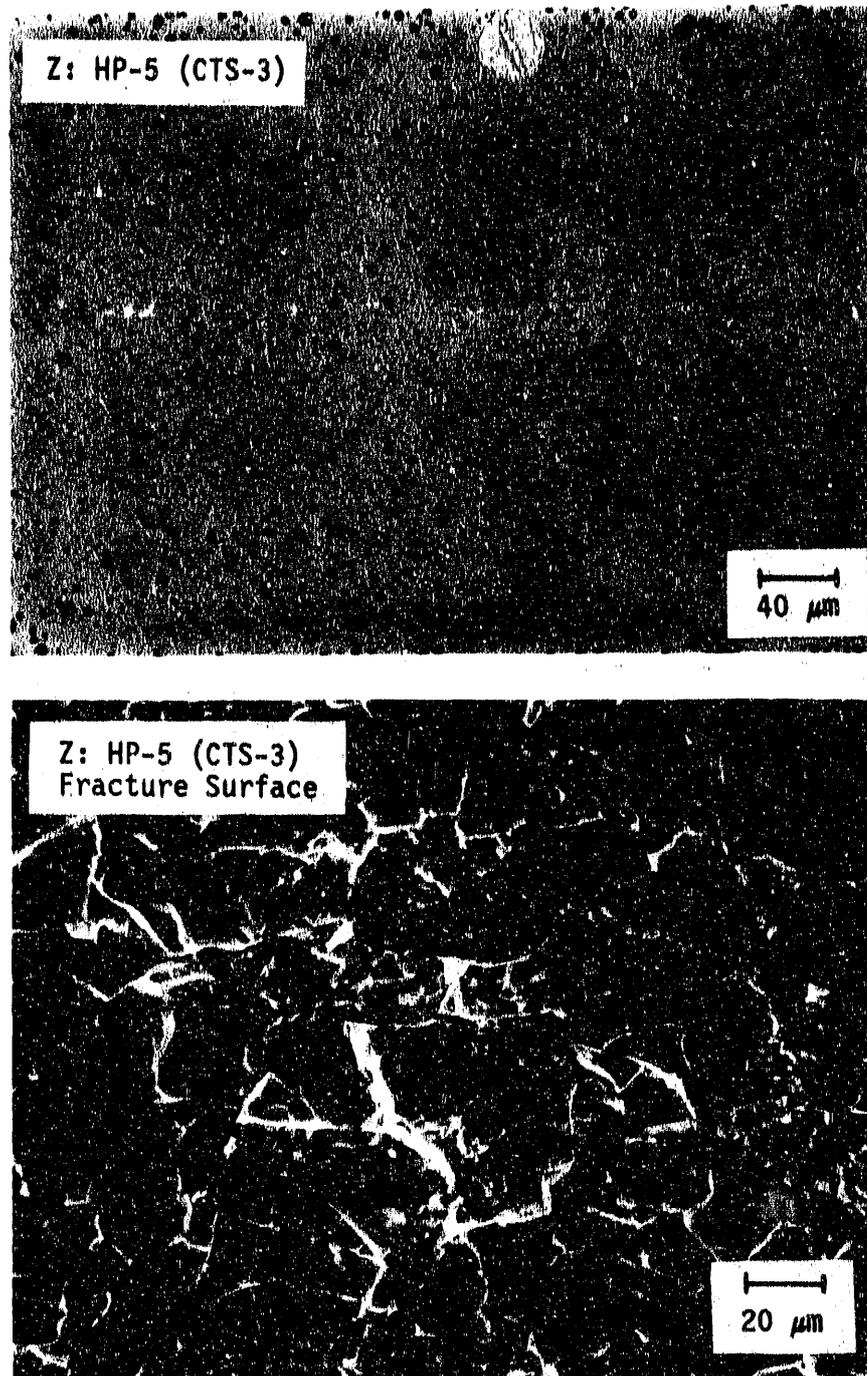


Figure 13. Top: Optical micrograph of large scale zirconia joint produced by hot-forging with the CTS-3 interlayer composition. Bottom: SEM micrograph of the fractured surface of a bend-test specimen produced from the above joint.

crude experiment was conducted to determine whether this material would be useful for joining. Some 9606 glass-ceramic material was crushed into a powder and applied as an interlayer between two pieces of zirconia, which were then heated (without pressure) to a temperature of 1300 C. The two pieces stuck together and survived drop tests.

More controlled joining experiments then were conducted with MAS interlayer powders with the 9606 glass-ceramic composition. Initial experiments with crushed 9606 glass-ceramic powder gave similar results to interlayer powders produced by milling and calcination at 1100 C. The use of MAST interlayers reduced the joining temperature to the range of 1320 to 1365 C. The best joining results were obtained when large amounts of zirconia powder (up to 80 weight percent) were added to the MAS interlayer powder. The family of magnesia-alumina-silica-zirconia interlayers are henceforth referred to as MASZ interlayers. MASZ interlayers with 67 and 80 weight percent zirconia were studied in detail in validation experimentation. The compositions of MASZ interlayers are provided in Table 4. The joining of zirconia ceramics using the MASZ interlayer powders required joining pressures of less than 2 MPa, much lower than those used for the CTS and CAS interlayers. This apparently was due to good wettability and high reactivity of the interlayer with the base zirconia material. The joint strengths achieved with MASZ interlayers also were much improved over CTS and CAS interlayers. For example, the best bend strength obtained for the CTS system was 66 MPa while the average strength of the MASZ-80 system was 158 MPa.

Table 4. MgO-Al₂O₃-SiO₂-TiO₂-ZrO₂ (MASZ) interlayer composition.

Powder No.	MgO	Al ₂ O ₃	SiO ₃	TiO ₂	ZrO ₂
MASZ-0	14.9	19.9	56.1	9.1	0
MASZ-33	10.0	13.3	37.6	6.1	33.0
MASZ-50	7.5	10.0	28.0	4.5	50.0
MASZ-67	4.9	6.6	18.5	3.0	67.0
MASZ-80	3.0	4.0	11.2	1.8	80.

The results of a detailed study of the joining of zirconia ceramics produced using MASZ interlayers were reported in two manuscripts which we show reproduced in Appendix B.

2.3 ZIRCONIA/NODULAR CAST IRON JOINTS

Two brazing processes were used in the fabrication of the zirconia/NCI joints. The active-substrate process developed at ORNL was initially selected because of the reported ability to form joints at temperatures sufficiently low to avoid degradation of the NCI^(a).

(a) Metals and Ceramics Information Center: Structural Alloys Handbook, 1989 ed., Vol. 1.

Repeated attempts to fabricate joints with contact areas on the order of 20 cm^2 using this process failed; these are described in Appendix C. At the suggestion of Dr. M. Santella of ORNL, the active-filler metal process was then employed in the fabrication of the successful joints used for validation studies. The active filler metal had the composition 59.18 Ag, 27.50 Cu, 12.13 In, and 1.18 Ti all in weight percent.

The surfaces of the billets were made flat and uniform by grinding to a 220 grit finish using standard machining practices for the NCI and a resin bonded diamond wheel for the zirconia. The NCI was used in the as-ground state, while (in latter experimentation) the zirconia was polished using a vibratory polisher to a specular finish using successive grades of diamond paste, typically to $9 \mu\text{m}$. Prior to assembly, or film deposition, all of the specimens were cleaned using acetone followed by a methanol rinse, typically using an ultrasonic bath. In most cases, filler-metal foils were lightly abraded to remove or break up any native oxide.

Brazing was carried out at OSU in a graphite resistance-heated vacuum hot press. The specimens were placed on a pedestal and brazing was carried out without an applied load. Typical vacuums were in the range of 2×10^{-5} to 1×10^{-4} torr. The heating schedule used with the active filler metal process was: 10-12 C/min to 650 C; 3 to 5 C/min to 735 C; and cooling at about 7 C/min to room temperature. Power was typically cut off when the temperature reached 735 C. It was found empirically that due to the thermal mass of the samples and the radiant properties of the graphite element, this procedure resulted in a maximum temperature very close to 750 C.

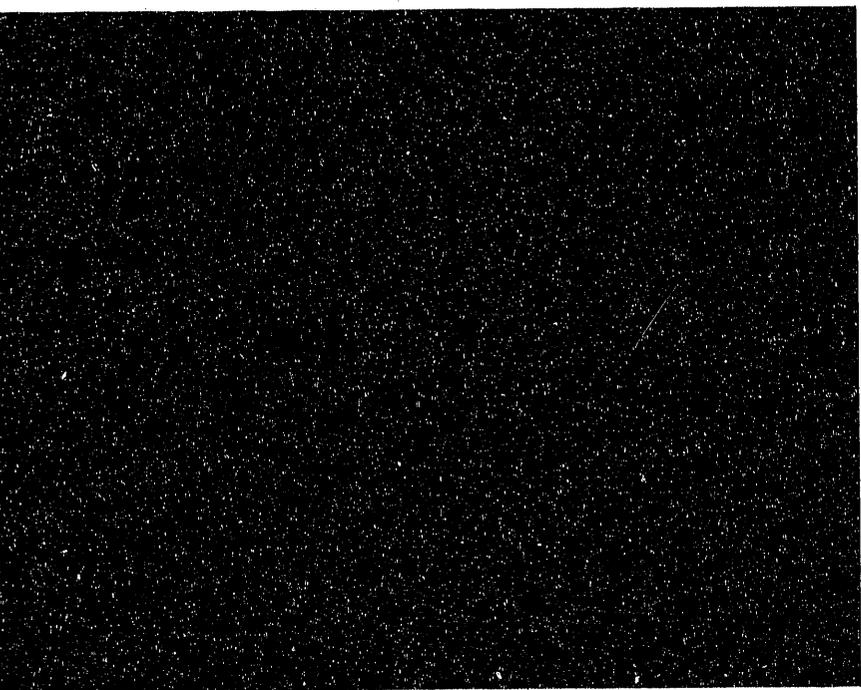
All efforts involving the active filler metal process resulted in strong joints. Ultrasonic scanning examination showed that the joints were free of microscopic defects; correspondingly, the unsuccessful joints fabricated using the active substrate process indicated a brain-like structure of the joint composed of large segregated regions of bonds and no bonds. Four large scale joints were brazed and used in the validation studies.

Microstructural analysis was performed on specimens taken from the first active filler metal process braze, using an electron microprobe using a wavelength dispersion spectroscopic (WDS) analyzer. Figure 14 shows a backscattered electron image and WDS element maps of the joint. Ti segregation to the region near the surface of the zirconia is evident (Figure 14b). Interestingly, a two-layer structure involving Ti is observed which is qualitatively very similar to that observed in the microstructure of the $2.0 \mu\text{m}$ thick Ti-layer active substrate joint. This is reasonable since the relative weight percentages of Ti, Ag and Cu are nearly the same for these systems (see Appendix C). Some differences, however, are apparent. The active substrate process employs Sn, and this element is found in significant proportion in the second Ti enriched region. In the active filler metal process Sn is replaced with In and careful examination of the element maps in Figure 14 reveals that the In is distributed in the Ag-rich phase and the outer Ti enriched region appears to be a Ti-Cu alloy. Also, the Ti-Sn-Cu phase in the active substrate joint is coarse relative to the Ti-Cu phase in the active filler metal joint.

Ti segregation appears to have had other beneficial aspects. Electron microprobe analysis identified a thin Ti enriched phase adjacent to exposed graphite nodules, perhaps involving carbide formation, and several isolated iron bearing particles, presumably milling debris, were encapsulated with a thin layer of Ti enrichment.

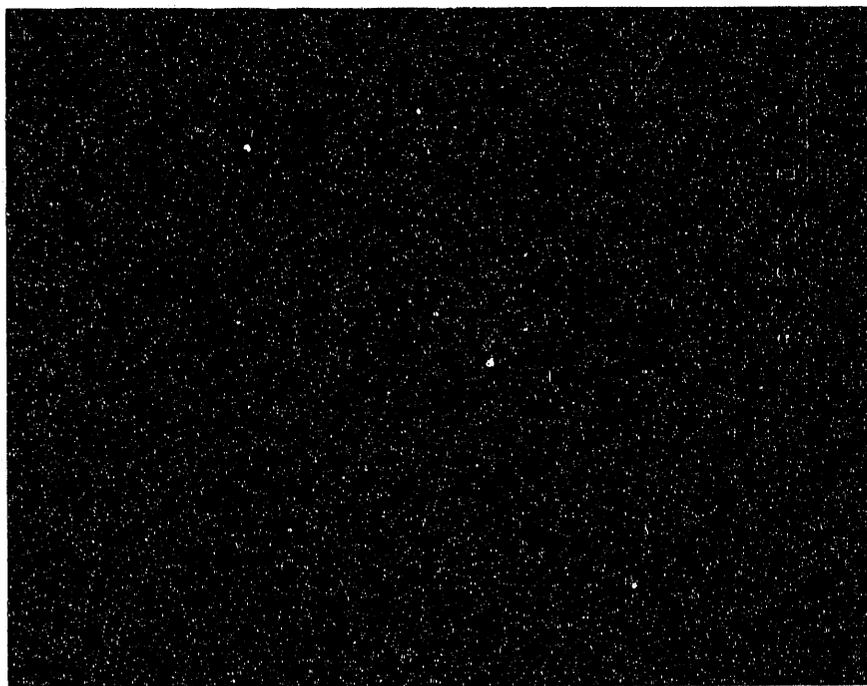


Figure 14a. Backscattered electron image of the interlayer region of an active filler metal joint fabricated using as-ground NCI, polished zirconia, and Incusil-ABA filler metal.



b

Figure 14b. WDS element map for Ti.



c

Figure 14c. WDS element map for In.

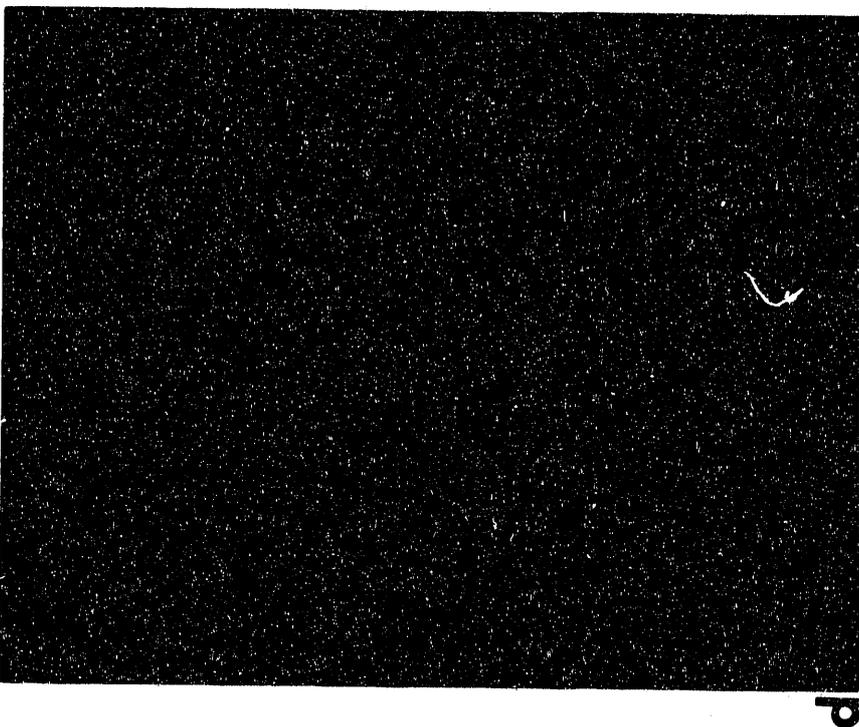


Figure 14d. WDS element map for Cu.

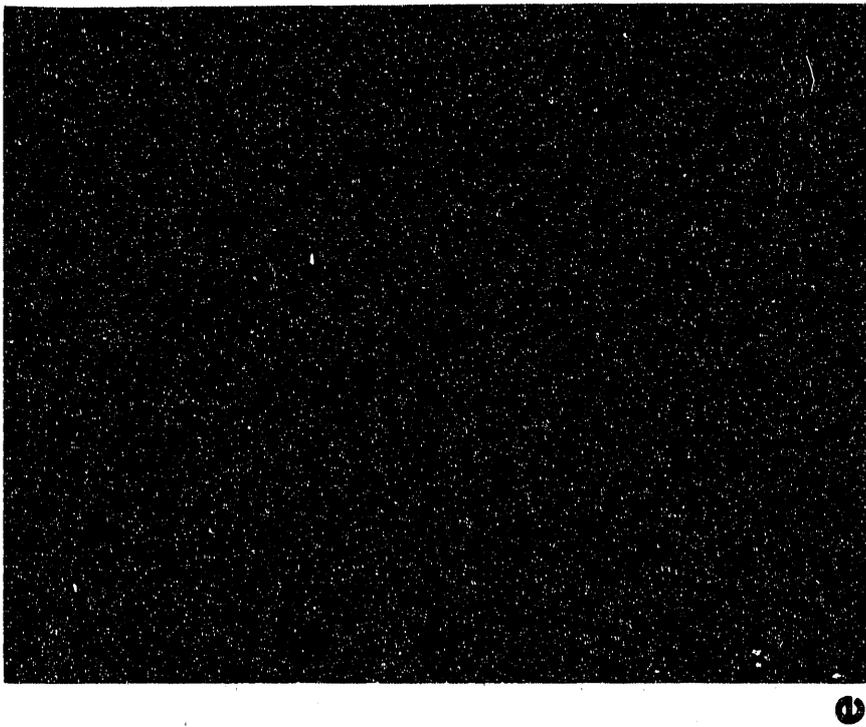


Figure 14e. WDS element map for Ag.

3.0 CHARACTERIZATION OF JOINTS AND CONSTITUENT MONOLITHIC MATERIALS

3.1 OBJECTIVES OF CHARACTERIZATION

Mechanical and thermal characterizations of materials (adherents and interlayers) were performed in support of the joint assessment and design model. The following were the three primary objectives:

1. To obtain constitutive properties of base materials and interlayer materials as input data to the JAD model.
2. To measure the load-bearing capability of small joint specimens (4-point notched/unnotched bend bars) for preliminary validation of the JAD model, and for incorporating appropriate modifications to the model to suit experimental results.
3. To measure the load bearing capability of large joint specimens (cracked disks) as part of the final validation procedure for establishing the JAD model.

The data generated included stress-strain relations and thermal expansion characteristics of base materials (adherents) and interlayer materials for input to the finite element analysis of the joints. Stress-strain data were obtained primarily at room temperature although a few experiments were also performed at elevated temperatures. Based on these results as well as data on the adherents in the published literature, estimates were obtained for the flow stress, ultimate tensile strength, elastic modulus, and Poisson's ratio of the materials as a function of temperature. The higher temperature data were needed primarily for calculating residual stresses, and it is envisaged that for more accurate residual stress estimates it would be necessary to actually perform additional tensile and bend tests at elevated temperatures.

Preliminary validation procedures involved determining the fracture strength and load-displacement behavior of small sized joints, and comparing the results with predictions of the JAD model. Notched bend bars, loaded in 4-point bending, were used for preliminary validation while unnotched 4-point bend bars were used for estimating the efficiency of the joints; efficiency is defined as the ratio of strength of the joint to strength of the monolithic ceramic. Experiments on unnotched specimens also helped in determining the preferred fracture path, namely, whether a crack propagated in the interlayer, the reaction zone of the base material, or through the base material. This served as an input to the JAD model. In this context, it may be noted that although strengths and toughnesses of adherents and interlayer materials can be determined separately, there is currently no straight-forward route for estimating a priori the corresponding properties of the reaction zone, or properties of the interface between the interlayer and adherents. Thus, unless the strength of these regions is greater than the adherent or the interlayer, or there is a convenient way of determining separately the strength and toughness of these regions, it is not possible to predict a priori the location of fracture, which is needed for the JAD model. Fortunately, as will be discussed later, failure of zirconia/zirconia joints occurred primarily through the interlayer, which coincided with the fact that the strength and toughness of the bulk

interlayer (either MASZ-67 or MASZ-80) was always less than the base heat-treated zirconia. On the other hand, in the case of zirconia/NCI joints, failure occurred partly through the reaction zone of the zirconia and partly through the Ti-rich region immediately adjacent to the zirconia. This fracture path could not be predicted based on bulk measurements, nor could the fracture load of notched bend bars be predicted based on data of braze metal or as-received zirconia. Such experiments further confirmed the importance of performing small-scale validation experiments as part of establishing a joint design methodology.

Final validation was performed by testing cracked disk specimens subjected to diametral compression, with the crack (along the joint plane) rotated at various angles to the loading direction. This specimen had a joint area that was 20 times that of the bend bars used in preliminary validation, and served to evaluate the JAD model for a scaled up joint. The disk specimen served two additional purposes. First, because the specimen geometry differed significantly from that of the bend bar, testing of the disk specimen helped in establishing geometry independence of the design methodology. Second, it helped in validating the joint model over a considerable mixed-mode (different combinations of opening and sliding modes) loading domain. This aspect, although not as significant in the fracture of monolithic materials, assumes considerable importance for joints, because of the inherent mixed-mode nature of loading of bimaterial interfaces.

