High-Temperature Properties and Failure Criteria for Rocket Nozzle Materials

A. A. VICARIO JR.,* W. T. FREEMAN JR.,† AND E. D. CASSEDAY‡
Hercules Incorporated, Cumberland, Md.

The adaptation of anisotropic failure criteria along with the availability of high-temperature material property data represents a new approach to the prediction of structural integrity in rocket nozzles. Strength, modulus, and elongation data in tension, compression and shear, and coefficient of thermal expansion data were obtained at temperatures up to 4000°F for five nozzle materials. Several failure criteria were investigated using off-axis compression specimens in initial screening tests. Modification of one of the distortion energy criteria appears to be a promising approach for the prediction of the structural integrity of these materials at elevated temperatures and combined loading.

Introduction

DURING the past ten years, sophisticated analytical techniques have been developed to aid in the analysis of nozzles and other rocket motor components. Computer programs are now available which are capable of predicting both thermal and structural response of an axially symmetric body composed of several materials and subjected to a variety of boundary conditions.

These thermal and structural programs provide a powerful tool to the analyst charged with determining accurate margins of safety. However, they are only a partial solution to the problems; of equal importance are correct material properties and an acceptable failure theory. The latter two requirements are interrelated and are the subject of this paper.

Material Characterization

To perform a stress analysis using a finite element program, the elastic constants which relate stress and strain must be known. In order to assess the integrity of the nozzle with respect to strength for the determined stress state, the analyst must also possess an accurate description of the material strength. For isotropic, homogeneous materials subjected to simple states of stress, the structural integrity can be determined by comparing the calculated stress with some fraction of the yield or ultimate strength of the material. These data are available from tension, compression, and shear tests. For more complex stress states and the nonhomogeneous, orthotropic materials which are encountered in rocket nozzles, it is not physically possible to conduct tests which model every complex stress state. It is therefore necessary to develop failure criteria to predict the strength and structural integrity of the nozzle.

The materials commonly used in nozzle construction fall into two general categories: refractories and ablators. The former are characterized by their ability to withstand high-temperature environments without undergoing either phase changes or chemical reactions. Ablative materials undergo a radical change in chemical structure at high temperature and pose a challenge to the investigator interested in determining useful material properties. In contrast to metals, ablative materials possess such distinguishing characteristics as heterogeneity, nonhomogeneity, and anisotropy. The basic micro-structure is a two-phase constituency: fiber and matrix; and at a macro level, the typical laminate is layered such that distinct stratums of fibers are present in the matrix. The micro-level phenomenon determines failure modes, but a quantitative understanding of all such mechanisms and their interaction is not yet available. Therefore, most engineering design and analysis must occur at the macro-level, which recognizes the unidirectional properties as the basic element.

The unidirectional elastic properties and strengths are determined experimentally. Seven independent engineering constants (three Young's moduli, three coefficients of expansion, three Poisson's ratios, and one shear modulus) must be known for an axisymmetric body with orthotropic properties.

For asymmetric loading or three-dimensional analysis, two additional shear moduli are needed. Figure 1 illustrates the basic material coordinate frame and the three shear properties for an anisotropic material: $\tau_{12}(G_{12})$, the in-plane shear strength (modulus); $\tau_{13}(G_{13})$ and $\tau_{23}(G_{23})$, the interlaminar shear strength (modulus). Some strength criteria require additional off-axis or other combined stress values.

