Dynamic and Static Fatigue of Silicate Glasses

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Fatigue of silicate glasses was studied using both static and dynamic tests. Static fatigue data for acid-etched soda-lime silicate glass determined at 74°F in 50 and 100% rh can be represented by a single universal fatigue curve (UFC). The UFC for acid-etched glass does not lie on the UFC of Mould and Southwick, which was determined for abraded soda-lime silicate glass; the acid-etched glass was less susceptible to static fatigue. The susceptibility of acid-etched soda-lime silicate glass to static fatigue differed little from that of pristine E-glass and fused SiO_2 fibers. Dynamic fatigue data for soda-lime, E, borosilicate, and fused quartz glasses agreed well qualitatively and quantitatively with fundamental crack velocity data for these glasses; the dynamic fatigue theory of Charles was used in the comparison. The theoretical implications of these results are discussed.

I. Introduction

THE dependence of fracture stress of silicate glasses on the time of loading is of great technological importance and scientific interest. This phenomenon is commonly termed static fatigue, or, if it involves glass failure under dynamic loading conditions, dynamic fatigue. Glass loaded rapidly or forced to support a given load for a short time is found to be relatively strong, whereas it is relatively weak if loaded slowly or forced to support the load for a long time. The principal experimental and theoretical results on static fatigue have been summarized[1-4]; it is generally believed that static fatigue results from a stress-dependent chemical reaction between H_2O vapor and the surface of the glass which causes a preexisting flaw to grow to the critical dimensions for spontaneous crack propagation. The theoretical stress corrosion model of Hillig and Charles[5] is the most complete theory for static fatigue and best satisfies available experimental data.

Although the fatigue behavior of silicate glasses has been studied extensively, most of the work has been conducted with "weak" specimens which had been abraded in preparation or testing. Because of its potentially very high strength-to-weight ratio, glass is a prime candidate for use in the pressure hulls of small deep-sea submersibles.[6] Fiberglass-reinforced plastics have already demonstrated strength-to-weight ratios greatly improved over those of conventional materials and are used widely in rocket motor cases, helicopter rotor blades, and pressure vessels. Before full advantage can be taken of pristine high-strength glass, however, its fatigue behavior must be well understood on an experimental and theoretical basis. By measuring the dependence of the fracture strength of acid-etched and abraded soda-lime silicate glass on loading rate, Ritter[7] established that the dynamic stress corrosion model of Charles[8] correctly predicts the effect of dynamic loading on high- and low-strength soda-lime silicate glasses. One objective of the present study was to extend these experiments to test the predictions of the stress corrosion model regarding the effect of static loading on pristine (acid-etched) soda-lime silicate glass.

Although the effect of chemical composition on corrosion behavior of silicate glasses has been studied extensively,[9] relatively little is known about the relation between stress corrosion and composition. Thus, another objective was to elucidate more clearly the dependence of stress corrosion susceptibility on chemical composition. It was hoped that this dependence could be established by measuring the sensitivity of fracture strength to loading rate of silicate glasses and comparing these results with the fundamental crack velocity data obtained by Wiederhorn and Bolz.[10,11]

II. Experimental Procedure

(1) Dynamic Fatigue Tests

Rods of 3-mm borosilicate glass‡ and of fused quartz§ were cut to 5-in. lengths. The borosilicate rods were annealed at 565°C for 0.5 h and furnace-cooled; the fused quartz rods were not annealed because of the possibility of devitrification. Specimens of each glass were then abraded with a standard blast of No. 240 SiC grit. The grit-blast apparatus and technique were similar to those described by Mould and Southwick,[12] except that the abrader was modified by the addition of a special holder for the round specimens and a motor for rotating the specimens at 250 rpm to give uniform damage around the circumference. Nitrogen compressed at 7 psi supplied the 5-s blast. After the samples were abraded, they were allowed to age in distilled H_2O for $\approx$40 h to normalize the strength.