The details of the characterization and analysis task are illustrated by the chart in Figure 15. As shown in the figure, the task has been grouped under the following headings:

1. Materials
2. Specimen designs
3. Key properties characterized
4. Test conditions
5. Microstructure
6. Fractography, particularly for determining the fracture path
7. Preliminary analysis

3.2 EXPERIMENTS

3.2.1 Materials Characterized

The bulk constituent materials characterized for input to the JAD model consisted of the following:

1. As-received partially stabilized zirconia. The data for this material were used for assessing the zirconia/NCI joints, since the thermal cycle associated with the brazing cycle (maximum temperature of 750 C) did not involve any phase

transformation, and was found not to significantly affect the bulk properties^(a) of the zirconia.

2. Heat-treated zirconia, involving a soak at 1350 C for 2 hours in air. The heat treatment was used to simulate the thermal cycle involved in the zirconia/zirconia joints. Although the thermal cycle (it involved phase transformations of the zirconia) was found to have negligible effect on the elastic properties of the zirconia it had significant effect on the flow strength, bend strength, and fracture toughness of the material as well as on the overall thermal expansion coefficient. For this reason it was believed appropriate that properties of the heat treated zirconia be used in the JAD model when analyzing zirconia/zirconia joints.
3. Nodular cast iron which was used for the zirconia/NCI joints. No heat treatment was applied to the NCI since the brazing temperature was too low to have any effect on this material.
4. Bulk MASZ-67 and bulk MASZ-80 interlayers. It may be recalled from the previous section that these magnesia-alumina-silica-zirconia interlayers were used for fabricating zirconia/zirconia joints.
5. Incusil-ABA active filler braze metal, used in fabricating zirconia/NCI joints. The active filler metal incorporated Ti, which was found to impart excellent wettability to the zirconia.

In addition to the bulk constituent materials, zirconia/zirconia and zirconia/NCI joints were characterized for validating the JAD model, and also for incorporating modifications as necessary in the model to account for observed phenomena. For example, preliminary validation experiments helped to identify the fracture path, and also to estimate an appropriate strain energy release rate value for zirconia/NCI joints.

The zirconia/zirconia joints that were characterized included joints fabricated using the following interlayer materials at the bonding temperature indicated:

1. Zirconia/zirconia joints fabricated using MASZ-50 interlayer and fabricated at 1350 C.
2. Zirconia/zirconia joints fabricated using MASZ-67 interlayer and fabricated at 1350 C.

(a) However, as will be indicated later, the brazing process involved formation of a reaction zone in the zirconia immediately adjacent to the braze metal. Because it was not possible to obtain bulk specimens from the reaction zone, in the JAD model it was assumed that the reaction zone had the same elastic and thermal properties as the bulk material. The toughness value was obtained by testing a notched bend bar with the crack located in the reaction zone but at a slight distance from the braze-metal/reaction zone interface. This toughness was found to be lower than that of the bulk zirconia.

3. Zirconia/zirconia joints fabricated using MASZ-67 interlayer and fabricated at 1400 C.
4. Zirconia/zirconia joints fabricated using MASZ-80 interlayer and fabricated at 1350 C.
5. Zirconia/zirconia joints fabricated using MASZ-80 interlayer and fabricated at 1400 C.

The zirconia/NCI joint that was characterized was fabricated using Incusil-ABA active filler braze metal at a joining temperature of approximately 750 C.

In addition to the above joint types, many zirconia/zirconia joints such as those fabricated using calcia-titania-silica (CTS) interlayer, or zirconia/NCI joints fabricated using the active substrate process, were characterized but they were not included for further analysis because of poor joint strengths. Those data are not included in this report, although some of the data are available as part of the published papers^[3,4] reproduced in Appendix B.

3.2.2 Specimen Design

As illustrated in Figure 15, a number of different specimen geometries were used for characterizing the mechanical behavior of bulk monolithic materials and joints. The specimen types were:

1. Unnotched rectangular bars (Figure 16a), loaded in 4-point pure bending, for determining the bend strength and stress-strain behavior of monolithic ceramic specimens and specimens containing joints.
2. Notched bend specimen (Figure 16b), loaded in 4-point bending, for determining the fracture toughness of monolithic ceramic specimens and specimens containing joints. The machined notch was aligned along the mid-plane of the bond. This specimen geometry was used as part of the preliminary validation procedure.
3. Tensile specimen, used for evaluating the stress-strain behavior of nodular cast iron and braze metal.
4. Uncracked disk specimen, loaded in diametral compression, with the bond plane rotated at an angle of 30 degrees to the load axis. This specimen design was used for evaluating the shear strength of zirconia/zirconia joints.
5. Cracked disk specimen, loaded in diametral compression, with the machined notch/crack at various angles to the loading axis. This specimen geometry was used as part of the final validation procedure, and it had a bond area that was approximately 20 times that of the bend bar used in preliminary validation of the joint assessment model.

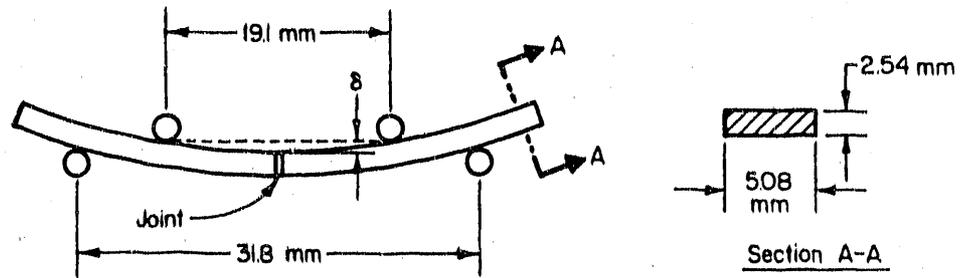


Figure 16a. Bend test geometry for evaluation of the bend strength of joints and of constituent materials. Displacement, δ , was measured between the upper loading pins and the center of the compression face of the bend bar, using a high-resolution LVDT.

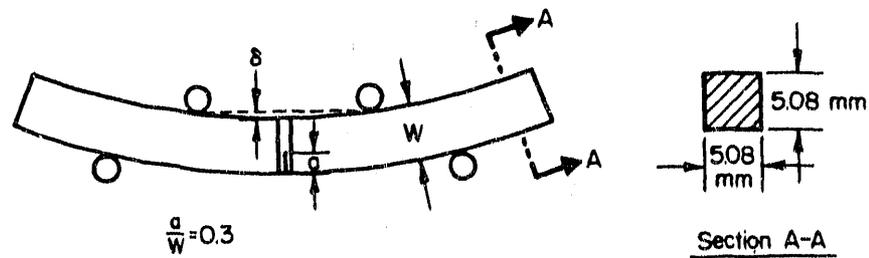


Figure 16b. Notched bend test geometry for evaluating the fracture toughness of constituent materials, and for preliminary validation testing of joints.

The 4-point bend specimen (Figure 16), was of nominal width 5.08 mm, thickness 2.54 mm, and length 38.1 mm. The specimen was finish ground using a 110-grit diamond wheel and was loaded under 4-point bending using a self-aligning fixture that allowed for free rotation in two directions. Figure 17 is a photograph of the experimental setup. The lower loading pins were 31.75 mm apart, while the upper loading pins were 19.05 mm apart. A high accuracy displacement measuring fixture was used to determine the center displacement of the bend bar with respect to the upper loading pins; a displacement of 0.1 μm was easily resolved by the LVDT assembly and calibration procedure. For some of the specimens that were used for generating stress-strain curves, the strain on the tension face of the bend bar was monitored using a strain gage bonded to that face. In this way, it was possible to determine the true stress-strain curve from the measured stress-strain and load-displacement plots.

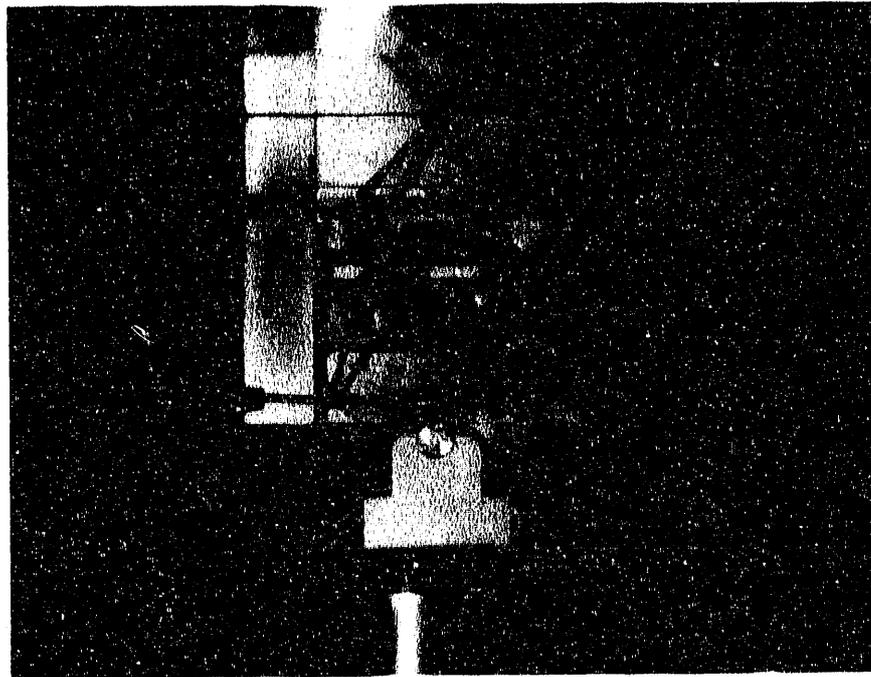


Figure 17. Experimental setup for bend testing of constituent materials and joints. The LVDT core-rod is located at the center, above the specimen.

The notched bend bar specimen is illustrated in Figure 16b. A fine slitting saw was used to make the notch, which was approximately 0.3 mm wide. The ratio (a/W) of crack length (a) to specimen depth (W) was approximately 0.3. Fatigue precracking was not attempted since earlier experiments, involving toughness evaluation of the as-received zirconia using straight notched and chevron notched bend bars as well as chevron-notched disk specimens, indicated that the straight-notched bend specimen provided fracture toughness data that were in excellent agreement with those generated using other specimen geometries.

The machined notch was centered along the mid-plane of the bond in the case of zirconia/zirconia bonds. However, for zirconia/NCI joints it was not always possible to maintain the notch along the mid-plane of the bond. Rather, because of softness of the braze metal, there was always a tendency for the fine slitting saw to deviate into the reaction zone of zirconia, although the extent of deviation was extremely small (typically less than 50 μm). Preliminary experiments indicated that this did not have any significant effect on the fracture toughness value. This was in agreement with the observed fracture path for unnotched bend specimens, which showed that crack propagation occurred partly through the reaction zone of the zirconia and partly through the Ti-interface next to the zirconia. Hence, for consistency purposes, the machined notch was cut in the reaction zone, and within 50 μm of the zirconia/braze interface. Fractographic evidence indicated that this did not prevent the crack from partly weaving through the Ti-rich zone of the braze metal.

The geometries of the uncracked disk and cracked disk specimens are illustrated in Figures 18a and 18b, respectively. The specimens were loaded in compression, with paper cards used to prevent crushing the specimens at the loading points. The uncracked disk specimen was used to determine the shear strength of joints by orienting the crack plane and bond at an angle of 30 degrees to the compression loading axis. For such an angle, the normal stress is zero at the center, and the shear stress (assuming homogeneous material and no residual stresses) is given by:

$$\tau = \sqrt{3} P / \pi RB$$

In the case of the cracked disk specimen, a fine slitting saw was also used to cut the notch, approximately 0.3 mm wide. Figure 19 is a photograph showing the fine slit in a disk specimen. The slit is located well within the dark band which is the reaction zone of the zirconia, although in this particular case the slit is not exactly centered on the bond-line; in some of the specimens, the tip of the notch did coincide with the center-line of the bond, but these specimens did not indicate any significant difference in fracture load compared with slits machined in the manner shown in Figure 19.

The ratio of half-notch length (a) at the specimen faces to the radius (R) of the disk was nominally 0.5, while the ratio of half-length of the chevron notch at mid-thickness to the radius was typically 0.41. For the chevron-notched disk specimen, a stable crack is generally formed that propagates through the entire chevron notch prior to fast fracture. However, it was observed that when the crack angle was large (typically greater than 15 degrees), there was a sharp deviation of the crack from the bond and notch plane even before the crack had propagated through the entire chevron notch. Therefore, in all cases, the crack length at fast fracture was estimated from the fracture surface; i.e., a/R that was used for calculation purposes was obtained from observations of the fracture surface. The crack opening displacements (COD) were measured in a direction perpendicular to the crack plane using a COD gage with strain-gage arms. The knife edges were located on the specimen at equal distances of 3.81 mm on either side of the slit. Figure 20 is a photograph of the experimental setup. The extensometer was calibrated over a maximum span of 25 μm , and this provided a resolution of better than 0.1 μm for monitoring crack opening displacements.

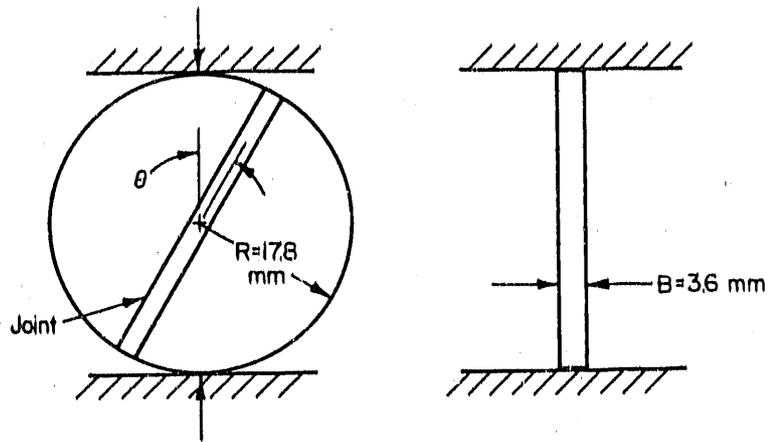


Figure 18a. Un-notched disk specimen for measuring the shear strength of joints.

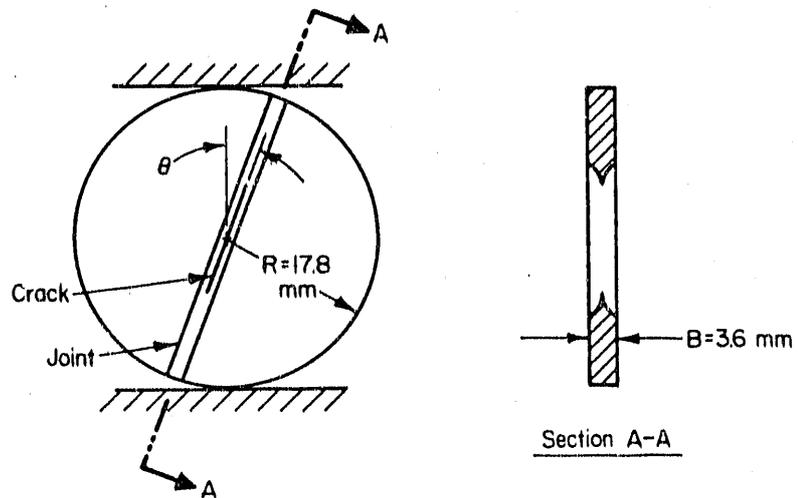


Figure 18b. Chevron-notched cracked disk specimen used in final validation testing of joints.

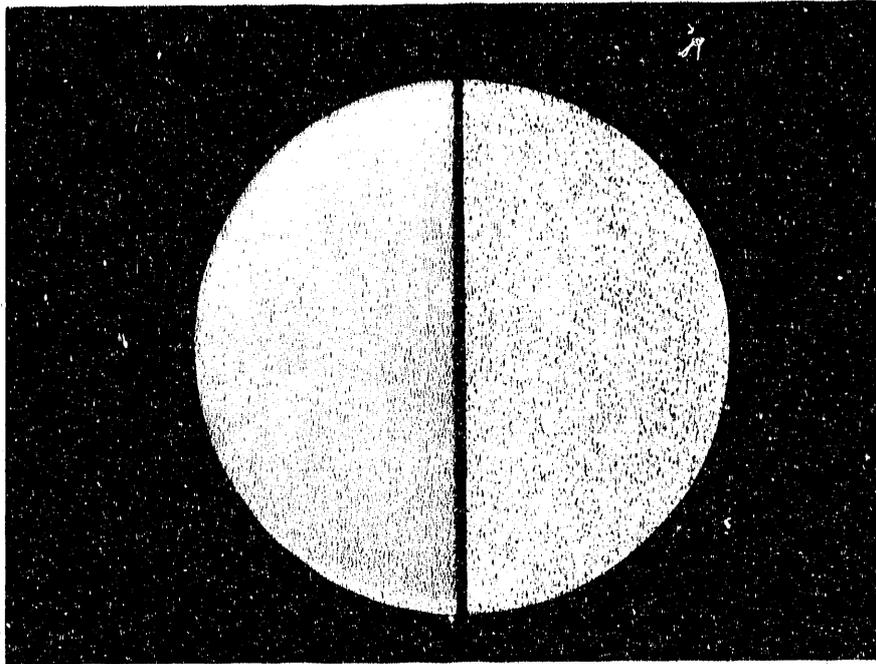


Figure 19. Photograph of zirconia/NCI cracked disk specimen. The zirconia is on the left and NCI is on the right.

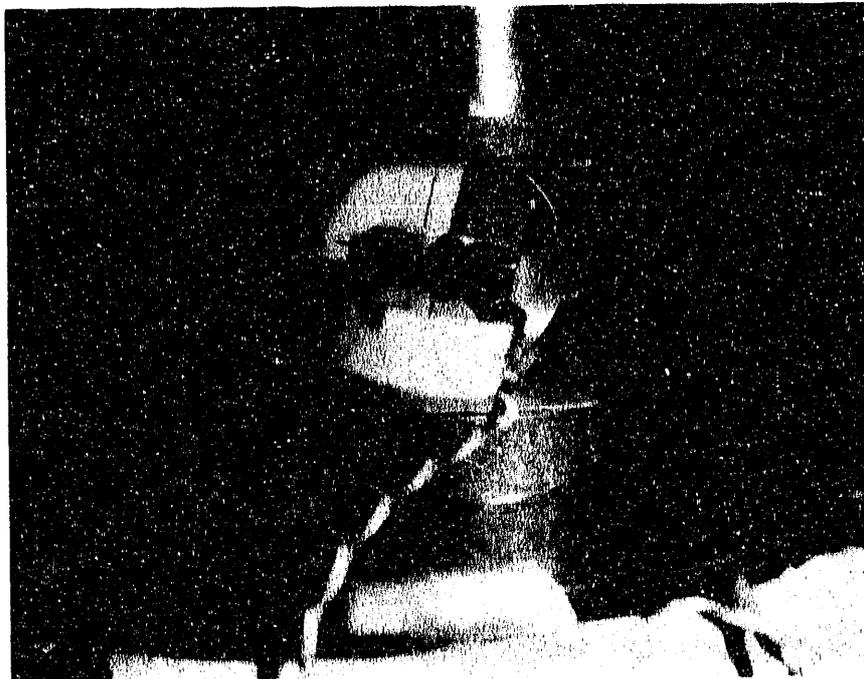


Figure 20. Experimental setup used in final validation testing of joints using cracked disk specimen.

Tensile sheet specimens were used for evaluating the stress-strain behavior of the braze metal and nodular cast iron. These were pulled to failure in a servo-hydraulic testing machine. Strain was monitored with an extensometer employing strain gages.

3.2.3 Key Properties Characterized

Thermal Expansion. The only physical property that was measured was the thermal expansion behavior of adherents and interlayers, and they served as input to the FEM code for evaluating residual stresses. The thermal expansion of as-received zirconia, NCI, and braze metal were determined from room temperature (RT) to 730 C, which was near the processing temperature for zirconia/NCI joints. The thermal expansion trace was essentially linear, and did not indicate any phase transformation over the temperature domain. The thermal expansion behavior of heat-treated zirconia was determined over the temperature range RT to 1350 C. The thermal expansion behavior of the ceramic interlayers, namely MASZ-67 and MASZ-80 interlayers, were only evaluated over the temperature range RT to 1200 C, since they were molten at temperatures above 1300 C. These interlayers indicated phase transformations over the temperature domain of interest.

Stress-Strain Behavior. The stress-strain behavior of adherents, bulk interlayer materials, and specimens containing joints were determined primarily at room temperature. The stress-strain curves of the constituent materials were then fit to a non-linear Ramberg-Osgood constitutive relation. Significant non-linear deformation was observed for the base zirconia material, NCI and braze metal. Even the ceramic interlayer materials indicated some non-linear deformation; for example, the proportional limit for MASZ-67 interlayer was only 63 percent of the material's bend strength. That is why inelastic fracture mechanics principles were used in the JAD model to account for the non-linear deformation response of the constituent materials.