Test Methods and Specimens

A viable approach which will minimize the costs involved in a high-temperature testing program is to assume that changes in properties as a result of lamina orientations are a structural

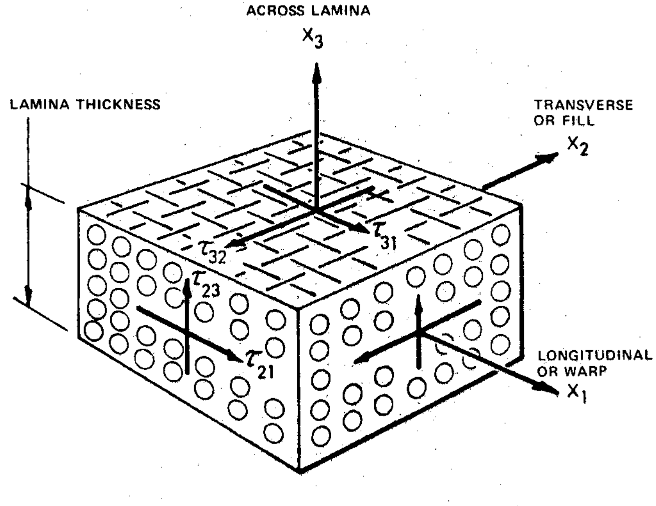

Fig. 1 Definition of material coordinate frame.

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* Senior Development Engineer, Alleghany Ballistics Laboratory.
† Development Engineer, Alleghany Ballistics Laboratory.
‡ Group Supervisor, Alleghany Ballistics Laboratory.

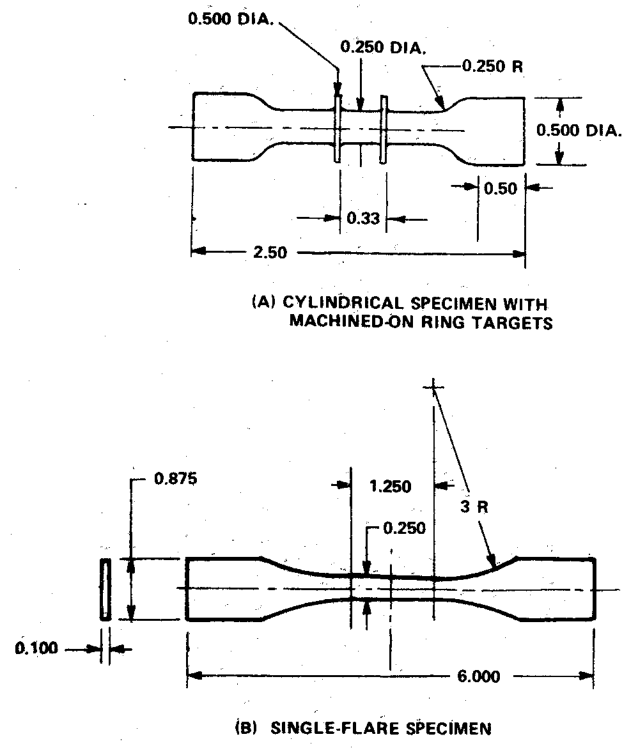

Fig. 2 Tensile test specimen configurations.

computation and not a parameter to be established for each orientation. Therefore the principal property data form the basis for all subsequent design computations, and extreme care must be exercised in their determination.

Strain Measurement

To determine the elastic moduli of nozzle materials, the strain response during the test must be accurately monitored. Strain gage and mechanical strain measurement techniques are limited to temperatures of less than 2000°F, and only optical measurements have proven reliable at temperatures above 2000°F. Optical strain measurement techniques generally employ two photomultiplier tubes that electro-optically track targets or objects having discontinuities in the intensity of reflected or emitted light. Details of this technique may be found in Ref. 1.

Poisson's ratio can be obtained from data determined from longitudinal and transverse tensile and compression tests by recording the lateral as well as the axial strain during the test.