Shortly after the aging, groups of 20 samples were broken in 4-point bending at 7 loading rates on a testing machine¶ The bending apparatus had inner and outer spans of 0.813 and 2.808 in., respectively. The supports were ball bearings 0.750 in. in diameter, and the inner rollers were fitted with brass sleeves having a peripheral radial groove with a minimum diameter of 0.780 in. This groove enabled a specimen to sit stably on the center rollers while the outside rollers were brought into contact with it.

The samples were wetted with distilled H_2O immediately before the test. For brief loadings, one dipping was sufficient to wet the samples for the duration of the test; for longer times, the samples were rinsed periodically with distilled H_2O during the test. The fracture stress was computed by the well-known simple bending formula.

(2) Static Fatigue Tests

Soda-lime silicate glass‖ rods 3 mm in diameter were cut to 6-in. lengths and annealed at 500°C for 1 h. Following a procedure similar to that of Proctor,[13] the specimens were etched in an aqueous 15% HF-15% H_2SO_4 (by volume) solution until $\approx$0.025 in. was removed from the diameter; this amount of etching should be adequate to give maximum strength.[13] After the diameter of each rod was measured at one end with a micrometer, the acid-etched samples were either placed in a

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desiccator or tested immediately. The samples were generally stored in the desiccator no longer than 5 days; this storage was shown to have no detrimental effect on strength.

A static fatigue testing station is diagramed in Fig. 1. Four stations were constructed to fit inside a controlled environmental chamber,* so that 4 samples could be tested simultaneously. To prevent accidental shock when a sample failed, the stations were mounted on rubber pads, and the loading arm of each station was caught by a rubber bumper after a specimen fractured. The stations were separated by plastic shields.

The loading arm was 12 in. long, providing a nominal mechanical advantage of 6.857:1. The inner and outer spans were 1.312 and 3.50 in., respectively. The outer supports were stainless-steel rollers 0.750 in. in diameter having a 0.125-in. radial groove 0.621 in. in diameter. The grooves enable a glass rod to rest stably in the center of the roller and align it accurately during testing. The inner rollers are sealed stainless-steel bearings 0.625 in. in diameter. The load and support rollers are pinned to allow the glass rod to deflect with a minimum of friction at the supports. In addition, the load rollers are attached to an Al block which is pinned so that it can pivot in the loading carriage. As the arm is lowered, the inner rollers adjust to a horizontal position when they contact the glass rod and remain essentially horizontal as the glass deflects. Thus, approximate 4-point loading is maintained at all deflections, and any slight deviation of the arm from the horizontal in the fully deflected position is compensated for. The load carriage, which is aligned perpendicular to the loading arm by a brass pin and carriage bolt, can be adjusted so that the loading arm is always horizontal when the samples are fully deflected, regardless of the applied load.

Each station was timed individually by an electric clock that could record time in tenths of a second. The clocks were controlled by microswitches which sensed the position of the loading arm during loading and after fracture. Loads were applied by lowering the loading arm with an externally connected pulley arrangement. The weights, which were from commercially available kilogram sets having a tolerance of 1.0 g/kg, were enamel coated to prevent corrosion. Stresses were calculated using the nonlinear bending analysis of Vrooman and Ritter.[14]

In a test, the desired temperature and humidity were established in the environmental chamber; the glass samples were then placed in position. When the desired temperature and humidity were reestablished, the specimens were loaded, and the timers were started. Temperature was controlled at 74°±2°F, and the actual humidities obtained during the nominally 100%-rh runs were 98 to 100%; for the 50%-rh runs, they were from 50 to 58%.

III. Results

The experimental data on the dynamic fatigue of abraded borosilicate glass and fused quartz are summarized in Fig. 2. The straight lines in these log-log plots were calculated by the least-squares method, with the slope as indicated.

The static fatigue results for acid-etched soda-lime silicate glass are summarized in Table I. To characterize the failure time for a given stress level, the median time was chosen so that both the immediate failures (<0.0025 min) and the nonfailed specimens could be taken into account. Figure 3 is a semilog plot of the static fatigue data with a least-squares straight line drawn through each set.