Elevated temperature tests consisted of experiments performed at 200 C and 400 C for braze material and NCI, and at temperatures of 400 C, 750 C, and 1000 C for the zirconia material. These results, along with those available in the published literature were used for estimating the stress-strain response at intermediate temperatures.

Bend Strength. Bend strengths of base materials and joints were obtained from 4-point bend tests. Standard mechanics of material formulas for a homogeneous continuum were used to evaluate the bend strength of the joint from the geometry of the specimen and the failure load. Although this provided only the apparent bend strength of the joint, the error was estimated to be less than 8 percent, because the elastic modulus mismatch of the materials was small for both the zirconia/zirconia and zirconia/NCI systems, and also because of small differences in the thermal expansion coefficient of the constituent materials. Nevertheless, it is useful to keep this point in mind when assessing the joint strengths in the tables listed later in this section.

A sufficient number of specimens were tested to obtain the Weibull modulus of the base materials and joints. This parameter is useful for assessing the variability of data, with a high number indicating lower scatter in strength compared with a material with a low Weibull modulus value. The efficiency of the joint was obtained from the ratio of the average bend strength of the joint with respect to the strength of the base zirconia. In all cases, efficiency was less than unity with the worst efficiency observed for zirconia/zirconia joints fabricated using the MASZ-50 interlayer.

For zirconia/zirconia joints, it was observed that the preferred fracture path was the interlayer material. Because of this crack path, it was possible to relate the bend strength of zirconia/zirconia joints with the bend strength of the corresponding interlayer. For zirconia/NCI joints, failure occurred partly through the reaction zone of the zirconia and partly through the Ti-rich layer of the braze metal. Accordingly, it was not possible to relate the bend strength of the constituent bulk materials to that of the joint.

Fracture Toughness. The fracture toughnesses of base materials and joints were evaluated using notched bend bar and cracked disk specimens. Appropriate formulas were used to determine fracture toughness from the applied load and specimen geometry. The fracture toughness data of constituent materials (adherents and interlayers) were used as input to the JAD model. Mixed-mode fracture toughness data were used for evaluating the dependence of strain energy release rate on mode mixity, expressed in terms of the phase angle Ψ , $\tan^{-1}(Q_{II}/Q_I)^{(a)}$.

3.2.4 Test Conditions

Mechanical properties of adherents and interlayer materials, and the load-bearing capability of joints were determined at RT, 400 C and at 1000 C. However, most of the experiments were performed at room temperature. The 400 C tests were used for evaluating the performance of zirconia/NCI joints at that temperature. 1000 C tests were used for evaluating the performance of zirconia/zirconia joints at that temperature. All tests were performed in air.

3.2.5 Microstructure

The microstructures of zirconia/zirconia and zirconia/NCI joints were observed using optical and scanning electron microscopy (SEM) techniques. Chemical analysis, using wavelength dispersive spectroscopy (WDS) on a JEOL 733 microprobe, was performed to determine the chemical species responsible for good wetting between interlayer and adherents, and also for evaluating any undesirable reaction product that may have formed as a result of the joining process. In addition, limited X-ray diffraction was performed on the MASZ interlayer materials to understand the source of R-curve behavior that was observed for notched zirconia/zirconia joint specimens. Details of some of the microstructures obtained for zirconia/zirconia joints, fabricated either with a CTS interlayer or an MASZ interlayer are provided in the papers^[3, 4, 5] in Appendix B.

Most of the microstructural examinations were performed at the beginning of this program, while developing appropriate bonding interlayers and joining conditions for reliable joints. During the validation stage, microstructural examination was limited to determining the thickness of the bonds. For zirconia/zirconia joints, interlayer thickness ranged between 100 and 150 μm , while for zirconia/NCI joints, the interlayer thickness ranged between 50 and 80 μm .

(a) For bimaterial systems, the counterpart to the monolithic fracture toughness parameter K is denoted by Q .

3.2.6 Fractography

The fracture surfaces of representative specimens were examined optically and by using SEM. Limited energy dispersive X-ray analysis (EDS) was conducted to obtain estimates of the chemistry in different regions of the fracture surface. Such observations and measurements helped in identifying the preferred fracture path, namely, whether a crack propagated through the adherents, or through the bonding interlayer, or through the interface between the interlayer and adherent. Additional confirmation of the fracture path was obtained by sectioning fractured samples perpendicular to the fracture surface, metallographically polishing those sections, and observing such polished sections using optical and scanning electron microscopy techniques.

3.2.7 Preliminary Analysis of Data

A preliminary analysis of the data included the following:

1. Reducing the load-versus-displacement data and load-versus-strain-gage data to determine the stress-strain behavior of the constituent materials. All the constituent materials showed some degree of non-linearity at room temperature, and the stress-strain results were mathematically expressed using the elastic-plastic Ramberg-Osgood relation:

$$\epsilon/\epsilon_o = (\sigma/\sigma_o) + D (\sigma/\sigma_o)^n$$

where σ denotes stress, ϵ denotes strain, σ_o yield stress, ϵ_o yield strain, σ_o/E represents the elastic modulus, E , and D is a constant.

2. Determination of apparent bend strengths of the joints from the load at failure, using the standard bend-bar formula for a homogeneous material. Although such a procedure neglects residual stresses from thermal expansion mismatch and shielding stresses from modulus mismatch, it provides a basis for comparing the strength of the joint with respect to the strength of the base ceramic. This is similar to the JAD model procedure used for notched specimens, where the distance measured, in the direction of the joint assessment vector, from the origin to the failure envelope is compared with the reference load, which is the load required to break a monolithic ceramic specimen.

The bend strengths of constituent materials and joints were expressed in the form of Weibull plots. The data were arranged in increasing values of strength, and the probability of failure was expressed through the relation:

$$P = i/(N + 1)$$

where i is the rank ($i = 1$ corresponding to the lowest strength), and N is the number of samples tested. The Weibull modulus was obtained by plotting $\ln(1/(1-P))$ versus strength on a log-log plot, and determining the slope of the best fit straight line.

Additional analytical efforts associated with bend strengths of unnotched specimens involved predicting the strength of joints from the strength of the bulk interlayer material. This procedure was feasible for zirconia/zirconia joints because it was observed that failure occurred preferentially in the interlayer. The predictive effort had to incorporate shielding effects, since the modulus of the interlayer was consistently less than the surrounding zirconia, thereby reducing the local stress at the failure location with respect to the far-field applied stress. These shielding effects are discussed in the attached paper^[6]. In the case of zirconia/NCI joints, such a predictive procedure could not be used because failure occurred partly through the reaction zone of the zirconia and partly through the Ti-rich zone of the braze metal. In this case, a predictive methodology for bend strength would require measurement of the bend strength of the reaction zone. Although this could be performed in principle, by depositing a Ti-film on the surface of an as-received zirconia bend bar, and then subjecting the sample to a 750 C heat treatment, such experiments were not performed in this program.

3. Evaluating the fracture toughness of notched bend bars using the following LEFM formula for a homogeneous material:

$$K_I = 6MY(a/W)\sqrt{a} / BW^2$$

where K_I is the mode I stress intensity factor, M is the magnitude of a pure bending moment, a is the crack length, W is the beam depth, B is the beam thickness, and Y is a function of a/W given by:

$$Y(a/W) = 1.99 - 2.47(a/W) + 12.97(a/W)^2 - 23.17(a/W)^3 + 24.80(a/W)^4$$

The fracture toughness of constituent materials and the apparent fracture toughness of joints were calculated using the above formulas. Of course, the apparent fracture toughness of joints is denoted by Q_I . The reason that the above formulas provide only apparent fracture toughness of joints is that the formulas do not take into account the elastic and thermal mismatch of the constituents, although the latter was small for the constituent materials considered in this investigation. Nevertheless the apparent toughness values provide an approximate means for comparing the fracture toughness of joints with those of the constituent materials, and are tabulated in Section 3.3. It may be noted, in this context, that the local Q of zirconia/zirconia joints, fabricated with MASZ 67 interlayer material, at failure locations inside the interlayer were calculated to be approximately 68 percent of the apparent Q value; for joints

fabricated with MASZ-80 interlayer material, the local Q was approximately 90 percent of the apparent Q value.

The apparent mixed-mode stress intensity factors for zirconia/zirconia joints were obtained using Atkinson's solution^[7] for a cracked disk specimen of a homogeneous material, with the crack oriented at various angles to the compression load line. For the zirconia/NCI disk specimens, the current FEM results for a bimaterial disk specimen provided by Professor C. F. Shih were used for determining the Mode I-Mode II stress intensity factors; Atkinson's analytical results were within 5 percent of the current FEM values, primarily because the modulus mismatch between zirconia and NCI was quite small. The apparent mixed-mode data were reduced using the current analytical results to compute the local Q_I and Q_{II} values at failure. These local Q values were then plotted in the form of G versus mode mixity, (Ψ), ($\Psi = \tan^{-1}(Q_{II}/Q_I)$). Such plots were helpful in developing and assessing an appropriate function which related strain energy release rate to Ψ . Currently there does not exist a universal functional form of G versus Ψ , and the form suggested by Evans and Hutchinson^[8] $G(\Psi) = G_0 \text{Sec}^2 \Psi$, had to be modified based on data from monolithic ceramics which suggested that the form $G(\Psi) = G_0 \text{Sec}^2(\Psi/\Psi_0)$, where Ψ_0 is a constant, was more appropriate.

3.3 TEST RESULTS

The presentation of the test results is divided into three sections:

1. Results of mechanical and thermal property measurements that were used for generating baseline data for constituent material properties. These results were used as input data to the FEM code and JAD model.
2. Results of mechanical tests on unnotched and notched bend bars that were used as part of preliminary validation of the JAD model.
3. Results of mechanical tests on cracked disk specimens that were used as part of the final validation of the JAD model.

3.3.1 Properties of Constituent Materials

Thermal Expansion. Figure 21 provides the thermal expansion trace of the base zirconia material over the temperature range 100 C to 1300 C; measurements were made over a gage length of 38.1 mm. The expansion trace provided an average thermal expansion (α_{th}) coefficient of approximately $10.3 \times 10^{-6}/C$, and this value was used in the analysis of zirconia/zirconia joints. Figure 22 is a thermal expansion trace of the base zirconia over a narrower temperature range, RT to 730 C. The plot shows minimum hysteresis, and here too the average thermal expansion coefficient is $10.3 \times 10^{-6}/C$. For NCI an α_{th} of $14.9 \times 10^{-6}/C$ was obtained. For the Incusil-ABA braze metal, a thermal expansion coefficient of $18.2 \times 10^{-6}/C$ was obtained from the material supplier.

The thermal expansion trace of bulk MASZ-67 interlayer material, that was used in zirconia/zirconia joining, is shown in Figure 23. In this case significant non-linear expansion may be observed; in particular, the volumetric expansion and contraction that are observed

in the temperature range 550 C to 900 C can be ascribed to a monoclinic to tetragonal (and vice versa) phase transformation mechanism most likely involving the retained zirconia particles in the interlayer material. A similar thermal expansion trace was obtained for the MASZ-80 interlayer. Because of the complexity of the thermal expansion trace, the actual data were used in the computation of residual stresses in the FEM model.

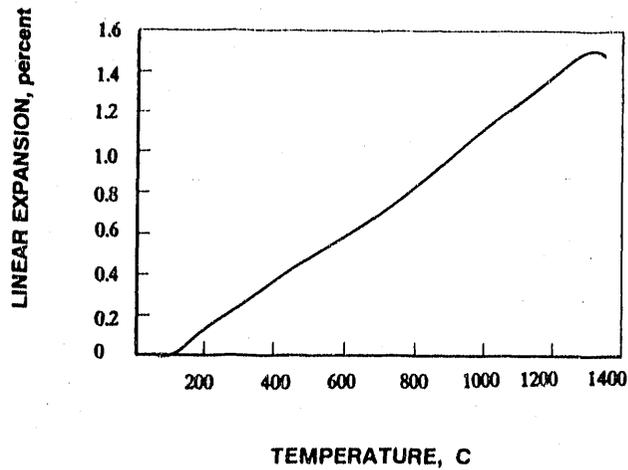


Figure 21. Thermal expansion trace of base zirconia from room temperature to 1300 C.

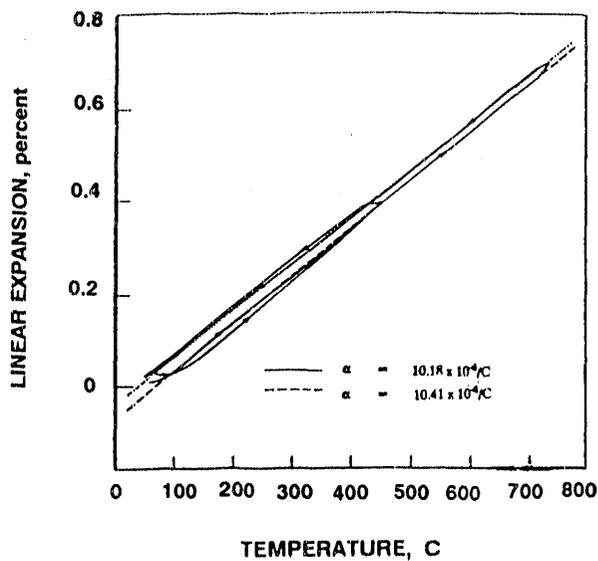


Figure 22. Thermal expansion of base zirconia material from room temperature to 730 C.

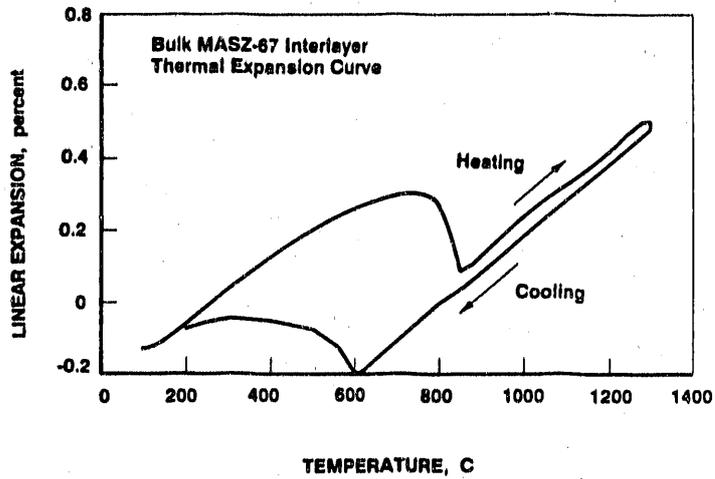


Figure 23. Thermal expansion of MASZ-67 material from room temperature to 1300 C.

For completeness, the thermal expansion trace of a zirconia/NCI joint is shown in Figure 24. The specimen was a bend bar of length 38.63 mm, and the expansion was measured in a direction perpendicular to the joint plane (braze metal thickness was approximately 70 μm). An average α_{th} of $11.84 \times 10^{-6}/\text{C}$ was obtained. This value may be compared with α_{th} of 10.3, 14.9, and 18.2 ppm/C for the zirconia, NCI, and braze metal, respectively.

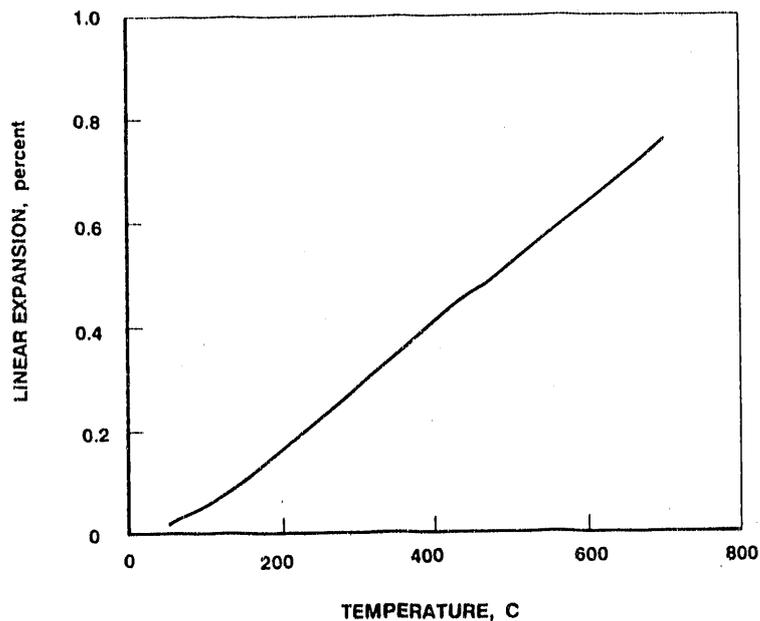


Figure 24. Thermal expansion of zirconia/NCI joint.

Stress-Strain Behavior of Constitutive Materials. Figures 25-30 illustrate the true stress-strain behavior of the following constituent materials at room temperature:

1. As-received zirconia (Figure 25),
2. Heat-treated zirconia, heat treated at 1350 C (Figure 26),
3. Nodular cast iron (Figure 27),
4. Incusil-ABA (Figure 28),
5. Bulk MASZ-67 ceramic interlayer, sintered at 1350 C (Figure 29),
6. Bulk MASZ-80 ceramic interlayer, sintered at 1350 C (Figure 30).

The true stress-strain data were fit using the Ramberg-Osgood relation given earlier in this section. Table 5 provides appropriate parameters used for fitting the stress-strain curves. The Poisson's ratios that are provided in the table were not measured but were obtained from the literature. The table also includes parameters for elevated temperature behavior. Among these latter results, the complete stress-strain behavior was experimentally measured at 400 C for the braze metal and NCI, and at 200 C for the braze metal; wire specimens were used in measuring the stress-strain property of braze metal, since thicker sections were not available. The stress-strain behavior of zirconia at elevated temperature was estimated based on the room temperature results confirmed with data available in the literature; only the elastic modulus and bend strength were measured for zirconia at temperatures of 400 C, 750 C and 1000 C.

The average bend strength of ceramic specimens are also tabulated in Table 5. Bend test results of individual specimens are provided in the master Table 6. Weibull moduli of constituent materials were determined, and they are summarized in a table provided later in this report.

In Tables 6 and 7 the individual specimens are identified by a key which indicates the billet, the specimen type, and location of specimen in the billet prior to machining. The first four characters of the specimen number correspond to the particular joined billet. Thus, a specimen number such as HP22-B13 is a specimen that was machined from joined billet number HP22. The letter identifiers B, F, UD, and CD indicate specimen type; bend bar, notched bend bar (fracture), uncracked disk, and notched disk. The last two digits of the specimen number provide an approximate idea of the specimen location within the billet, with the first digit corresponding to the row number and the second digit corresponding to the column number. This is illustrated in Figure 31, and, except for the as-received zirconia, bulk interlayer materials, and the HP22 block (where row numbering was started with 0 rather than 1), this convention was followed for all the specimens.

Fracture Toughness of Constituent Materials. The fracture toughness of constituent materials and apparent fracture toughness of joints are tabulated in Table 7. Table 8 is based on Table 7, and provides average Q_{Ic} and corresponding G_{Ic} values for the various constituent materials. In this table, the toughness of the reaction zone of zirconia/NCI joints was evaluated from a notched-bend bar test, where the crack propagated entirely through the reaction zone of the zirconia, away from the Ti-rich interface of the braze metal.

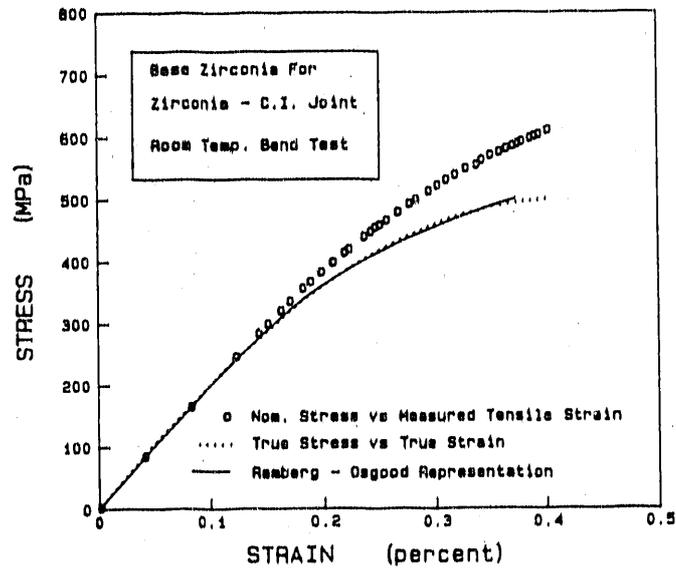


Figure 25. Room temperature stress-strain behavior of as-received zirconia.