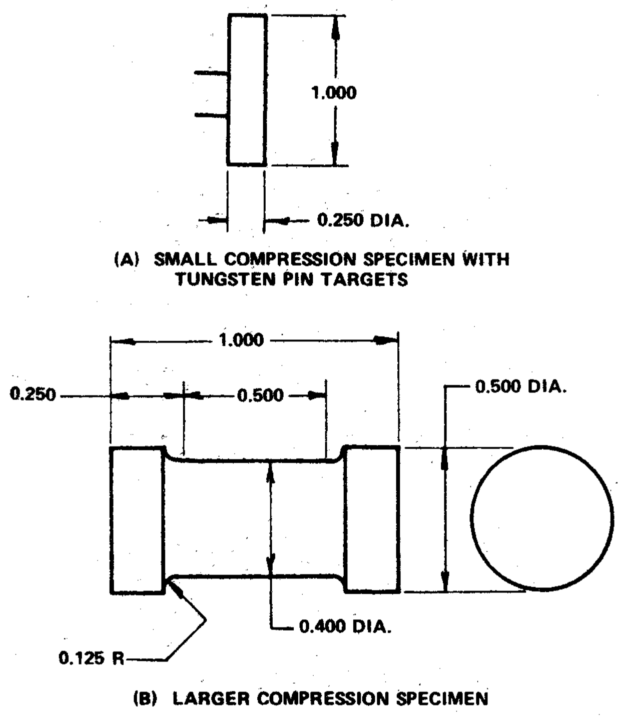

Fig. 3 Compression test specimens.

Tensile Testing

Two specimen configurations that have been used in tensile testing of ablative materials are shown in Fig. 2. Small specimens have the advantage of being obtainable from actual nozzles. Special loading cells are necessary to ensure alignment and load application to the specimens.

Compression Testing

The two major parameters that affect compression test results and account for a large variation in reported property data are: 1) the manner in which the stabilization process augments the matrix material in support of the filaments against buckling, and 2) the end buckling, brooming, or splitting associated with the end-loaded type of specimen.

Two types of specimens suitable for high-temperature compression testing are shown in Fig. 3. Both specimens have a length-to-diameter ratio sufficient to stabilize the specimen and prevent buckling.

Shear Testing

Since most nozzle and exit cone materials are anisotropic, the shear properties are independent parameters that must be determined experimentally. A number of methods are available for the determination of shear properties of anisotropic composite materials.

The plate twist, or four-point loading test, is useful only in determining shear modulus. A complete description of the test and analytical development is given in Ref. 2. The $\pm 45°$ laminate test is a technique for determining the in-plane shear stress-strain response of unidirectional composite materials. A detailed discussion of this procedure may be found in Refs. 3 and 4. For cross-plied ablative materials, this test yields reasonable data.

A notched specimen is sometimes used to determine the shear strength of nozzle materials. This specimen and its inherent difficulties are discussed in Refs. 5 and 6. The short beam shear test is another popular technique used to determine the interlaminar shear strength of fiber-reinforced materials. Nonshear and mixed mode failures can result if the span-to-depth ratio or width-to-depth ratio is improperly chosen. The effects of these parameters are discussed in Ref. 7.

The off-axis test has been successfully used to determine both the shear modulus and the strength of laminated composite materials. The details of this test are discussed in Refs. 8–10.

Results from Material Tests

Table 1 presents some typical data for a graphite-phenolic and asbestos-phenolic material. The graphite phenolic is a square weave fabric laminate which is often used as a nozzle exit cone liner. The asbestos-phenolic is a felt tape-wrapped laminate often used as nozzle exit cone insulation. The materials were tested at a heating rate of 100°F/min.

Examination of the tensile stress-strain curves for both the fill and wrap directions indicated that at room temperature these materials tended to brittle fracture. This behavior continues at elevated temperatures up to 2000°F with the graphite-phenolic being the more ductile of the two materials. Above this range, both materials tended to become very ductile.