The static fatigue data may be normalized in the reduced units of the universal fatigue curve (UFC) of Mould and Southwick.[15] The reduced stress is defined as σ/σ_N, the failure stress divided by the failure stress at liquid N temperature, and the reduced time is defined as log $(t/t_{0.5})$, where t is the failure time and $t_{0.5}$ the failure time at ½ the liquid N stress. The liquid N strength for similarly acid-etched soda-lime silicate glass has been determined to be 470,500 psi.[16] The failure time at ½ the liquid N strength found from Fig. 3 is 8.92×10^{-4} min for 100% rh and 0.148 min for 50% rh. Figure 4 shows that all the static fatigue data can be represented by one UFC with a slope of −0.031.

*Model MC08, Develco Inc., Norwalk, Conn.

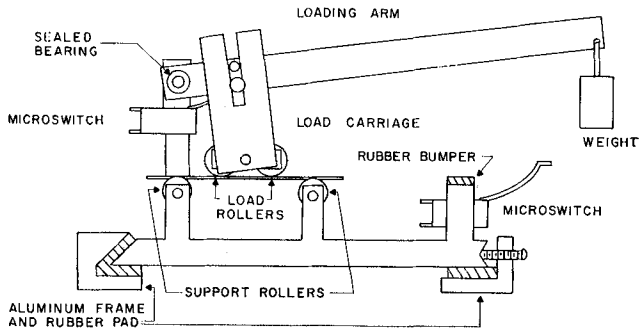

Fig. 1. Diagram of static fatigue testing equipment.

IV. Discussion of Results

(1) Dynamic Fatigue

To describe flaw growth under dynamic loading, Charles[6] assumed that flaw growth at a constant temperature is described by an equation of the form

$$v_x = k(\sigma_m)^n e^{-A/RT} \qquad (1)$$

where v_x = velocity of the flaw tip in the x direction (the direction of the maximum tensile stress gradient), σ_m = normal tensile stress at the flaw tip, A, k, and n = constants, R = gas constant, and T = absolute temperature. By using Inglis' stress concentration relation[17] and Orowan's criterion[18] of brittle frac-

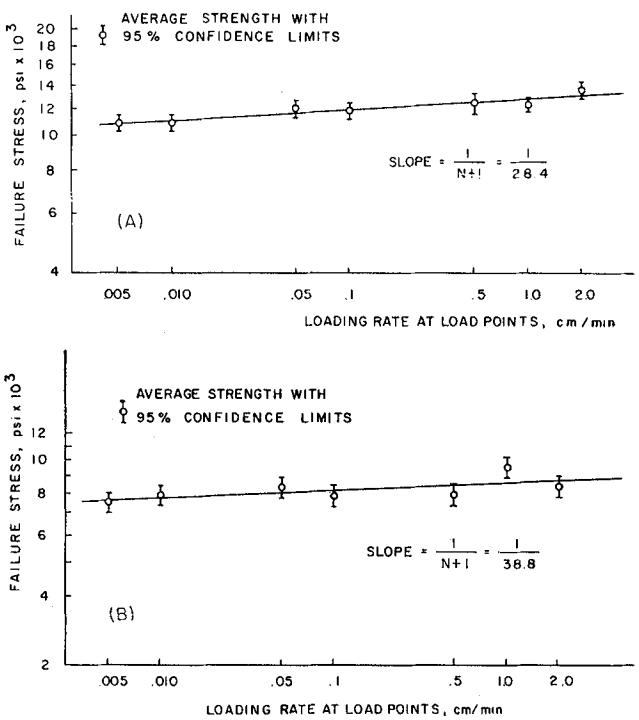

Fig. 2. Effect of loading rate on room-temperature strength of wet-tested (*A*) abraded borosilicate glass and (*B*) fused quartz.