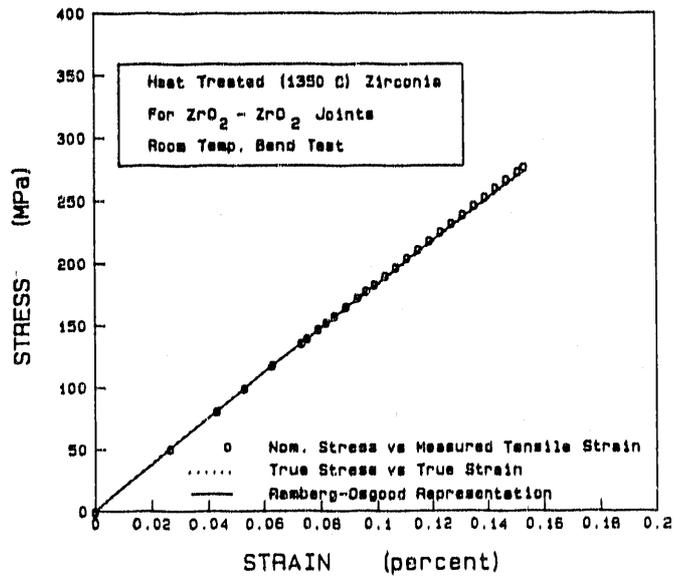


Figure 26. Room temperature stress-strain behavior of heat treated zirconia.

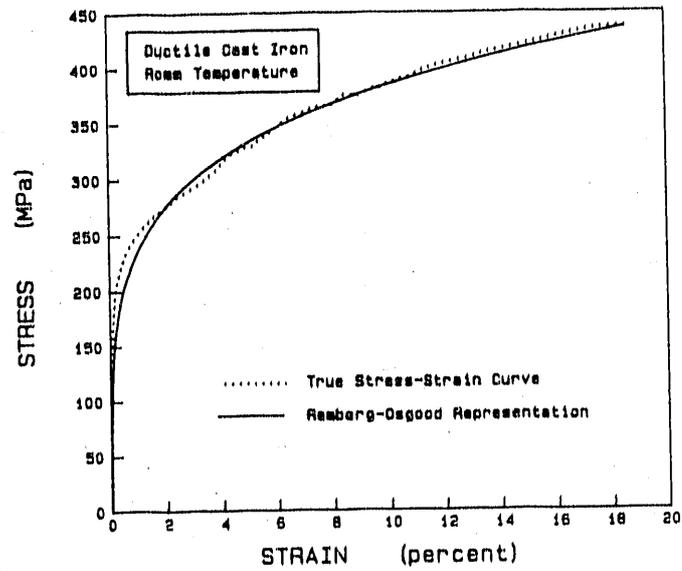


Figure 27. Room temperature stress-strain behavior of nodular cast iron.

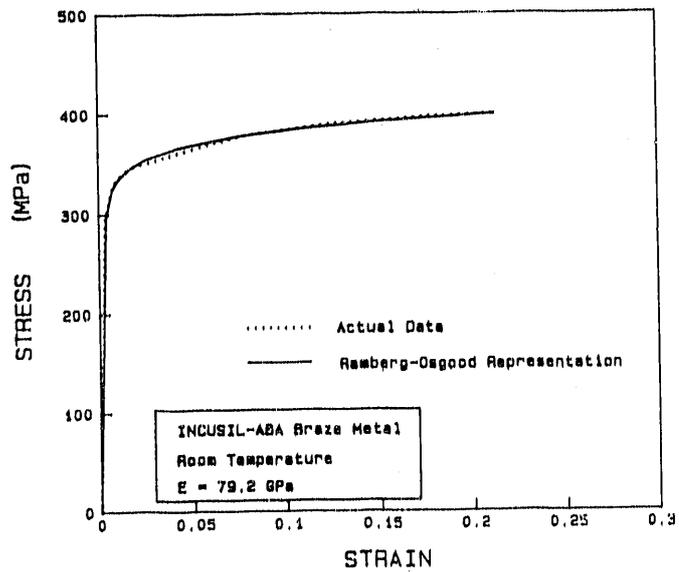


Figure 28. Room temperature stress-strain behavior of Incusil ABA braze metal.

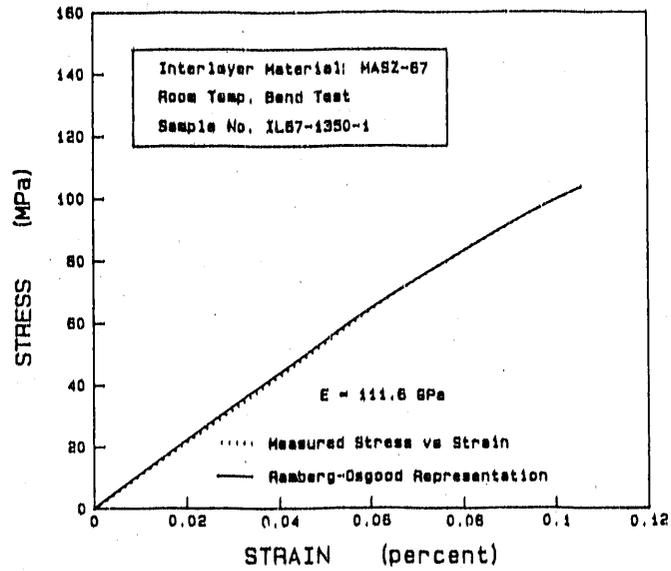


Figure 29. Room temperature stress-strain behavior of MASZ-67 material.

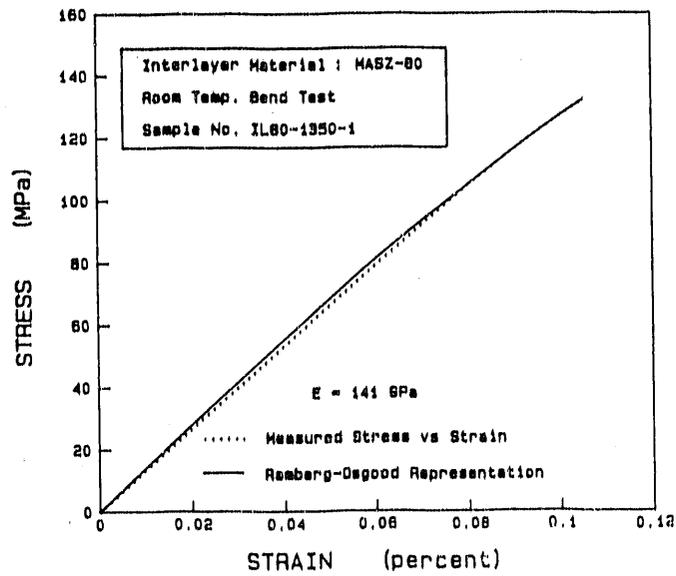


Figure 30. Room temperature stress-strain behavior of MASZ-80 material.

Table 5. Stress-strain parameters for constituent materials used in zirconia/zirconia and zirconia/NCI joining.

Constituent Material	σ_0 MPa	ϵ_0	E, GPa	Fracture Strength, MPa	n	α	Poisson's Ratio
Room Temperature Data							
As-Received Zirconia	301.8	0.000150	202.0	589	5.66	0.05	0.22
Heat-Treated Zirconia	140.6	0.000698	201.4	291	1.62	0.08	0.22
Bulk MASZ-67 Interlayer	74.0	0.000662	111.7	122	3.93	0.05	0.22
Bulk MASZ-80 Interlayer	101.7	0.000720	141.2	158	3.15	0.07	0.22
Nodular Cast Iron (NCI)	221.9	0.000136	163.4	437	4.99	4.61	0.28
Incusil-ABA Braze	295.6	0.000373	079.3	402	19.58	0.15	0.36
Elevated Temperature Data							
NCI, 260 C (a)	199.2	0.001245	160.0	431	7.50	2.50	0.28
NCI, 400 C	159.2	0.001010	157.5	276	10.00	0.06	0.28
NCI, 538 C (a)	122.3	0.000870	140.0	240	10.00	0.06	0.28
NCI, 607 C (a)	90.6	0.000686	132.0	200	10.00	0.06	0.28
NCI, 716 C (a)	45.3	0.000390	116.0	170	10.00	0.06	0.28
Incusil-ABA, 200 C	362.7	0.007510	048.2	414	74.20	0.00	0.28
Incusil-ABA, 400 C	101.4	0.007340	013.8	53.4	109.00	0.08	0.28
As-received ZrO ₂ , 200 C (b)	269.5	0.001368	197.0	483	5.70	0.05	0.22
As-received ZrO ₂ , 400 C (b)	232.3	0.001213	191.4 (c)	453 (c)	5.70	0.05	0.22
As-received ZrO ₂ , 600 C (b)	195.1	0.001063	183.5	381	5.70	0.05	0.22
As-received ZrO ₂ , 800 C (b)	158.0	0.000910	173.2	308	5.70	0.05	0.22
As-received ZrO ₂ , 1000 C (b)	120.8	0.000753	160.5 (d)	236 (d)	5.70	0.05	0.22
As-received ZrO ₂ , 1200 C (b)	83.6	0.000575	145.4	163	5.70	0.05	0.22
As-received ZrO ₂ , 1400 C (b)	46.5	0.000363	127.9	91	5.70	0.05	0.22

(a) E and sigma-0 estimated from Structural Alloys Handbook (H88), modified to account for current room temperature results. Rest of terms are best estimates.

[H88]: Structural Alloys Handbook, Vol. 1, Ed. J.B. Hallowell, MCIC, Battelle (1988)

(b) Estimates, based on current data and data in the literature, particularly, "Engineering Property Data on Selected Ceramics, Vol. 3, Single Oxides", MCIC Report No. MCIC-HB-07, Battelle Memorial Institute, Columbus, Ohio (1982)

(c) Measured modulus and bend strength were 176.5 GPa and 249 MPa respectively.

(d) Measured modulus and bend strength were 142 GPa and 225 MPa, respectively.

Table 6. Bend test results of constituent materials and joints.

Specimen Number	Temperature C	Width, B mm	Height, W mm	Load, P1 N	Strength* MPa
As-Received Zirconia					
BZ-B0	25	5.01	2.55	983.5	575
BZ-B2	25	4.98	2.56	1041.3	610
BZ-B7	25	5.01	2.56	1001.3	581
BZ-B8	25	5.03	2.55	1014.6	593
BZ-B4	400	5.00	2.56	235.9	249
BZ-B5	750	5.09	2.54	224.7	237
BZ-B3	1000	4.98	2.56	206.9	220
BZ-B9	1000	5.08	2.56	220.3	230
Zirconia/Zirconia Joint With MASZ-67 Interlayer, 1350 C Processed					
HP22-B01	25	5.87	3.08	360.5	123
HP22-B02	25	5.87	3.02	387.2	138
HP22-B03	25	5.86	3.04	369.4	130
HP22-B04	25	5.87	2.95	369.4	138
HP22-B05	25	5.85	2.90	275.9	107
HP22-B11	25	5.32	2.72	222.5	108
HP22-B12	25	5.51	2.72	231.4	108
HP22-B13	25	5.59	2.73	233.8	107
HP22-B14	25	5.36	2.73	264.8	126
HP22-B15	25	5.52	2.73	267.0	123
HP22-B16	25	5.30	2.73	244.8	118
HP22-B21	25	5.15	2.56	214.0	121
HP22-B23	25	5.15	2.56	242.5	137
HP22-B25	25	5.15	2.56	182.5	103
HP22-B26	25	5.15	2.56	264.8	149
HP22-B27	25	5.15	2.56	174.4	98
HP22-B28	25	5.13	2.55	244.8	140
HP23-B22	25	5.08	2.51	213.6	127
HP23-B11	1000	5.06	2.55	58.7	62
HP24-B11	1000	5.09	2.55	51.6	54
HP24-B13	1000	5.08	2.55	55.2	58
HP24-B14	1000	5.07	2.52	62.3	67
HP24-B15	1000	5.09	2.56	60.1	63
Zirconia/Zirconia Joint With MASZ-80 Interlayer, 1350 C Processed					
HP25-B11	25	5.09	2.53	240.3	140
HP25-B12	25	5.08	2.56	284.8	163
HP25-B13	25	5.08	2.55	204.7	118
HP25-B17	25	5.09	2.56	262.6	151
HP25-B21	25	5.08	2.52	329.3	194
HP25-B22	25	5.08	2.55	293.7	170
HP25-B28	25	5.08	2.55	195.8	113
HP25-B23	25	5.08	2.52	329.3	194
HP25-B29	25	5.07	2.55	316.0	182
HP25-B24	1000	5.08	2.55	71.6	75
HP25-B15	1000	5.06	2.55	75.7	80
HP25-B18	1000	5.07	2.55	82.3	87

* For joints, these strengths are apparent bend strengths.

Table 6. (continued)

Specimen Number	Temperature C	Width, B mm	Height, W mm	Load, Pf N	Strength* MPa
Zirconia/Zirconia Joint With MASZ-50 Interlayer, 1350 C Processed					
HP27-B13	25	5.07	2.54	93.5	54
HP27-B14	25	5.07	2.54	89.0	52
HP27-B17	25	5.07	2.54	89.0	52
HP27-B21	25	5.09	2.55	77.0	45
HP27-B22	25	5.09	2.54	102.4	59
Base Zirconia, Heat-Treated at 1350 C to Simulate Fabrication of Zirconia/Zirconia Joints					
HP28-B11	25	5.08	2.58	449.5	258
HP28-B12	25	5.07	2.56	491.7	282
HP28-B14	25	5.07	2.55	494.0	286
HP28-B17	25	5.07	2.57	520.7	296
HP28-B19	25	5.07	2.56	498.4	285
HP28-B21	25	5.08	2.57	542.9	309
HP28-B22	25	5.10	2.57	560.7	318
HP28-B23	25	5.09	2.57	525.1	298
HP28-B24	1000	5.09	2.57	212.7	220
HP28-B26	1000	5.07	2.55	195.6	206
Zirconia/Zirconia Joint With MASZ-80 Interlayer, 1400 C Processed					
HP29-B11	25	5.06	2.53	173.6	102
HP29-B11	25	5.06	2.53	182.5	107
HP29-B13	25	5.06	2.53	186.9	109
HP29-B14	25	5.06	2.53	160.2	94
HP29-B21	25	5.09	2.53	204.7	119
HP29-B22	25	5.09	2.53	191.4	112
HP29-B23	25	5.09	2.54	184.7	107
HP29-B24	25	5.09	2.53	173.6	101
HP29-B25	25	5.09	2.53	178.0	104
Zirconia/Zirconia Joint With MASZ-97 Interlayer, 1400 C Processed					
HP30-B11	25	5.09	2.52	104.6	62
HP30-B12	25	5.09	2.53	93.5	55
HP30-B14	25	5.09	2.53	93.5	55
HP30-B15	25	5.09	2.53	111.3	65
HP30-B23	25	5.09	2.54	102.4	59
HP30-B24	25	5.10	2.53	135.7	79
HP30-B25	25	5.10	2.53	102.4	60
Bulk MASZ-50 Interlayer Material, 1350 C Processed					
IL50-1350-1	25	5.08	2.52	104.1	62
IL50-1350-2	25	5.06	2.53	97.9	58
Bulk MASZ-97 Interlayer Material, 1350 C Processed					
IL67-1350-1	25	5.06	2.54	173.6	101
IL67-1350-2	25	5.07	2.55	164.7	95

* For joints, these strengths are apparent bend strengths.

Table 6. (continued)

Specimen Number	Temperature C	Width, B mm	Height, W mm	Load, P _f N	Strength* MPa
IL67-1350-3	25	5.07	2.55	158.0	92
IL67-1350-4	25	5.03	2.55	160.2	93
IL67-1350-5	25	5.01	2.54	160.2	94
IL67-1350-6	25	5.06	2.54	142.4	83
IL67-1350-7	1000	5.07	2.55	65.4	69
IL67-1350-8	1000	5.07	2.54	65.9	70
Bulk MASZ-67 Interlayer Material, 1400 C Processed					
IL67-1400-1	25	5.09	2.55	124.6	72
IL67-1400-2	25	5.09	2.55	142.4	82
IL67-1400-3	25	5.07	2.55	138.0	80
IL67-1400-4	25	5.08	2.55	129.8	73
IL67-1400-5	25	5.09	2.52	135.7	80
IL67-1400-6	25	5.09	2.55	142.4	82
Bulk MASZ-90 Interlayer Material, 1350 C Processed					
IL80-1350-1	25	5.09	2.51	218.1	130
IL80-1350-2	25	5.07	2.52	224.7	133
IL80-1350-3	25	5.07	2.52	235.9	139
IL80-1350-4	25	5.08	2.55	211.4	122
IL80-1350-5	25	5.07	2.53	234.5	138
IL80-1350-6	25	5.07	2.54	249.2	145
IL80-1350-8	1000	5.07	2.54	65.0	69
Bulk MASZ-90 Interlayer Material, 1400 C Processed					
IL80-1400-1	25	5.05	2.54	191.4	112
IL80-1400-2	25	5.06	2.53	213.8	125
IL80-1400-3	1000	5.04	2.54	65.0	69
Zirconia/Nodular Cast Iron Joint					
LMC3-B21	25	5.08	2.57	471.7	267
LMC3-B22	25	5.09	2.34	309.3	212
LMC3-B23	25	5.08	2.54	427.2	248
LMC3-B24	25	5.09	2.53	316.0	185
LMC3-B27	25	5.09	2.52	316.0	210
LMC3-B31	25	5.06	2.56	299.3	166
LMC3-B32	25	5.07	2.56	373.8	215
LMC3-B33	25	5.07	2.56	471.7	272
LMC3-B34	25	5.06	2.56	445.0	256
LMC4-B11	25	5.09	2.34	311.5	214
LMC4-B12	25	5.76	3.28	1061.0	328
LMC4-B21	25	5.07	2.57	371.6	211
LMC4-B22	25	5.07	2.57	342.7	195
LMC4-B24	25	5.07	2.57	391.6	223
LMC4-B28	25	5.07	2.57	467.3	267
LMC4-B31	25	5.09	2.51	331.5	196
LMC4-B32	25	5.10	2.51	493.4	295
LMC4-B33	25	5.09	2.52	252.8	149
LMC4-B36	25	5.10	2.51	369.4	218
LMC5-B13	25	5.08	2.50	218.1	130

* For joints, these strengths are apparent bend strengths.

Table 6. (continued)

Specimen Number	Temperature C	Width, B mm	Height, W mm	Load, Pf N	Strength* MPa
LMC5-B14	25	5.08	2.50	436.1	261
LMC5-B25	25	5.08	2.51	319.5	191
LMC3-B35	400 C	5.07	2.56	104.1	109
LMC5-B12	400 C	5.09	2.55	97.0	102
LMC5-B16	400 C	5.07	2.55	100.1	105

* For joints, these strengths are apparent bend strengths.

Table 7. Fracture toughness test results of constituent materials and joints.

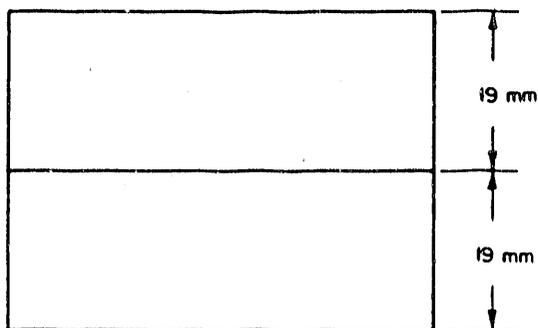
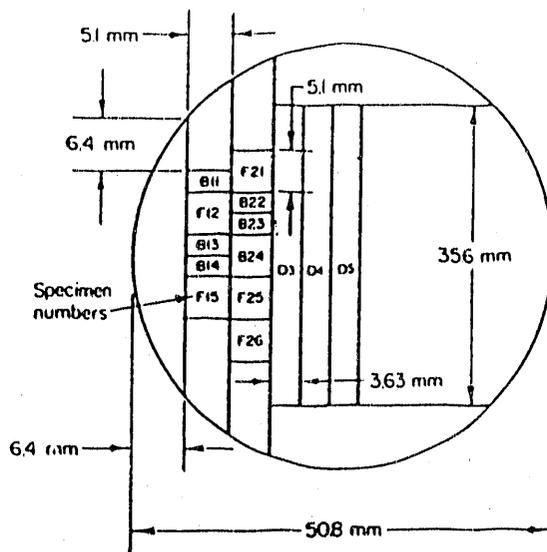
Specimen Number	Temperature, C	Width, B mm	Height, W mm	Crack Length, a mm	Fracture Load, Pf, N	Y(a/W)	Q1c*, MPa√m
Zirconia/Zirconia Joint With MASZ-67 Interlayer, 1350 C Processed							
HP22-F15	RT	2.56	5.14	1.53	140.2	1.99	3.07
HP23-F18	RT	5.08	5.10	1.54	209.2	2.00	2.36
HP23-F27	RT	5.09	5.09	1.54	213.6	2.00	2.42
HP24-F15	RT	5.08	5.07	1.63	173.6	2.03	2.07
HP24-F14	1000	5.08	5.09	1.57	80.5	2.01	1.69
HP24-F23	1000	5.10	5.07	1.56	69.9	2.01	1.46
Zirconia/Zirconia Joint With MASZ-80 Interlayer, 1350 C Processed							
HP25-F16	RT	5.09	5.09	1.63	264.8	2.03	3.14
HP25-F26	RT	5.08	5.07	1.63	268.3	2.03	3.19
HP25-F14	1000	5.09	5.07	1.61	82.3	2.02	1.77
Zirconia/Zirconia Joint With MASZ-67 Interlayer, 1400 C Processed							
HP30-F16	RT	5.09	5.08	1.63	107.7	2.03	1.28
HP30-F27	RT	5.09	5.08	1.63	160.2	2.03	1.90
Zirconia/Zirconia Joint With MASZ-80 Interlayer, 1400 C Processed							
HP29-F26	RT	5.09	5.09	1.60	195.8	2.02	2.28
HP29-F27	RT	5.06	5.10	1.63	200.3	2.03	2.37
Base Zirconia, Heat Treated at 1350 C to Simulate Fabrication of Zirconia-Zirconia Joints							
HP26-F16	RT	5.10	5.08	1.71	458.8	2.06	5.67
HP26-F21	RT	5.05	5.08	1.63	458.4	2.03	5.47
Bulk MASZ-50 Material, 1350 C Processed							
IL50-1350-F1	RT	5.07	5.05	1.71	122.4	2.07	1.54

* Q1c=K1c for monolithic materials, and Q1c corresponds to apparent Mode I fracture toughness for joints.