The compressive behavior in the warp and fill directions tends to be brittle in nature. Elevated temperatures seem to have an embrittling effect. Across-laminate compressive strains are quite

Table 1 Material properties

Material load	Temp., °F	Strength, 10^3 psi Warp	Strength, 10^3 psi Fill	Strength, 10^3 psi Across lamina	Modulus, 10^6 psi Warp	Modulus, 10^6 psi Fill	Modulus, 10^6 psi Across lamina	Ultimate strain, % Warp	Ultimate strain, % Fill	Ultimate strain, % Across lamina
Graphite	77	23.8	12.0	1.10	1.47	1.33	0.75	2.2	2.6	0.18
Phenolic	1000	14.4	4.68	0.34	0.92	0.25	0.27	2.3	3.6	0.52
Tension	4000	10.5	4.35	0.30	0.50	0.27	0.07	4.96	3.65	0.66
Graphite	77	17.2	13.9	56.0	2.34	1.82	1.02	0.78	1.19	5.95
Phenolic	1000	4.0	3.34	12.0	1.85	0.55	...	3.2	1.28	6.4
Compression	4000	2.1	2.10	19.0	0.23	0.25	0.10	1.70	0.67	12.0
Asbestos	77	15.7	8.32	1.85	3.29	1.11	1.00	0.53	0.96	0.32
Phenolic	1000	7.4	1.00	0.60	1.08	0.35	0.10	0.80	0.30	0.20
Tension	2000	2.6	1.12	0.15	0.62	0.75	0.12	0.46	0.15	0.20
Asbestos	77	21.7	21.1	58.0	2.20	1.34	1.28	2.92	2.84	4.94
Phenolic	1000	8.37	4.20	20.7	1.93	0.94	0.26	0.42	0.50	0.31
Compression	2000	9.23	5.00	12.5	0.98	1.87	0.09	1.07	0.6	10.85

high. Some tendency to S-shaped curves was observed; i.e., resin compression at low modulus, fiber compression at high modulus, followed by a yield point and further strain to failure.

Failure modes in the warp and fill directions are shown in Fig. 4. All specimens exhibited shear mode fractures with failures generally occurring at a 20°–30° angle across the plies of the material. Table 2 presents a comparison of shear modulus and strength data from the various test techniques discussed in this section.

Thermal Expansion

The stress state that occurs in a nozzle during a motor firing results from internal pressure and thermal expansion loads. Stresses and strains due to thermal expansion are generally most critical in nozzle design and are further complicated by the temperature-time dependence and the effects of chemical reactions on thermal expansion. Since even low thermal expansion coefficient materials undergo relatively large dimensional changes at temperatures above 4000°F, the importance of accurate thermal expansion data for high-temperature stress analysis is evident.

High-temperature thermal expansion measurements are generally made with a graphite tube dilatometer which consists of a potentiometer or dial gage that measures deflection of a push rod which rests on the specimen in a concentric tube. Specimen length determines the required instrument measurement accuracy for low expansion materials.

Buch, Zehms, and Rossi of Aerospace Corporation[11] were the first to experimentally demonstrate that heating rate greatly influences the thermal expansion characteristics of phenolic materials. The degradation of phenolic resins, which results in the release of water at temperatures less than 400°F and gases at higher temperatures, is a thermally activated process, and is a function of both time and temperature. Another variable involved is the release of gaseous decomposition products that must escape by diffusion through the resin matrix during the early stages of decomposition. From a thermochemical standpoint, heating rates control the amount of gaseous decomposition products formed as well as the amount that accumulates

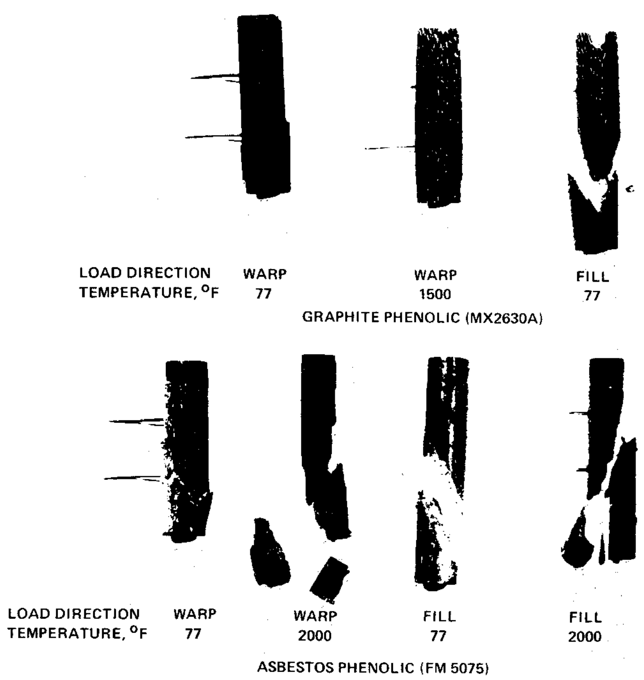

Fig. 4 Compression failures of specimens loaded in the warp and fill directions.

Table 2 Off-axis compression and shear modulus test results for graphite phenolic and asbestos phenolic composites

	Graphite phenolic MX 2630A	Asbestos phenolic FM 5075
In-plane shear modulus, 10^6 psi		
±45° off-axis	0.53 at 77°F	
	0.16 at 1500°F	
	0.38 at 2500°F	
Plate twist	0.73 at 77°F	
	0.21 at 1500°F	
	0.31 at 2500°F	
	0.12 at 4000°F	
Ultimate strength, 10^3 psi[a]		
Interlaminar shear, 1–3	14.1	19.5
Interlaminar shear, 2–3	15.9	21.9
Initial modulus, 10^6 psi[a]		
Interlaminar shear, 1–3	1.34	1.10
Interlaminar shear, 2–3	1.90	0.85
Ultimate strain, %[a]		
Interlaminar shear, 1–3	2.65	2.80
Interlaminar shear, 2–3	1.78	3.24
Calculated initial shear modulus, 10^6 psi[a]		
Interlaminar shear, 1–3	0.49	0.35
Interlaminar shear, 2–3	1.04	0.28

[a] At room temperature and 45° off-axis specimen.