Table 7. (continued)

Specimen Number	Temperature, C	Width, B mm	Height, W mm	Crack Length, a mm	Fracture Load, P _f , N	Y(a/W)	Q1c*, MPa√m
Bulk MASZ-67 Interlayer, 1350 C Processed							
IL67-1350-F1	RT	5.09	5.07	1.70	169.1	2.06	2.10
IL67-1350-F3	RT	5.11	5.05	1.70	160.2	2.06	1.99
IL67-1350-F2	1000	5.10	5.07	1.60	68.1	2.02	1.46
Bulk MASZ-80 Interlayer, 1350 C Processed							
IL80-1350-F1	RT	5.12	5.08	1.71	235.9	2.06	2.89
IL80-1350-F3	RT	5.12	5.09	1.65	231.4	2.04	2.75
IL80-1350-F2	1000	5.09	5.09	1.63	73.9	2.03	1.59
Bulk MASZ-67 Interlayer, 1400 C Processed							
IL67-1400-F1	RT	5.12	5.11	1.63	146.9	2.02	1.71
IL67-1400-F2	RT	5.09	5.08	1.63	149.1	2.03	1.77
Bulk MASZ-80 Interlayer, 1400 C Processed							
IL80-1400-F1	RT	5.09	5.09	1.63	445.0	2.03	5.27
Zirconia/ Nodular Cast Iron Joint							
LMC2-F25	RT	5.00	5.09	1.17	529.6	1.89	5.03
LMC3-F25	RT	5.07	5.11	1.63	445.0	2.03	5.23
LMC3-F36	RT	5.12	5.11	1.63	429.4	2.02	5.00
LMC4-F25	RT	5.11	5.07	1.63	600.8	2.03	7.12
LMC4-F26	RT	5.07	5.08	1.60	547.4	2.02	8.42
LMC4-F34	RT	5.09	5.09	1.63	534.0	2.03	6.31
LMC5-F18	RT	5.09	5.09	1.63	422.8	2.03	5.00
LMC5-F26	RT	5.08	5.09	1.60	596.3	2.02	6.97

* Q1c=K1c for monolithic materials, and Q1c corresponds to apparent Mode I fracture toughness for joints.



- B:** Uncracked bend bar, 2.54 x 5.08 x 38.1 mm
F: Notched bend bar, 5.08 x 5.08 x 38.1 mm
D: Cracked or uncracked disk specimen, 35.6 mm dia. x 3.63 mm thick

Figure 31. Source of specimens machined from the joined 2-inch billets.

Table 8. Fracture toughness test results of constituent materials.

Constituent Material	E, GPa	Poisson's Ratio	K _{Ic} , MPa√m	G _{Ic} , N/m	Avg. Approx. Th. Exp. Coeff., x10 ⁻⁶ /C
Room Temperature					
As-received zirconia	202.0	0.22	9.2	394.4	10.3
Heat-treated zirconia	201.4	0.22	5.6	146.6	13.1
Nodular cast iron	163.4	0.28	85.0	40750.1	14.9
Incusil ABA	79.3	0.36	30.0	9878.4	18.2
Bulk MASZ-50, 1350 C processed	109.0	0.22	1.5	20.4	9.5
Bulk MASZ-67, 1350 C processed	111.7	0.22	2.1	35.8	9.5
Bulk MASZ-80, 1350 C processed	141.2	0.22	2.8	53.6	9.5
Bulk MASZ-67, 1400 C processed*	111.7	0.22	1.7	25.8	10.0
Bulk MASZ-80, 1400 C processed*	141.2	0.22	5.3	187.2	10.0
Reaction zone of zirconia (in zirconia/NCI joints)**	202.0	0.22	7.1	237.5	10.3
1000 C					
Bulk MASZ-67, 1350 C processed***	71.7	0.22	1.5	28.3	9.5
Bulk MASZ-80, 1400 C processed*	90.6	0.22	1.6	26.6	9.5

* E and Poisson's ratio are only estimates, based on present data. K_{Ic} actually measured.

** Data obtained by testing a notched bend bar containing a zirconia/NCI joint, where the crack propagated only in the reaction zone of the zirconia.

*** Poisson's ratio was estimated.

3.3.2 Preliminary Validation Results

Bend Test Results. Table 6 provides bend test results of constituent materials as well as zirconia/zirconia and zirconia/NCI joints. No significant correlation was found between the specimen strength and its location within the joined billet, suggesting uniformity of bonding, and small residual stress effects (after machining) at least at a distance of 6.5 mm from the billet periphery.

Table 6 includes data generated at room temperature as well as those generated at elevated temperatures; namely, 400 C for zirconia/NCI joints and 1000 C for zirconia/zirconia joints. Test results obtained with different processing temperatures, namely 1350 C and 1400 C for zirconia/zirconia joints, are also included in the table.

Figure 32 is a Weibull plot which provides a comparison of the room temperature bend strengths of zirconia/zirconia joints fabricated using MASZ-67 interlayer with those of the constituent materials. Although the average strength of the joint is less than that of the heat-treated zirconia, it is higher than the strength of the bulk MASZ-67 interlayer. Nevertheless, failure of zirconia/zirconia joints occurred primarily in the ceramic interlayer. As shown in reference^[6], this behavior can be rationalized in terms of shielding of the joint interlayer by the surrounding higher modulus zirconia adherent.

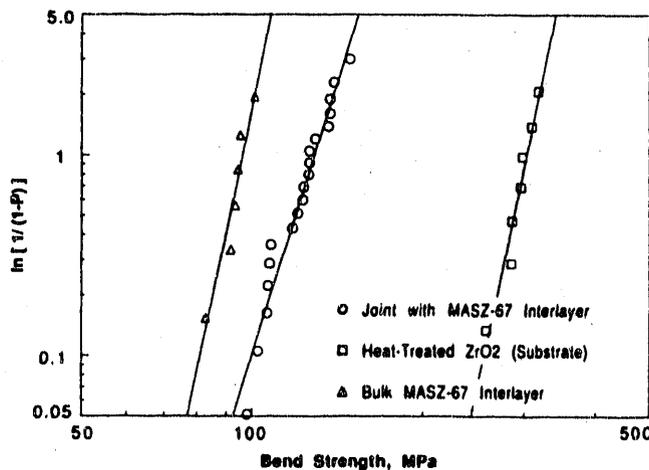


Figure 32. Weibull plot showing comparison of strength of zirconia/zirconia joints and bulk constituent materials.

Figure 33 is a Weibull plot comparing bend strengths of various zirconia/zirconia joints with their corresponding interlayer materials. The actual data points are not included in the plot for the purpose of clarity. This plot indicates that for both the MASZ-67 and MASZ-80 interlayer systems, joints perform better than their corresponding interlayer materials.

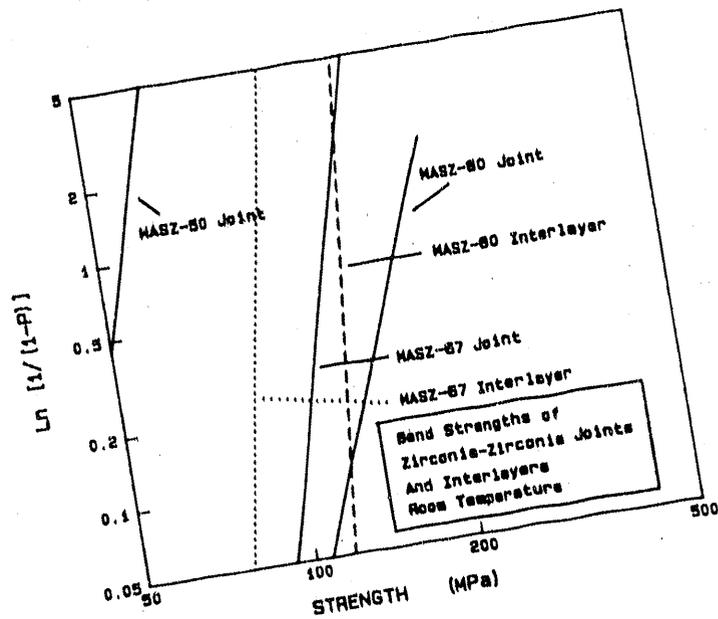


Figure 33. Weibull plot showing comparison of zirconia/zirconia joints and constituent materials as a function of percent zirconia powder in interlayer.

Figure 34 is a Weibull plot of the bend strength of zirconia/NCI joints. The average strength of the joint was 223 MPa compared with an average strength of 589 MPa for the as-received zirconia. A major reason for the strength degradation was the formation of the reaction zone in the zirconia due to oxygen depletion of the Ti-rich interface. As has been noted above, the crack path was partly through the reaction zone and partly through the Ti-rich interface with approximate percentage areas of each ranging between 40 and 60 percent. Fracture toughness measurements, to be discussed later, indicated that the fracture location had lower toughness (6 to 7 MPa√m) than the base zirconia (approximately 9.2 MPa√m). However, this difference cannot account for the large decrease in strength for the joined bend bar. Rather, it is believed that the joining process introduced defects primarily at the braze/zirconia interface, and these larger defects acted to reduce the strength of the joint. This argument is qualitatively in agreement with the observation that the Weibull modulus of the joint was 5.4, while the base zirconia typically has a Weibull modulus of 9.5; in fact, our limited data on the as-received zirconia provide an approximate Weibull modulus of 8.6, while the heat-treated zirconia had a Weibull modulus of 13.4, indicating a rather tight flaw population, i.e., small scatter in flaw size.

Bend strengths of constituent materials and joints are summarized in Table 9. The efficiency of the joints was calculated by dividing the strength of the joint by the strength of the adherent, and they are also tabulated in Table 9.

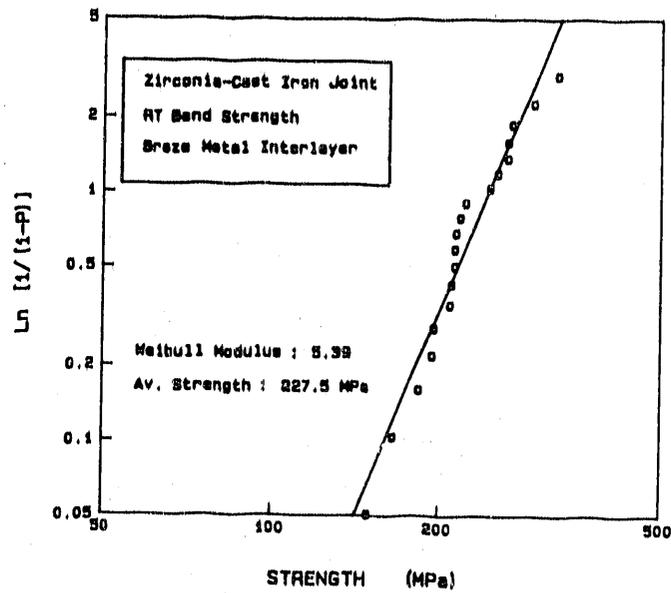


Figure 34. Weibull plot for zirconia/NCI joints.

Table 9. Average bend strength of zirconia/zirconia and zirconia/NCI joints.

Material	Bulk Material		Joints		
	Average Strength, MPa	Weibull Modulus	Average Strength, MPa	Weibull Modulus	Efficiency*, percent
Heat-Treated ZrO ₂ Substrate	293	13.4	-	-	-
MASZ - 80	134	20.8	158	6.0	54
MASZ - 67	93	13.6	122	9.0	42
MASZ - 50	59	-	52	-	18
Zirconia/NCI	-	-	223	5.4	38**

* Joint Efficiency = Strength of Joint / Strength of Substrate

** Efficiency of zirconia/NCI joint based on strength of 589 MPa for as-received zirconia.

The load versus displacement data for a joint fabricated with MASZ-67 interlayer, and those of the constituent materials are illustrated in Figure 35. As expected, the load versus displacement plot for the joint agrees very well with that of the heat-treated zirconia, since the thickness of the interlayer was negligible compared with the adherent. Figure 36 is a similar plot for a zirconia/NCI joint. This plot indicates significant non-linearity before fracture and the observed non-linearity is consistent with the fact that the specimen failed at a stress of 261 MPa, while the flow stress of zirconia was only 221.9 MPa. FEM calculations were not performed to check whether there was agreement between the observed load-displacement behavior and calculated elastic-plastic results.

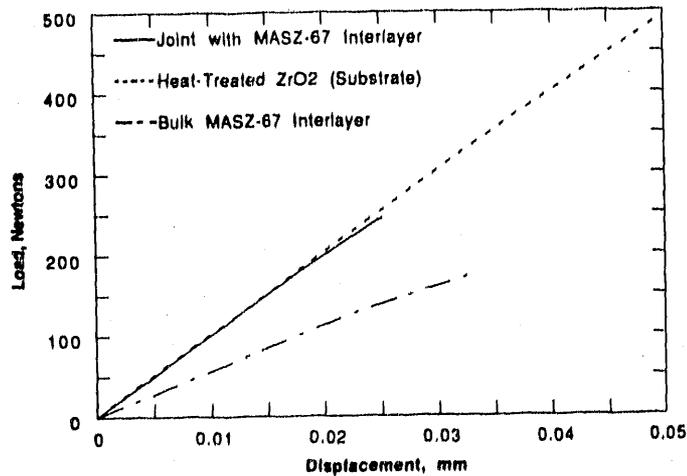


Figure 35. Load versus displacement plots for bend bars fabricated with MASZ-67 interlayer and its constituent materials.

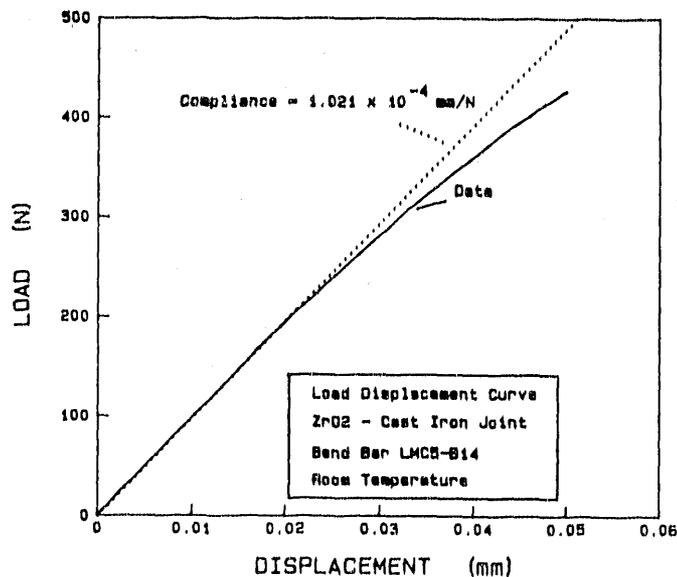


Figure 36. Load versus displacement plot for a zirconia/NCI joint.

Notched Bend Bars and Disk Specimens. The apparent fracture toughnesses corresponding to notched bend bars with joints have already been presented in Table 7. Table 10 provides comparisons of average fracture toughness results of zirconia/zirconia joints, and it has been arranged to show a comparison of the joint apparent toughness with that of the corresponding interlayer material. Similar to unnotched bend bars, there is a slight increase in the toughnesses of zirconia/zirconia joints (fabricated at 1350 C) compared with those of the corresponding interlayer materials. Although the higher toughness of the joint compared with the bulk interlayer can partly be explained^[6] by a shielding effect, it appears that there was degradation of the interlayer material for zirconia/zirconia joints fabricated with MASZ-67 interlayer. For this material system, the apparent toughness of the joint was 2.5 MPa√m, and analytical results indicate that this would correspond to a local stress intensity of 1.7 MPa√m at the interlayer location of the joint. On the other hand, the bulk interlayer had a toughness of 2.1 MPa√m; this would imply a 19 percent loss in toughness of the interlayer material due to the joining process. In the case of the MASZ-80 system, however, there did not appear to be any degradation in toughness of the interlayer material due to the joining process.

Table 10 indicates that the zirconia/zirconia joints fabricated at 1400 C behaved differently, in that joint toughnesses were lower than bulk interlayer toughnesses. This was particularly true for the MASZ-80 interlayer fabricated at 1400 C, which has potential as a good structural material by itself. The lower joint toughnesses compared with interlayers can probably be explained by the observation that the crack propagated primarily through the reaction zone of the zirconia; i.e., the reaction zone was the weakest link rather than the ceramic interlayer material. Use of the JAD model indicated that the measured failure load for those specimens would require a G_{I_0} that lay between 10 and 30 N/m for the reaction zone, and these G_{I_0} values would correspond to a toughness between 1.46 MPa√m and 2.1 MPa√m for the reaction zone of the base zirconia (significantly lower than 5.6 MPa√m for the heat-treated zirconia). Thus, 1400 C was obviously too high a temperature for zirconia/zirconia joining in that it caused intensive degradation of the base zirconia material.

For zirconia/NCI joints, the toughness of the joint was much less compared with either the base zirconia, NCI, or the braze metal. The explanation lies in the fact that the crack propagated partly through the reaction zone of the zirconia and partly through the Ti-rich interface at the braze-metal/zirconia interface. These locations were not available as bulk materials for independent toughness measurements. Because of this problem, it was recognized that the toughness of the braze metal or the base zirconia was not suitable for incorporation in the JAD model, because they did not represent local fracture energies. Therefore, an indirect approach was followed in that the load to failure of zirconia/NCI notched bend bars was used for back-calculating the local G_{I_0} at the failure location. It was understood that because of this approach, the JAD model could not utilize the results of notched bend bar tests, performed on zirconia/NCI joints, for validation purposes. However, the approach allowed for validation using the cracked disk specimen for zirconia/NCI joints. The fracture energy of the zirconia/NCI joint was determined as 184.5 N/m using the above procedure. This value was in good agreement with a test where failure in a notched zirconia/NCI specimen occurred entirely through the reaction zone of the zirconia; this particular specimen indicated a G_{I_0} of 237.5 N/m for the reaction zone.

Table 10. Average toughness values of constituent materials.

Material	Specimen Geometry	Q_{Ic}^* , MPa \sqrt{m}
Zirconia/Zirconia Joints		
Joint (MASZ-67, 1350 C)	Notched Bend Bar	2.48
Joint (MASZ-67, 1350 C)	Cracked Disk	2.12
Bulk MASZ-67, 1350 C	Notched Bend Bar	2.05
Joint (MASZ-80, 1350 C processed)	Notched Bend Bar	3.17
Joint (MASZ-80, 1350 C processed)	Cracked Disk	2.46
Bulk MASZ-80, 1350 C processed	Notched Bend Bar	2.82
Joint (MASZ-67, 1400 C processed)	Notched Bend Bar	1.59
Bulk MASZ-67, 1400 C processed	Notched Bend Bar	1.74
Joint (MASZ-80, 1400 C processed)	Notched Bend Bar	2.33
Bulk MASZ-80, 1400 C processed	Notched Bend Bar	5.27
Heat-Treated Zirconia, 1350 C processed	Notched Bend Bar	5.57
Zirconia/NCI Joints		
Zirconia/NCI Joint	Notched Bend Bar	5.89
Zirconia/NCI Joint	Cracked Disk, 0-degree	6.63
As-Received Zirconia	Notched Bend Bar**	9.70
As-Received Zirconia	Cracked Disk, 0-degree	8.60

- * Q_{Ic} = K_{Ic} for monolithic materials, and it corresponds to apparent Mode I fracture toughness for joints.
- ** Chevron-notched bend bar.

For predicting mixed-mode fracture conditions, it is important to determine the variation of the critical strain energy release rate, G_o , as a function of mode mixity, Ψ . Evans and Hutchinson^[8] have suggested a function of the form,

$$G_o / (\cos^2 \Psi + k \sin^2 \Psi) \quad (38)$$

for joints, where G_o is the critical strain energy release rate under pure Mode I loading and k is a material constant. The analysis of data on monolithic ceramic materials indicated that a function of the form

$$G_o = G_o \sec^2 (\Psi / \Psi_o) \quad (39)$$

represents the monolithic data very well, where Ψ_o is a constant depending upon the material. Based on this evaluation, the functional form given by Equation (39) was selected a priori for the JAD model. The value of G_o was determined from testing bulk interlayer cracked bend bars (for zirconia/zirconia joints), and from the preliminary validation results using cracked bend bars (for zirconia/NCI joints). However, the value of Ψ_o was not known.