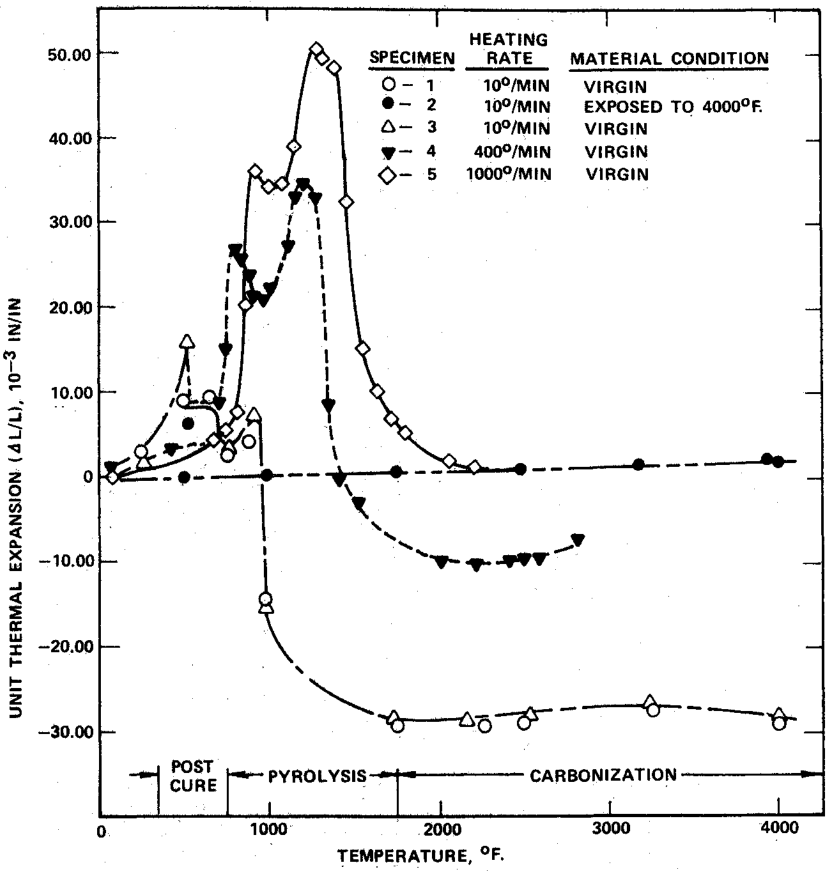

Fig. 5 Effect of heating rate on thermal expansion of graphite phenolic (MX 26 30A) in across-lamina direction.

within the resin matrix from the competing processes that generate and release gases.

The importance of heating rate on thermal expansion is shown in Fig. 5 which presents the unit thermal expansion versus temperature for the across-lamina direction (perpendicular to the warp and fill direction of the cloth reinforcement material) of graphite phenolic tested at various heating rates.

Figure 5 (Specimen 2) also shows the stabilizing nature of carbonizing the matrix system before measuring expansion at low heating rates. The graph indicates that as heating rate is increased for virgin materials, the competing gas generation and gas release processes control expansion and shrinkage of the material. High heating rates do not allow sufficient time for the gas to escape, and swelling results. Low heating rates allow sufficient time for removal of water from the material, and the material undergoes small contraction. At high temperatures, material loss by molecular bond cleavage and decomposition explains the gross contraction that accompanies heating, and further contraction is characteristic of the forming of amorphous carbon material.

Proof of phenolic gas evolution at high heating rates was demonstrated in thermal expansion tests by extensive specimen smoking at heating rates in excess of 100°F/min. This behavior provides evidence that thermal expansion is influenced by the matrix chemical reactivity and volatilization, and such volatilization (internal pressurization) possibly influences the mechanical properties of the material. Future work to evaluate the effects of high heating rate on mechanical properties may provide a solution to the thermochemical dependence apparent in the asbestos phenolic material.

Evaluation of Standard Failure Criteria for Nozzle Materials

A comprehensive survey of failure theories for both isotropic and anisotropic materials is given by Sandhu.[12] It would be convenient to be able to apply one of the existing theories to nozzle analyses. However, the statement of a criterion for a general six-dimensional or a four-dimensional stress state, which normally exists in most nozzle configurations, has not been verified by experiment. The anisotropy and lamination of the material as well as the testing difficulties of obtaining combined stress states at elevated temperatures have greatly affected progress in the selection of a suitable criterion.

In an attempt to screen candidate criteria, high-temperature, off-axis compression tests were conducted for graphite and asbestos phenolic at angles of $\theta = 20°$ and 45° with respect to the warp and fill directions. The off-axis test places the material in a biaxial stress condition and is suitable for failure criteria evaluation. The interlaminar shear strength was calculated for each test using the off-axis test and principal material strength data in each of the candidate criteria. The consistency of the criteria is judged by the predicted interlaminar shear strengths F_{13} and F_{23} for the two off-axis angles and by comparison with results from other test methods.

The strength criteria considered in this study are: maximum stress, maximum strain, maximum shearing stress, and distortional energy. The theories of these criteria are discussed in the following subsection.

Maximum Stress Theory

Failure of a laminated composite results when one of the ultimate strengths in the principal material directions is reached.

$$\begin{aligned} &\sigma_1 \leqq F_1{}^t \quad \text{or} \quad \sigma_1 \leqq F_1{}^c \\ &\sigma_2 \leqq F_2{}^t \qquad\quad \sigma_2 \leqq F_2{}^c \\ &\sigma_3 \leqq F_3{}^t \qquad\quad \sigma_3 \leqq F_3{}^c \\ &\tau_{12} \leqq F_{12} \\ &\tau_{13} \leqq F_{13} \qquad\quad \tau_{23} \leqq F_{23} \end{aligned} \tag{1}$$

where σ = applied stress and F = allowable strength.

Maximum Strain Theory

Failure by this theory is expressed in terms of strains and strain allowables in the principal material directions by the following inequalities:

Table 3 Graphite phenolic (MX 2630A) shear strength comparison

Failure criteria	Test temperature, °F 77	1500	2500	4000
1) *Interlaminar shear* (τ_{13} psi):				
Max. stress (20° off-axis)	4756	720	860	690
Max. stress (45° off-axis)	7050	1440	1500	1000
Max. shear stress (20° off-axis)	3710	720	850	680
Max. shear stress (45° off-axis)	3410	1400	1520	1000
Azzi-Tsai (20° off-axis)	6500	1000	1500	1000
Azzi-Tsai (45° off-axis)	7000	1300	1500	1000
2) *Interlaminar shear* (τ_{23} psi):				
Max. stress (20° off-axis)	4500	630	690	720
Max. stress (45° off-axis)	7950	1130	930	1090
Max. shear stress (20° off-axis)	3650	635	700	900
Max. shear stress (45° off-axis)	3730	1130	930	1000
Azzi-Tsai (20° off-axis)	7500	900	1100	1050
Azzi-Tsai (45° off-axis)	8000	1130	950	1050

$$\begin{array}{lll} \varepsilon_1 \leqq e_1{}^t \quad \text{or} & \varepsilon_1 \leqq e_1{}^c & \\ \varepsilon_2 \leqq e_2{}^t & \varepsilon_2 \leqq e_2{}^c & \\ \varepsilon_3 \leqq e_3{}^t & \varepsilon_3 \leqq e_3{}^c & \\ \varepsilon_{13} \leqq e_{13} & \varepsilon_{23} \leqq e_{23} & \varepsilon_{12} \leqq e_{12} \end{array} \tag{2}$$

where ε = applied strain and e = allowable strain.

Two features characteristic of both the preceding criteria are: a) no interaction between stresses is accounted for, and b) a failure mode is predicted according to which inequality governs the strength for the given stress state. The first feature is generally considered a shortcoming although operational simplicity is gained. The latter is useful information in the design process.

Maximum Shearing Stress Theory

The maximum shearing stress theory was originally proposed by Tresca as the yield condition of constant maximum shearing stress in isotropic, homogeneous, ductile materials. For a three-dimensional continuum, this condition can be expressed in terms of principal stresses as

$$[(\sigma_{I}-\sigma_{III})^2-4k^2][(\sigma_{III}-\sigma_{II})^2-4k^2][(\sigma_{I}-\sigma_{II})^2-4k^2]=0 \tag{3}$$

where k is the yield limit in simple shear.

During plastic flow, one of the differences has the absolute value of $2k$. Here σ_{I}, σ_{II}, σ_{III} refer to principal stresses which are not necessarily the stress components in the material symmetry directions (σ_1, σ_2, σ_3).

This theory was chosen as a candidate since failed warp and fill compression test specimens revealed shear mode fractures. The limitations of this criterion when applied to ablative phenolic materials are that the material is not isotropic and that material strengths are known only for the material principal directions. To apply the criterion to reinforced phenolics, two assumptions must be made: 1) σ_{I}, σ_{II} and σ_{III} must be principal material stresses, and 2) the criterion accounts for the interaction between the normal and shear strengths of the material.

In many fiber-reinforced materials the off-axis strength is high for low off-axis angles and decreases as the off-axis angle increases because of low strength in the across orientation. This is not the case with graphite phenolic (MX 2630A). Test results indicate that the off-axis strength of the 45° specimen is approximately 18% higher than that of the 20° specimen. This is because of the very high compressive strength in the across-lamina direction and the fact that in a woven material the "frictional" forces increase as the off-axis angle increases.