It was decided that the data from cracked disk specimens would be used for determining Ψ_o . Because the cracked disk specimen data were also used for validation of the JAD model, it was decided that only one data-point from the disk specimens would be used for evaluating Ψ_o , and then the rest of the disk data would be used for comparing predicted and measured fracture loads.

Table 11 provides the apparent mixed-mode fracture toughness data for the cracked disk specimens. The analytical results of Atkinson et al.,^[7] for homogeneous material were used for computing the apparent stress-intensity factors from the failure loads. As already indicated, Atkinson's results were within 3 percent of the FEM results on zirconia/NCI cracked specimens, primarily because of the good matching of the elastic constants of zirconia and NCI. From the apparent stress-intensity values, the local stress intensity factors and G at the interlayer location were calculated using the JAD model calculations, and these local G values were plotted versus Ψ . Figures 37 and 38 are plots of local $G(\Psi)$ versus Ψ for zirconia/zirconia and zirconia/NCI joints, respectively. Although there is only one data-point corresponding to each value of non-zero Ψ , the results at $\Psi = 0$ show a degree of scatter in the joint toughness. The solid curves in Figures 37 and 38 were obtained by using the data at $\Psi = 72.3$ degrees, and equating G_o with the average of G at $\Psi = 0$ degree ($G(0) = 20.9$ N/m for zirconia/zirconia joints, and 237.7 N/m for zirconia/NCI joints), thereby calculating Ψ_o according to Equation (39) above. An angle of 72.3 degrees was selected for determining Ψ_o because it was a sufficiently large angle to estimate mixed-mode effects, and also because it was sufficiently removed from 90 degrees, where crack-face rubbing could mask out true toughness values. However, any other angle could in principal be chosen (such as, say, 45 degrees), although an angle between 65 and 80 degrees is desirable because it spans a reasonably large range of Ψ .

Using the above approach, values of Ψ_o of 1.14 and 1.49 were obtained for the zirconia/zirconia and zirconia/NCI joints, respectively. These values, together with the $G(0)$ data, were substituted into Equation (39) to obtain the solid curves in Figures 37 and 38. Figure 37 shows that the solid curve appears to represent the data trend quite well, except at conditions approaching 90 degrees. The lack of agreement may partly be due to the fact

Table 11. Results of fracture tests on cracked and uncracked disk specimens.

Specimen Number	Thickness, B mm	Diameter, D mm	Crack Length, 2a mm	Angle, Degrees	Load, P N	QI, MPa/m	QII*, MPa/m	ψ Degrees	G, local** N/m
Zirconia/Zirconia Joint With MASZ-67 Interlayer									
Uncracked Disk Specimen									
HP22-UD1	2.41	27.43	0.00	30.0	5785.0	-	Sh. S.E.=96.5 MPa	-	-
HP22-UD4	2.43	27.43	0.00	10.0	7120.0	-	No Failure	-	-
Cracked Disk Specimen									
HP22-CD3	2.53	27.43	12.70	0.0	1441.8	2.47	0.00	0.0	27.9
HP22-CD1	2.43	27.43	11.51	30.0	2202.8	-1.21	6.07	101.3	98.4
HP22-CD4	2.53	27.43	11.94	15.0	2225.0	1.96	3.99	63.8	78.6
HP23-CD2	3.59	35.56	16.51	0.0	2047.0	2.08	0.00	0.0	19.8
HP23-CD3	3.59	35.56	16.51	0.0	2314.0	2.35	0.00	0.0	25.2
HP24-CD2	3.62	35.56	16.51	0.0	1691.0	1.72	0.00	0.0	13.5
HP24-CD3	3.61	35.56	16.51	0.0	1958.0	1.98	0.00	0.0	17.9
HP24-CD4	3.60	35.56	15.49	22.5	3168.4	0.27	4.86	86.8	78.9
HP24-CD5	3.63	35.56	16.51	10.0	2296.2	1.88	2.02	47.1	32.7
HP23-CD4	3.63	35.56	14.99	18.0	3982.8	1.60	5.02	72.3	103.8
Zirconia/Zirconia Joint With MASZ-80 Interlayer									
HP25-CD4	3.63	35.56	16.51	0.0	2420.8	2.46	0.00	0.0	-
Zirconia/Nodular Cast Iron Joint									
LMC3-CD2	3.63	35.56	17.78	0.0	4138.5	4.73	0.36	4.4	116.2
LMC3-CD3	3.69	35.56	16.00	15.0	6630.5	3.67	7.80	64.8	334.1
LMC4-CD1	3.59	35.56	17.78	0.0	6764.0	7.82	0.60	4.4	317.7
LMC4-CD4	3.63	35.56	17.78	0	6408.0	7.33	0.56	4.4	279.15
LMC5-CD3	3.61	35.56	17.78	10	6319.0	5.46	5.86	47.0	311.66
LMC5-CD6	3.62	35.56	16.51	20	8544.0	2.18	12.88	80.4	687.25
LMC5-CD7	3.63	35.56	14.99	18	8588.5	3.44	10.78	72.3	541.06

* QI and QII correspond to apparent Mode I and Mode II stress intensity factors for joints.

** $G_{local} = (f_s^2)^* \{ (QI^2)^2 + (QII^2)^2 \} / E$; is the local strain energy release rate in the interlayer location. f_s is a shielding factor that local stress intensity factor, and arises from modulus mismatch effects.

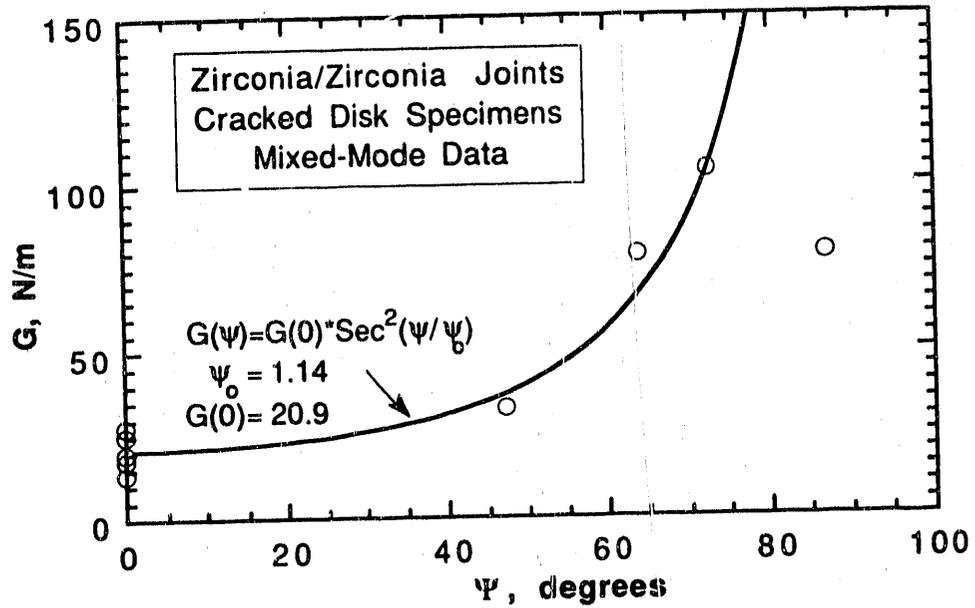


Figure 37. Strain energy release rate as a function of mode mixity for zirconia/zirconia specimens.

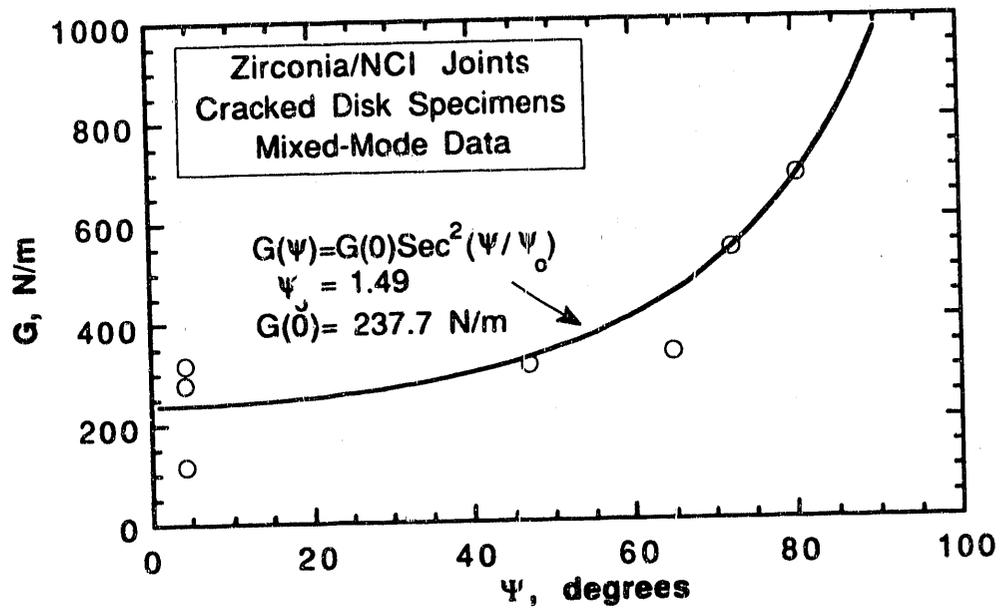


Figure 38. Strain energy release rate as a function of mode mixity for zirconia/NCI specimens.

that at angles approaching 90 degrees, closure of crack surfaces can provide erroneous results. More likely, a larger data base is necessary to evaluate whether the data at 86.8 degrees is indeed an outlier or whether the functional form shown in Equation (39) needs to be modified. For the zirconia/NCI joints, the solid curve provided excellent correlation with most of the data.

In the final validation of the JAD model, the above values of Ψ_0 , together with values of G_0 obtained earlier from experiments on bulk interlayer material, were used for assessing fracture under mixed-mode conditions; note that average $G(0)$ (at $\Psi = 0$ degree) for disk specimens were not used in the final validation of the JAD model.

Figures 39 to 42 are load-displacement plots of zirconia/zirconia and zirconia/NCI disk specimens. The COD displacements were measured across the crack mouth at a distance of 3.81 mm on either side of the slit. Some amount of non-linearity is observed for the zirconia/NCI joint (Figure 42), and may have been the result of small scale plasticity prior to fast fracture. The experimental and predicted load-displacement plots were not compared in this program. However, the experimental traces are provided for future reference and for further validation of the JAD analysis.

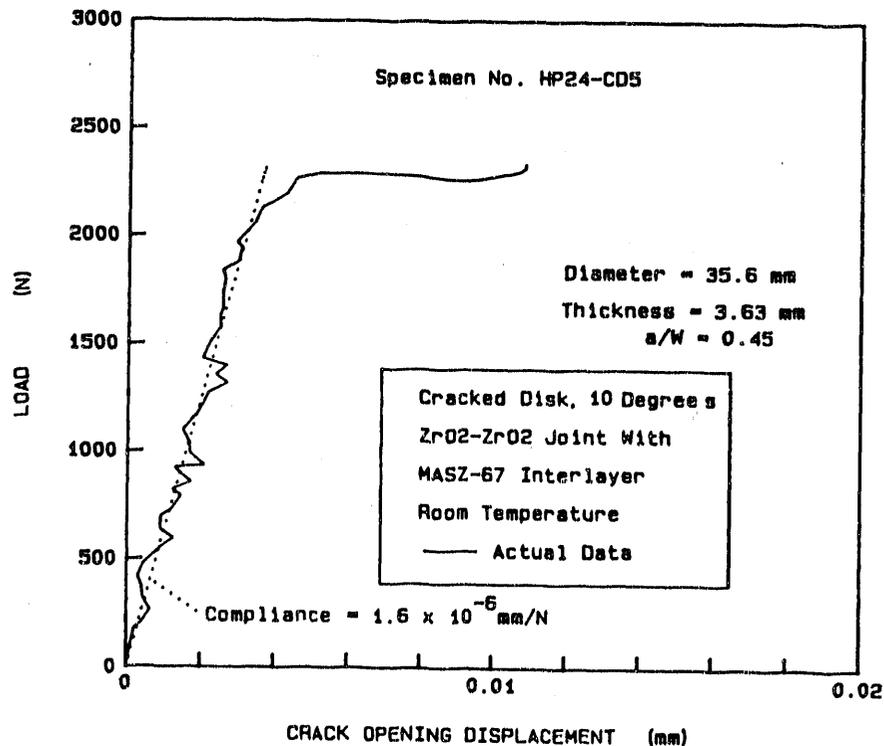


Figure 39. Load versus crack opening displacement plot for a zirconia/zirconia cracked disk specimen. Specimen loaded at 10 degrees to joint.

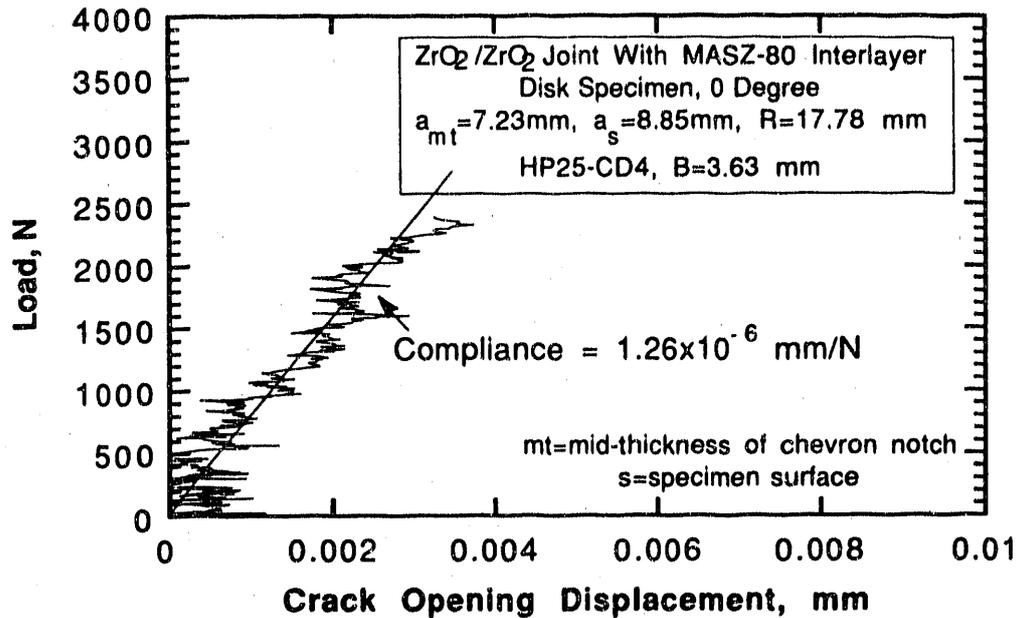


Figure 40. Load versus disk crack opening displacement plot for a zirconia/zirconia disk specimen.

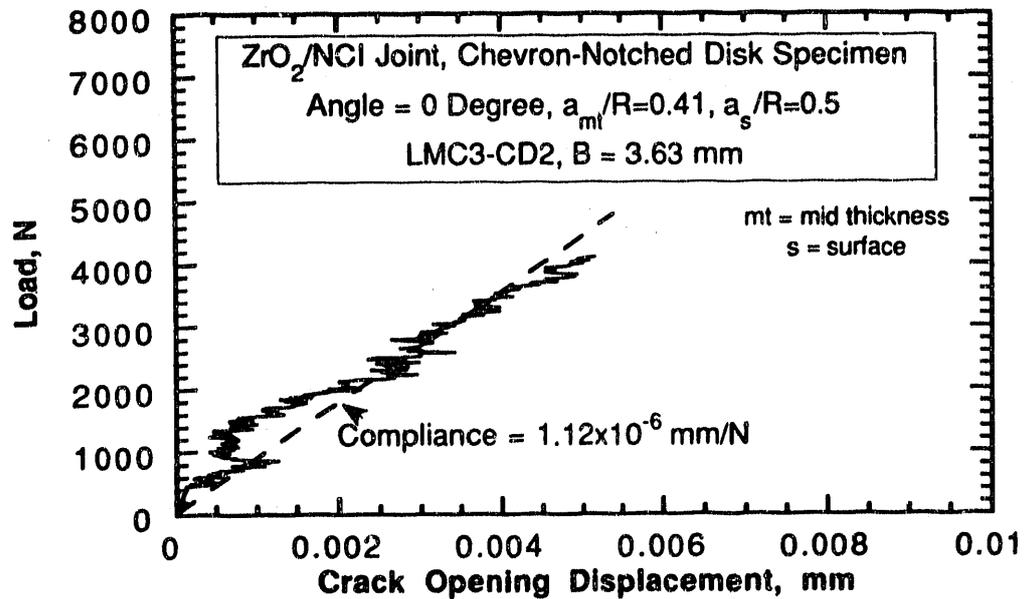


Figure 41. Load versus disk crack opening displacement plot for a zirconia/NCI cracked disk specimen. Specimen loaded at 0 degrees to joint.

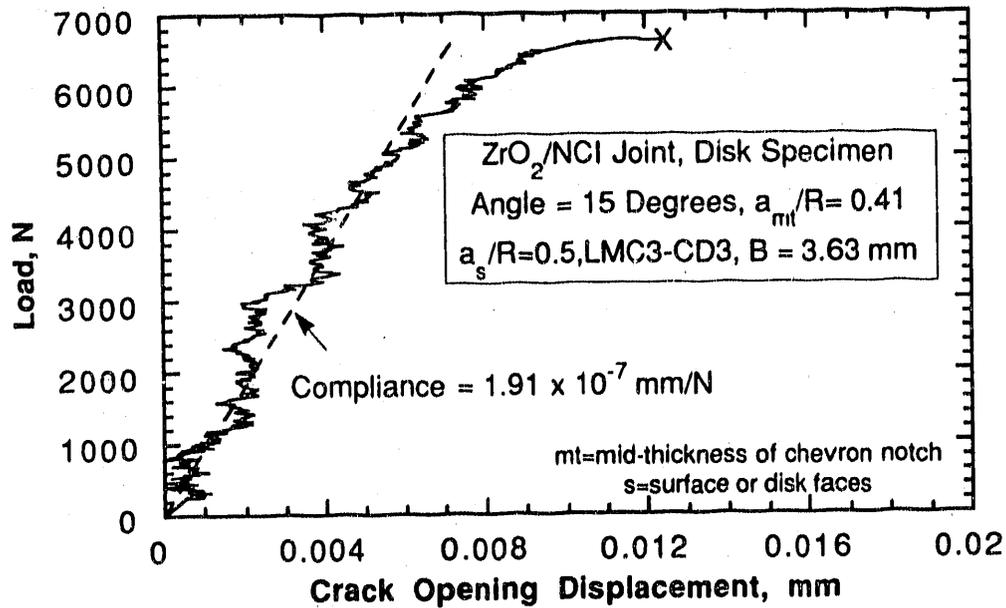


Figure 42. Load versus disk crack opening displacement plot for a zirconia/NCI cracked disk specimen. Specimen loaded at 15 degrees to joint.

3.3.3 Microstructure and Fractography of Joints

Zirconia/Zirconia Joints. Figure 43 is a microprobe back scattered electron (BSE) image of a joint fabricated with MASZ-67 interlayer at 1350 C. The figure shows that the joint had a thickness of approximately 150 μm , and the diffused reaction zones extended approximately 50 μm from the joint surfaces. The three largest dark areas in Figure 43 correspond to pores or joint defects, but the smaller dark regions are not pores. Those regions appear dark in the figure because of the higher concentration of lighter atomic-weight elements.

Higher magnification micrographs^[5] indicated that the bright white circular particles in the figure were retained zirconia particles. X-ray diffraction results^[6] showed that the particles were primarily tetragonal zirconia rather than monoclinic zirconia, although nominally pure zirconia powder was used in preparing the MASZ interlayer. These retained tetragonal zirconia particles explain why improved joint properties were observed with increased zirconia concentration in the interlayer. The dark phase in Figure 43 contained a high concentration of magnesia, and the blocky grey phase was evaluated to be zircon (ZrSiO_4), most likely produced by the reaction of zirconia with silica. The undesirable formation of zircon reduced the bond properties, and explains why a minimum of 67 weight-percent zirconia was needed in the interlayer material for producing acceptable bond strengths. Microprobe investigations also indicated that Mg and Si were primarily responsible for good wettability between the interlayer and the substrate.

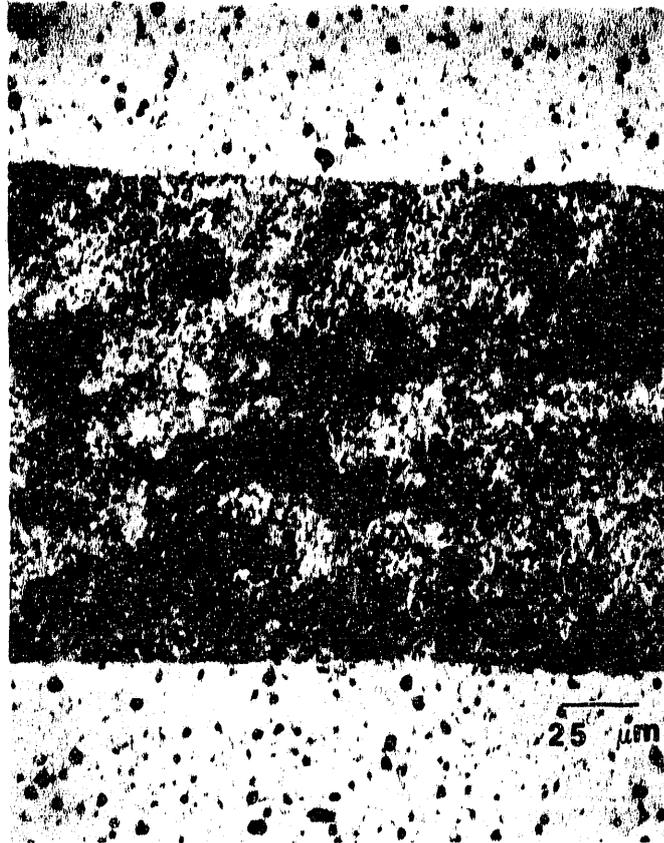


Figure 43. Microprobe BSE image of a large scale zirconia/zirconia joint fabricated using MASZ-67 interlayer.

Figure 44 is a fractograph of the base zirconia, and it shows that the base material had a substantial concentration of pores. These pores were located primarily at grain boundary triple points. For the joining experiments, the pores acted as preferential sites for accumulation of Mg and Si, since high concentrations of those elements were observed in metallographic specimens in the broad reaction zones on either side of the joint.

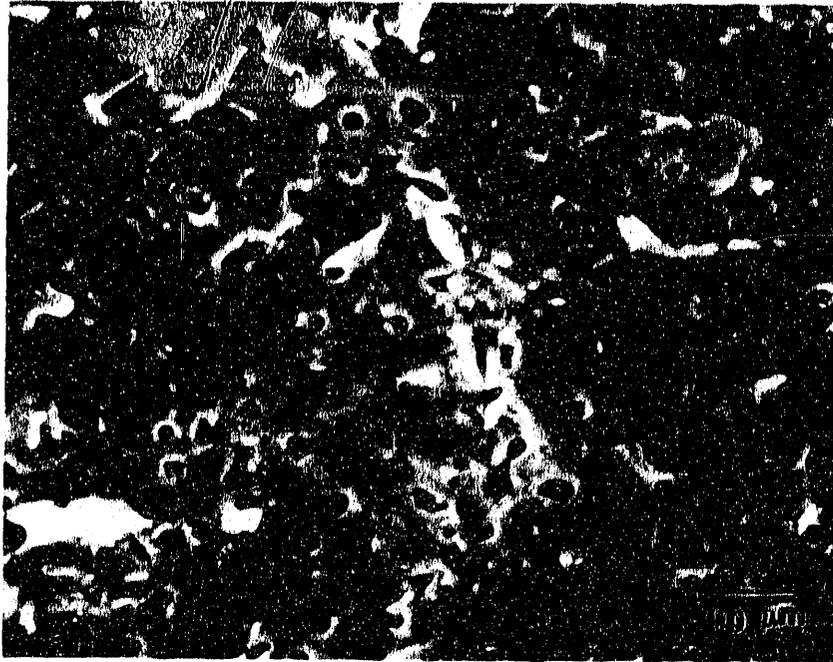


Figure 44. SEM micrograph of fracture surface of as-received zirconia.

On a macroscopic scale, the fracture surfaces of zirconia/zirconia joints were quite rough, indicating good fracture toughness. Figure 45 illustrates the fracture surface of a joint fabricated with the MASZ-67 interlayer. The fracture surface has a close resemblance to the microstructure of the interlayer material in that fine particles can be observed in a smooth-textured matrix. Energy dispersive spectroscopic (EDS) analyses provided the following approximate elemental composition (weight percent) of the fracture surface: 5.6 Mg, 7.1 Al, 14.4 Si, 5.7 Ti, 67.2 Zr. From the elemental composition, and the fracture surface morphology, it was concluded that the failure propagated primarily through the interlayer material.

Although most of the bend bars showed that the preferred fracture path was through the interlayer, there were a few instances where the crack appeared to propagate partly through the reaction zone. However, the bend strengths in these instances were not

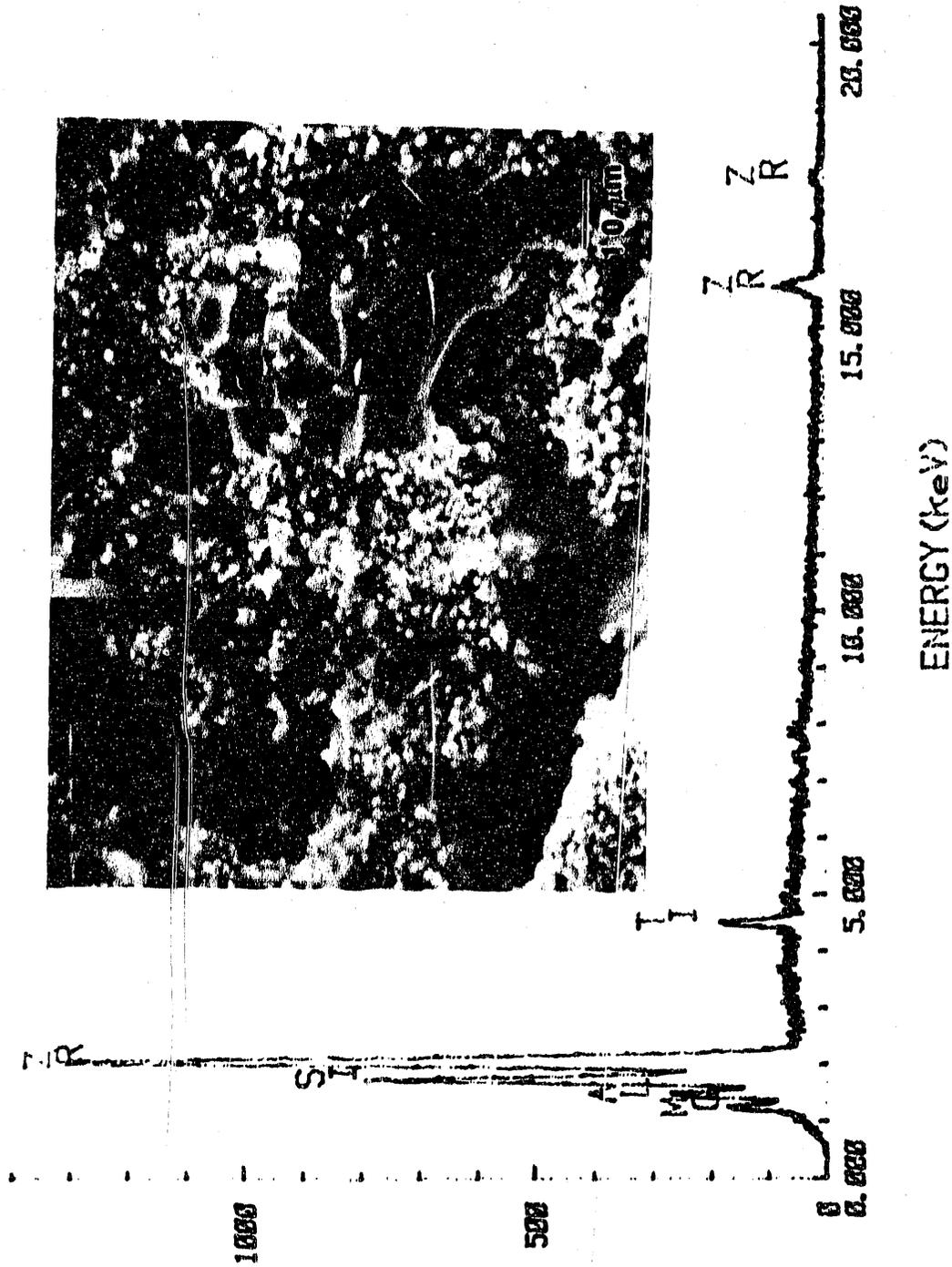


Figure 45. SEM micrograph and corresponding EDS trace of a zirconia/zirconia joint fabricated with MASZ-67 interlayer. The micrograph and EDS trace indicate that the crack propagated through the MASZ-67 interlayer.

significantly different compared with the case where the crack propagated completely through the interlayer, indicating that the reaction zone had a strength that was similar to the interlayer. In the particular case of a zirconia/zirconia joint fabricated with MASZ-80 interlayer at 1400 C, the failure propagated completely through the reaction zone rather than the interlayer. This behavior was consistent with the fact that the bulk interlayer fabricated at that temperature had a toughness of greater than 5 MPa√m, whereas the reaction zone was estimated to have a toughness of only 2.4 MPa√m (much less than the heat treated zirconia, 5.6 MPa√m).

Zirconia/NCI Joints. Figure 46 contains electron microprobe images of a zirconia/NCI joint. Figure 46b is a BSE image based on Figure 46a, and shows the braze-metal/zirconia interface at a high magnification. The white region in this figure is primarily Ag, although Cu and In are also present. Figures 46c and 46d are Ti and Cu elemental maps, respectively, corresponding to Figure 46b. These figures indicate that there is primarily a pure Ti-layer (may contain oxygen) at the zirconia interface, followed by a Cu-Ti layer immediately adjacent to the pure Ti-layer. Clearly Ti was responsible for good bonding between the zirconia and the braze metal. Although the zirconia next to the braze metal did not appear to be microstructurally different from the as-received zirconia, a dark-brown band (0.3-0.4 mm wide) could easily be observed in the zirconia next to the braze metal; the as-received zirconia had a yellow color. This region corresponded to the reaction layer, and work done at ORNL indicated that the reaction zone corresponds to non-stoichiometric zirconia (ZrO_{2-x}) with depleted oxygen content.

Figures 47a and 47b are fractographs of the NCI side and the zirconia side, respectively, of a zirconia/NCI joint broken under Mode I loading. EDS analyses indicated that the void containing projections (region A3) in Figure 47a were composed of pure zirconia, indicating that the propagating crack dug into the reaction zone of the zirconia. The flatter regions, such as A4, contained primarily Ti and Cu, with Ti being the dominant element. On the zirconia side (Figure 47b), regions such as A2 corresponded to some braze metal pockets held back by the zirconia. The smooth regions (such as A1a) corresponded to the zirconia surface, but it was found to contain Ti and Cu, with Ti again being the dominant element. On the other hand, regions such as A1B corresponded to pure zirconia, being exact counterparts of the projected regions shown in Figure 47a. Thus, these fractographs indicate that the crack propagated partly through the reaction zone of the zirconia and partly through the extremely fine Ti-rich layer between the braze metal and the zirconia. Based on an examination of a number of fracture surfaces, the reaction-zone and Ti-rich layer each occupied between 40 and 60 percent of the area of the fracture surface.

Figure 48 illustrates the microstructure of a surface obtained by sectioning perpendicular to the fracture plane. The left side contains the NCI and braze metal, the central region corresponds to a space, and the right side corresponds to zirconia. The fracture halves were not correctly positioned when preparing the metallurgical sample, so that the fracture regions are not matching halves. Nevertheless, the micrographs illustrate the general fracture morphology. The micrograph shows that the crack propagated partly through the reaction zone of the zirconia and partly through the Ti-rich zone of the braze-metal/zirconia interface.

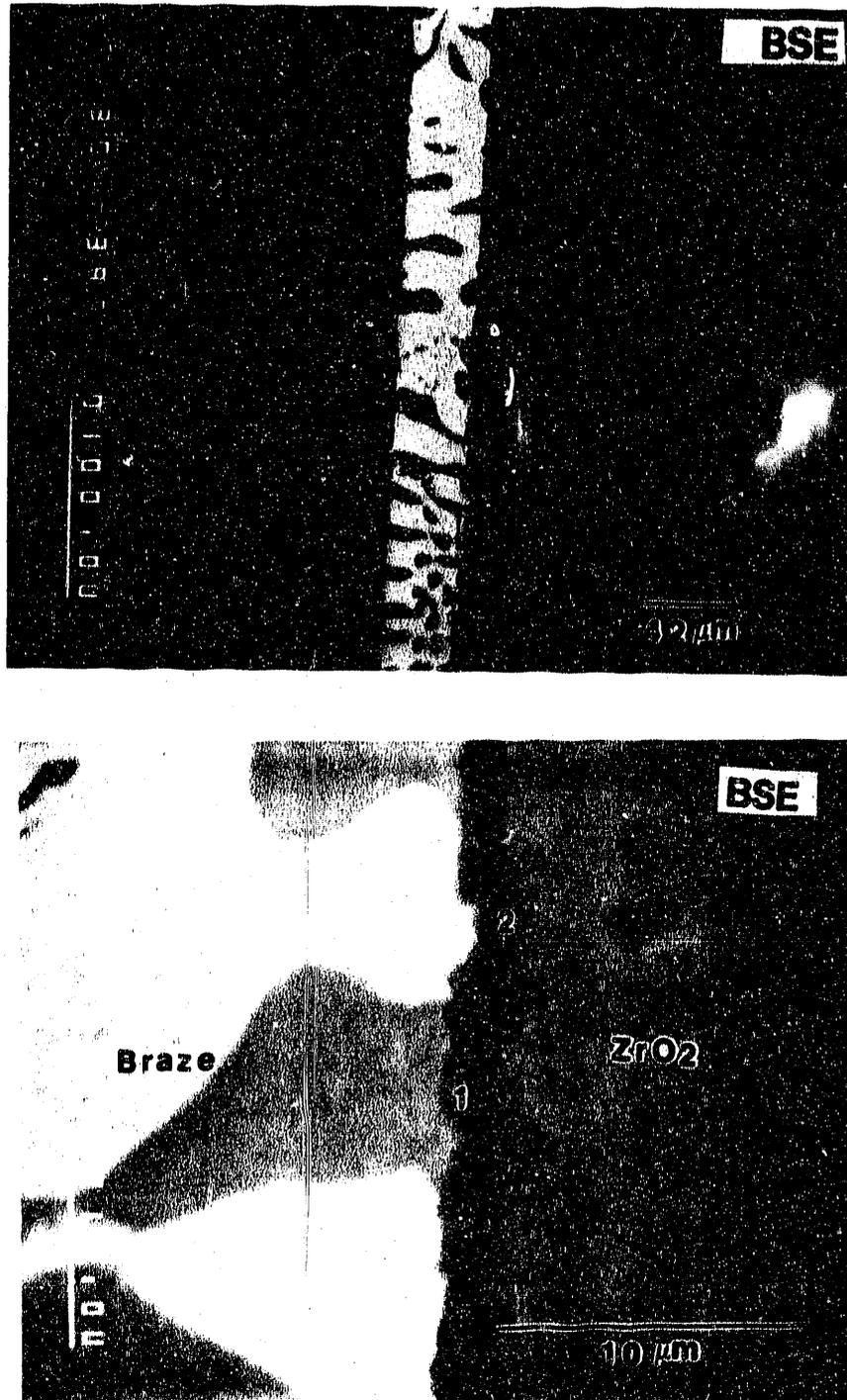


Figure 46. Microprobe BSE image and elemental maps for a zirconia/NCI joint. (a) low magnification micrograph with NCI on left side, braze in the middle, and zirconia on the right side. (b) BSE image of the braze/zirconia interface at a high magnification. (c) and (d) are Ti and Cu maps, respectively, of the same region as (b).

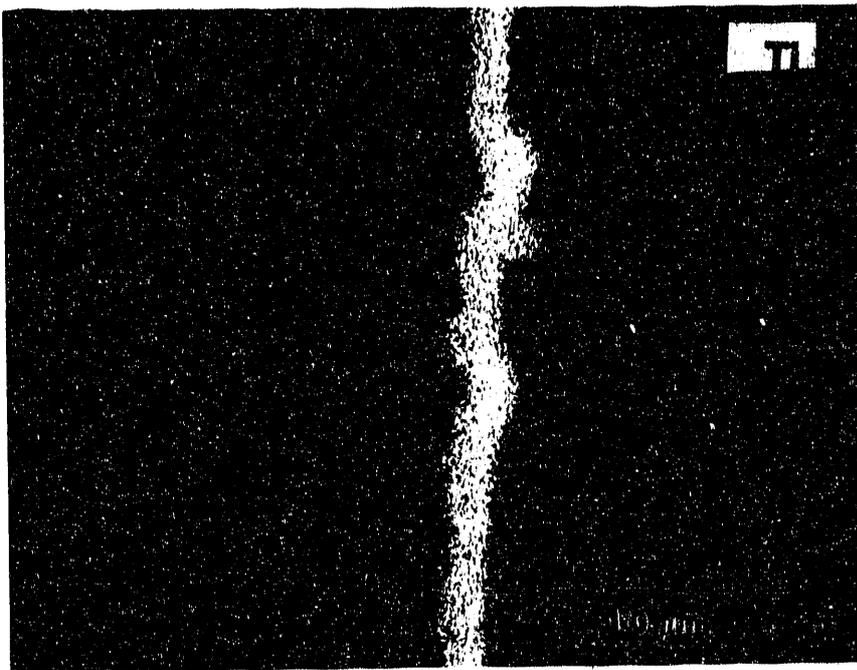
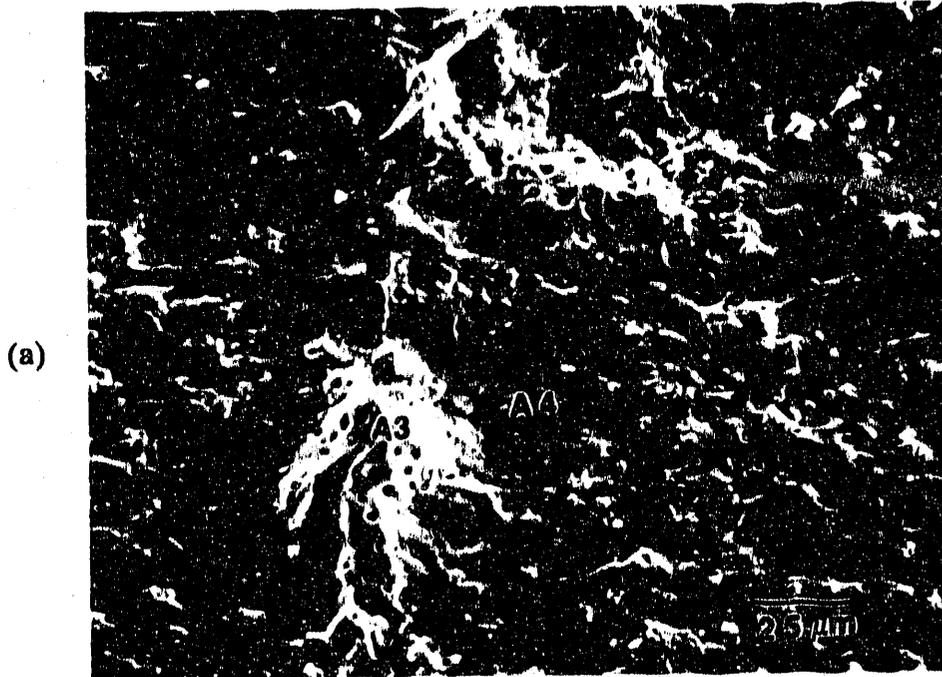
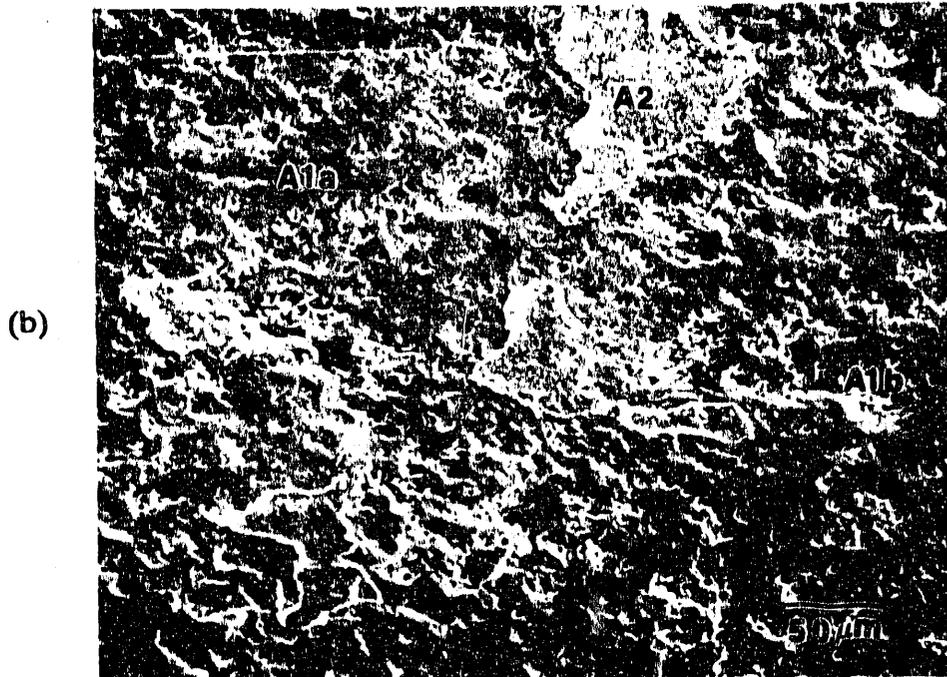


Figure 46. Cont.