Distortional Energy Theory

This popular approach to the prediction of failure in composite materials has its roots in the yield criterion for isotropic, ductile and homogeneous metals attributed to Von Mises. Hill[13] generalized Von Mises' criterion to account for anisotropy; this modified criterion was used to describe the unidirectional fiber composite failure under plane stress. In its general form, Hill's equation is

$$F(\sigma_2-\sigma_3)^2+G(\sigma_3-\sigma_1)^2+H(\sigma_1-\sigma_2)^2+2L\tau_{12}{}^2+2K\tau_{23}{}^2+2J\tau_{13}{}^2=1 \tag{4}$$

where F, G, H, L, K, and J are material coefficients characteristic of the state of anisotropy and 1, 2, and 3 are the axes of material symmetry.

Application to composite materials was first introduced by Norris.[14] Many forms of this criteria can be found in the literature including those developed by Azzi and Tsai,[15] Hoffman,[16] Tsai[17] for the determination of the brittle strength of orthotropic materials, and Tsai and Wu[18] who have defined the newest approach. Each of these criteria functions basically in the same manner. Each, however, had a notable feature or contribution at the time of its development. The Azzi-Tsai[15] criterion is evaluated in this paper using the off-axis test data.

In contrast to the maximum stress and strain theories, interaction between stresses is considered as a constant relation. Also, no mode of failure is predicted, only its onset.

Table 4 Asbestos phenolic (FM 5075) shear strength comparison

Failure criteria	Test temperature, °F 77	500	1000	2000
1) *Interlaminar shear* (τ_{13} psi):				
Max. stress (20° off-axis)	5300	4200	1080	1200
Max. stress (45° off-axis)	9750	10,150	3210	1900
Azzi-Tsai (20° off-axis)	7000	3500	1100	1250
Azzi-Tsai (45° off-axis)	10,500	11,700	3400	1900
2) *Interlaminar shear* (τ_{23} psi):				
Max. stress (20° off-axis)	6100	5080	556	935
Max. stress (45° off-axis)	10,940	8550	1060	1150
Azzi-Tsai (20° off-axis)	9790	7930	590	1075
Azzi-Tsai (45° off-axis)	12,340	9330	1080	1175

Comparison

Results of the shear strength data obtained using all of the techniques discussed are presented in Tables 3 and 4. The strengths predicted for graphite phenolic from the off-axis tests using the Azzi-Tsai distortional energy approach appear to be the most consistent criteria. These data are also in good agreement with the shear strengths predicted by the maximum normal and shear criteria using 45° off-axis data. The 20° off-axis data are consistently lower for the maximum stress criteria. This trend was also found by Tsai[19] when these criteria were compared with experimental data for a glass-epoxy composite unidirectional laminate. A better match with the data was obtained for the distortional energy, while the maximum stress and shear criteria predicted $\theta = 20°$ off-axis strengths that were approximately twice the experimental data. Because of the large scatter in off-axis strain data, the maximum strain criteria were not evaluated at this time.

No criterion was able to consistently predict the shear strength for asbestos phenolic. It is believed that at the higher temperature, the contraction and the unique thermochemical response of the material cause stresses within the material which have not been accounted for in the criteria. More test data and detailed examination of the criteria are required before any conclusions can be drawn for a failure criteria for asbestos phenolic.

Conclusions and Recommendations

It appears that some form of the distortional energy theory will provide an adequate prediction of failure for graphite phenolic materials up to 4000°F. When both tension and compressive stress states are present, a form other than that of Azzi-Tsai will probably have to be employed.

The load-, temperature-, and time-dependence of asbestos phenolic complicates the selection of a satisfactory failure criterion. The transient response of this type of material causes severe thermochemical changes and stresses that must be accounted for in the failure criteria.

More testing to determine the load-, temperature-, and time-dependence of these materials, and test specimens which allow more general multiaxial stress states are needed before an accurate general failure criterion can be developed to predict the structural integrity of nozzle materials subject to actual firing environments.

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