NCI Side



H39

ZrO₂ Side

84141

Figure 47. Fracture surfaces of a zirconia/NCI joint for a notched bend bar. (a) NCI side of the fracture surface. (b) zirconia side of the fracture surface.

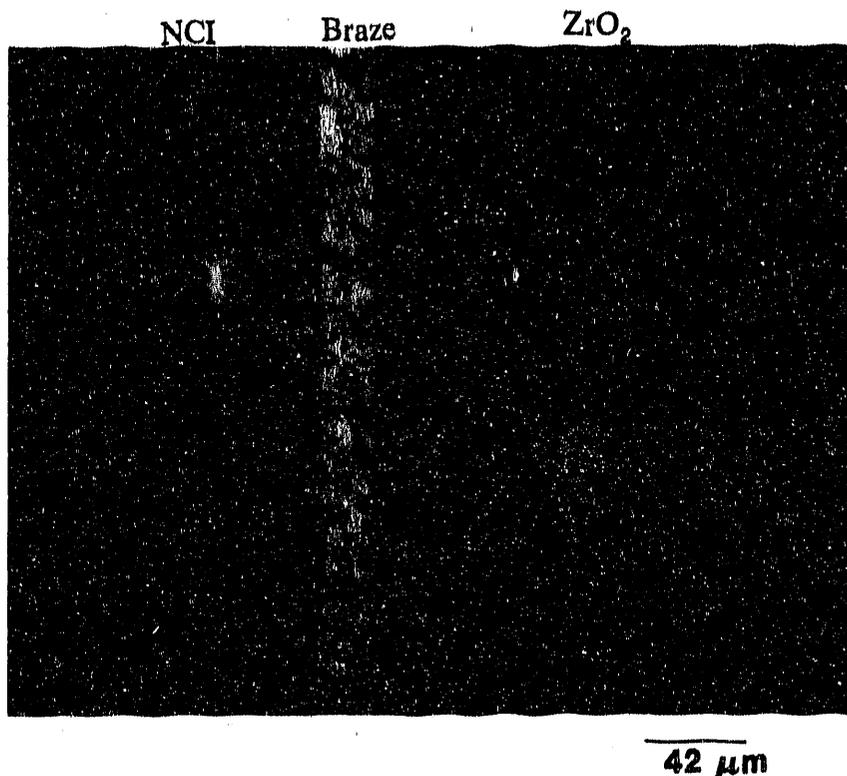
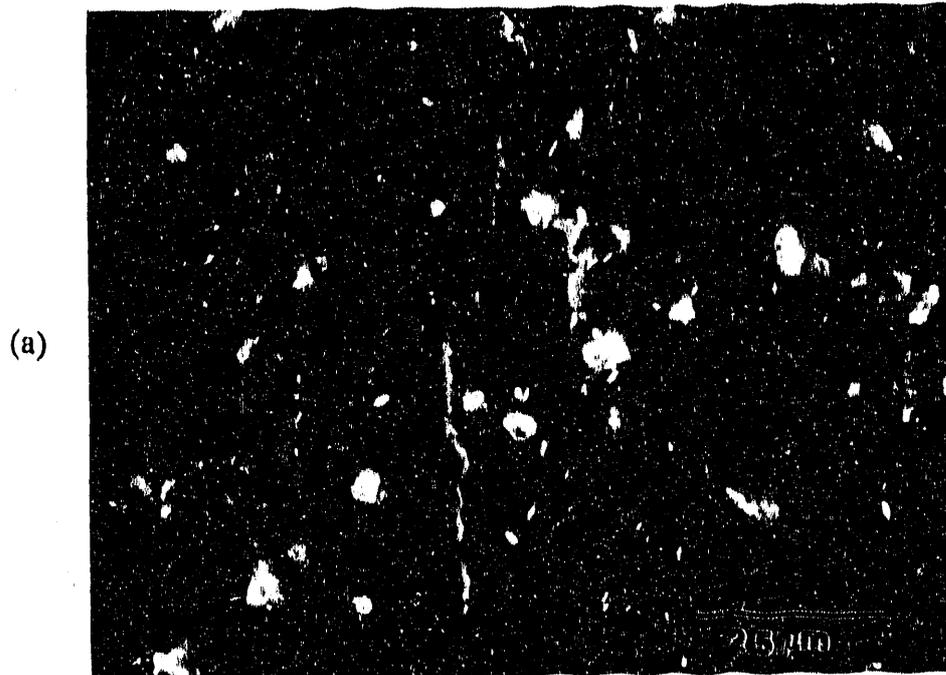
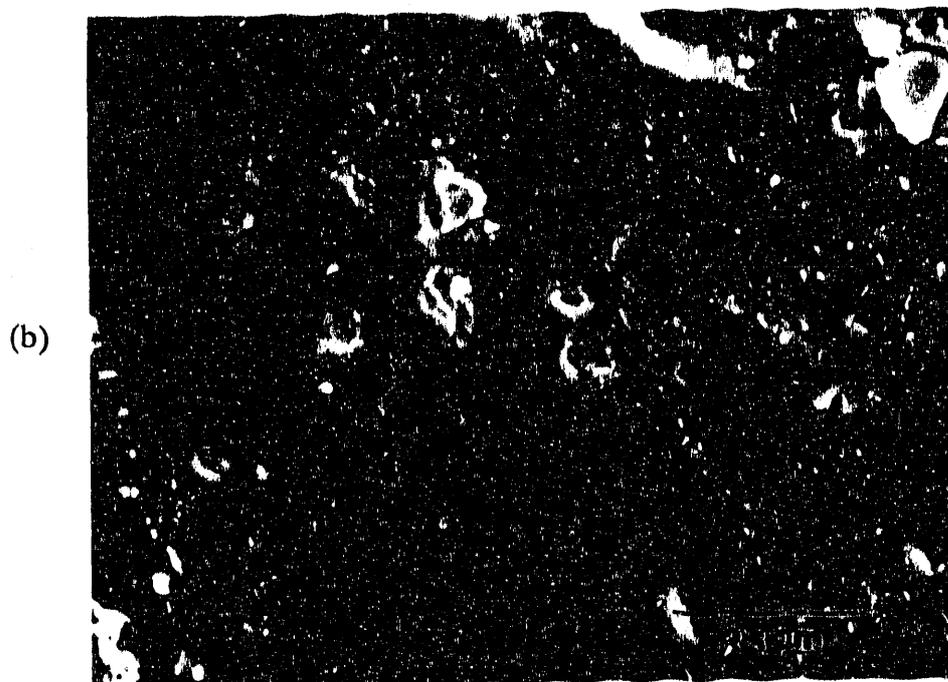


Figure 48. Microstructure of the NCI (left) and zirconia (right) immediately adjacent to the fracture surface. The cross-sections are not matching halves of the fracture surface.

With increased mixity of loading (increasing ψ), there was a gradual change in the fracture path and fracture morphology. On the zirconia side of the disk specimen, the crack deviated from the notch plane and propagated towards the loading point. This behavior was similar to what has been observed for monolithic ceramic specimens. The extent of crack growth along the bond-line prior to crack deviation (for zirconia side) depended on the loading angle, being approximately 6 mm for an angle of 10 degrees, and being less than 1 mm above 15 degrees. On the NCI side, however, the crack could not propagate into the NCI; rather the crack propagated all along the bond line. Figures 49a and 49b correspond to the NCI side and zirconia side, respectively, of the fracture surfaces of a disk specimen where the crack propagated through the bond line. The sample was loaded with the notch at an angle of 20 degrees with respect to the load line ($\psi = 80.4$ degrees). EDS analysis indicated that on the NCI side the surface layer was rich in Ti and Cu, with Ti being the dominant element. However, contrary to Mode I fracture, hardly any zirconia was found on the fracture surface. The wavy structure seen in Figure 49a probably corresponds to shear deformation of the Ti-rich layer and of the braze metal beneath it. On the zirconia side, the primary elements detected were Zr and Ti and Cu. Here too, unlike Figure 47b, very little zirconia appeared to have been pulled out by the braze metal during fracture. Overall, it appeared that under shear loads there was significant plastic shear deformation of the braze metal, including the Ti-rich layers, and these resulted in shear failure of the interface without any significant accompanying failure of the reaction zone of the interlayer. Such fracture morphology differences with the Mode I loading probably were responsible for elevating the G_c with increasing ψ .



86172



86167

Figure 49. Fracture surfaces of a zirconia/NCI joint for a cracked disk specimen, with the crack at an angle of 20 degrees ($\Psi = 80.4$ degrees) with respect to the load line. (a) NCI side of the fracture surface. (b) Zirconia side of the fracture surface.

4.0 VALIDATION

Validation of the JAD model was performed in two steps:

1. Preliminary validation, where the model predictions were compared with measured failure loads of notched and unnotched bend bars.
2. Final validation using cracked disk specimens that contained 20 times more bonding area than bend bars, and comparing the predicted failure loads with measured failure loads.

The joint assessment diagrams for four representative specimens were presented earlier. Calculations like those were conducted to predict the failure loads of cracked disk and bend bar specimens. Predictions of the strength of unnotched bend bars were performed only on zirconia/zirconia joints and they were based on the strength of the ceramic interlayer, appropriately modified to account for shielding effects due to moduli mismatch.

Figure 50 provides comparisons of measured and predicted failure loads and is a plot of the data displayed in Table 12. The figure includes data from zirconia/zirconia and zirconia/NCI joints, unnotched and notched bend bars, and cracked disk specimens loaded at various angles to the crack. Each data point is an average of several readings, particularly for the bend bar specimens. Thus, the plot contains an extensive database.

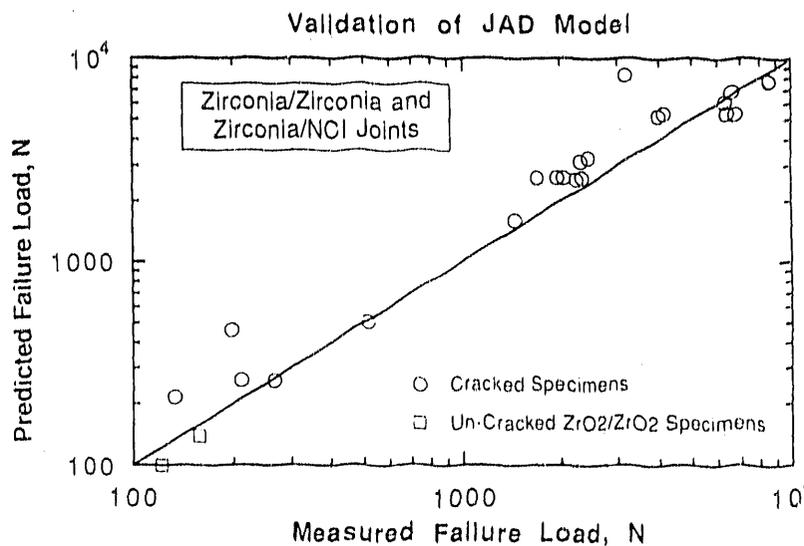


Figure 50. Results of validation analyses and testing with zirconia/zirconia and zirconia/NCI joints. The bend bars were used for preliminary validation and the disk specimens were used for final validation.

In Table 12, the geometric data needed to calculate the failure loads are also displayed. Shown for each experiment, where applicable, are the joining temperature, the specimen width, B , and depth, W , the half-crack length, a , the radius of the disk, R , the angle between the loading points and the crack, ϕ , the percent zirconia in the MASZ interlayer, and the calculated angle the assessment vector makes with the P_r -axis.

The comparisons between predicted and calculated failure loads are encouraging. The exceptions that appear are believed not to be due to errors in model calculations. Rather, we believe them to be the result of differences in failure path (for example, failure in the reaction zone of zirconia/zirconia joints fabricated at 1400 C) which could not be predicted a priori, and also degradation of the MASZ-67 interlayer in zirconia/zirconia joints. Note that the measured failure loads are consistently below the predicted failure loads for zirconia/zirconia joints fabricated using MASZ-67 interlayer. For these joints, if the results of preliminary validation are used to modify the G_c of the interlayer, then better agreement is obtained in the final validation. The exceptions in Table 12 point out the need to evaluate the crack path as part of any preliminary investigations, and to include some form of the process parameters in the JAD model calculations.

Overall, the degree of correlation between predicted and measured failure loads of different joints and with different specimen geometries strengthens the validity of the joint design model.

Joint Type	MASZ Percent	Joining Temperature	Beam Depth, W	Width, B	Half-Crack Length, a, mm (a)	Radius, R	Crack Angle, Degrees	Failure Load Computed, N	Failure Load Measured, N
Preliminary Validation (Bend Bars)									
Zirconia/Zirconia(b)	67	1350 C	5.08	1.55	1.55	-	-	260	211
Zirconia/Zirconia(b)	80	1350 C	5.08	1.63	1.63	-	64	260	267
Zirconia/Zirconia(b)	67	1400 C	5.08	1.63	1.63	-	64	215	134
Zirconia/Zirconia(b)	80	1400 C	5.08	1.60	1.60	-	80	460	198
Zirconia/Zirconia(c)	67	1350 C	2.54	2.54	2.54	-	-	100	122
Zirconia/Zirconia(c)	80	1350 C	2.54	2.54	2.54	-	-	140	158
Zirconia/NCI (b)	-	760 C	5.08	1.60	1.60	-	75	505	514
Final Validation (Cracked Disks)									
Zirconia/Zirconia	67	1350 C	-	2.53	13.72	0	79	1600	1442
Zirconia/Zirconia	67	1350 C	-	3.59	17.78	0	80	2600	2047
Zirconia/Zirconia	67	1350 C	-	3.62	17.78	0	80	2600	2314
Zirconia/Zirconia	67	1350 C	-	3.61	17.78	0	80	2600	1691
Zirconia/Zirconia	67	1350 C	-	3.63	17.78	10	79	3135	1958
Zirconia/Zirconia	67	1350 C	-	2.53	13.72	15	77	2550	2225
Zirconia/Zirconia	67	1350 C	-	3.63	17.78	18	78	5200	3982
Zirconia/Zirconia	67	1350 C	-	3.60	17.78	22.5	75	8200	3168
Zirconia/Zirconia	80	1350 C	-	3.63	17.78	0	81	3250	2421
Zirconia/NCI	-	760 C	-	3.63	17.78	0	85	5350	4139
Zirconia/NCI	-	760 C	-	3.59	17.78	0	85	5291	6764
Zirconia/NCI	-	760 C	-	3.63	17.78	0	85	5350	6408
Zirconia/NCI	-	760 C	-	3.61	17.78	10	85	6100	6319
Zirconia/NCI	-	760 C	-	3.69	17.78	15	85	7750	6631
Zirconia/NCI	-	760 C	-	3.63	17.78	18	85	7750	8589
Zirconia/NCI	-	760 C	-	3.62	17.78	20	86	7690	8544

- (a) a is the full crack length for notched bend bars, and half crack length for disk specimens.
- (b) Average of multiple tests reported here.
- (c) Un-cracked (smooth) bend bars. Average of multiple tests reported here.

Table 12. Validation results - comparisons of predicted and measured failure loads of joints.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The most significant accomplishment of the work described in the present report is the development of an engineering model applicable to design improvement and structural integrity assessment of ceramic/ceramic and metal/ceramic joints. A novel modeling approach was adopted which marries state-of-the-art knowledge in mechanics of material interfaces with engineering fracture mechanics concepts. This resulted in a model which provides a rational link between constituent material properties and joining process variables, and load carrying capacity of a joint. As shown in Figure 51, the JAD model can be used for (a) assessing a given joint's load carrying capacity and (b) designing improvement by systematically changing constituent properties and joining process variables.

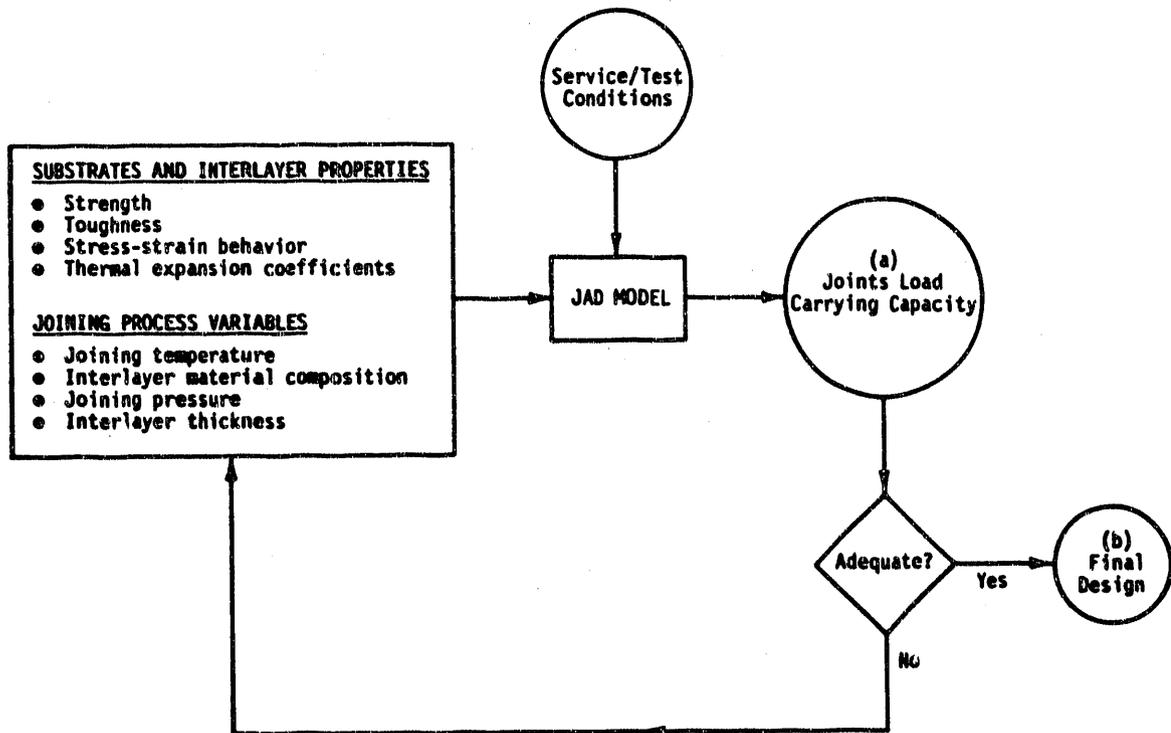


Figure 51. Role of JAD model: (A) structural integrity assessment and (b) joint design.

Besides the analytical modeling effort, extensive laboratory experimentation was performed involving fabrication and testing of zirconia/zirconia and NCI/zirconia joints. Remarkable success was achieved in fabricating zirconia/zirconia joints by a novel hot forging method. A versatile test specimen in the shape of a disk was introduced for characterizing joints. In addition to experiments on joints, which provided data for validating JAD model predictions, laboratory tests were performed to generate thermo-mechanical property data for each constituent material: zirconia, cast iron, braze and the interlayer material for zirconia/zirconia joints. These constituent property data were used as part of the input to the model.

The JAD model was validated by comparing predictions with the measured load carrying capacity of two significantly different joint configurations - the bend bar and the disk. The reasonably good agreement between predictions and measurements for both configurations indicates that the model is not geometry specific. Therefore, it would be useful in engineering applications involving other joint configurations.

Overall, the work represents a major step toward the development of an analytical design methodology for ceramic/ceramic and metal/ceramic joints. Nevertheless, the model does need additional validation and to be augmented to formulate a more generally useful joint design methodology.

Additional validation of the JAD model is most needed at elevated temperatures which are representative of engine operating conditions. Also, the model needs to be validated for crack sizes smaller than those used in the present work and more representative of typical flaw sizes anticipated in as-fabricated joints. It would also be worthwhile to assess the accuracy of the JAD model predictions for joints between other metals and ceramic materials than those included in the present work. For this purpose, the recent experiments by GTE Laboratories, Inc. and the Norton Company involving silicon nitride and Inconel joints may provide the needed data.

In its current state of development, the JAD model is only applicable for joints subjected to quasi-static, monotonically increasing loads. In engine applications, a joint is more likely to experience conditions which may consist of load reversals as well as dwell periods giving rise to relatively low sustained loads. Thus, creep, fatigue and possible subcritical crack growth under such conditions may give rise to failure mechanisms not considered in the present work. We recommend that future research be directed toward augmenting the JAD model to include creep and fatigue considerations.

Finally, it is recommended that the JAD model should be formulated in a probabilistic analysis framework to more meaningfully address the issue of joint reliability. Anticipating at the beginning of the present work that a probabilistic design model would eventually be needed, the JAD model was purposefully formulated in a manner such that its incorporation in a probabilistic analysis framework would be relatively straightforward. Because application of the JAD model does not require case by case finite element analyses, the model readily lends itself to practically useful probabilistic analyses which could be performed using a personal computer. In fact, the JAD model has already been implemented in a PC code which can be readily adapted for this purpose.

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