Table I. Static Fatigue of Acid-Etched Soda-Lime Silicate Glass at 74°F

Stress (psi×10³)	No. of samples failing within indicated time (min) decade									No. tested	Median failure time (min)
	<3×10⁻³	10⁻²	10⁻¹	10	10¹	10²	10³	10⁴	10⁵		
						100% rh					
250	47	13	0	2	0	0	0	0	0	62	0.0016[a]
230	49	21	9	5	7	2	4	0	0	97	0.0025
210	13	8	5	8	9	6	1	0	0	50	0.0405
200	5	8	10	12	16	4	1	1[b]	0	57	0.2141
180	9	12	1	5	8	11	6	9[c]	0	61	2.5500
170	2	2	0	4	15	11	7	16[b]	3[d]	60	40.7766
160	0	0	0	0	0	1	1	1[b]	0	3	
150	0	1	0	0	0	0	2	15[b]	4[b]	22	Fatigue limit
						50% rh					
270	11	3	1	1	2	1	0	0		19	0.0025
250	8	39	13	11	10[e]	4	4	6[b]		95	0.0141
230	12	9	8	2	5	3	4	7[b]		50	0.0437
215	1	2	4	8	11	9	3	2[b]		40	2.6995
200	0	3	2	6	9	6	10	13[b]		49	82.3466
180	0	0	0	1	0	0	0	6[f]		7	
170	0	0	0	0	0	1	0	4[g]		5	
160	0	0	1	0	0	0	0	14[b]		15	Fatigue limit

[a]Time less than min. accurate timing for apparatus.
[b]Tests terminated; samples survived.
[c]2 samples failed and 7 survived when tests were terminated.
[d]1 sample failed and 2 survived when tests were terminated.
[e]1 sample had test terminated.
[f]2 samples failed and 4 survived when tests were terminated.
[g]2 samples failed and 2 survived when tests were terminated.

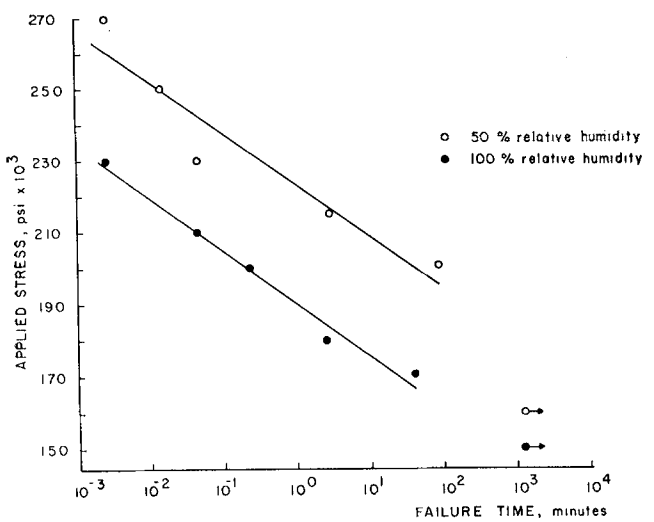

Fig. 3. Static fatigue curves of acid-etched soda-lime silicate glass at 74°F at 50% and 100% rh. Data points with arrows denote fatigue limit.

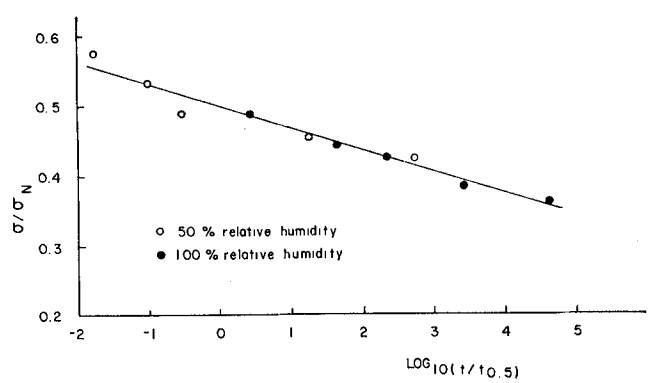

Fig. 4. Universal static fatigue curve of acid-etched soda-lime silicate glass at 74°F.

ture, Charles deduced that the fracture strength at a constant temperature should vary with the rate of stress application as:

$$\sigma_{af} = K\beta^{\frac{1}{n+1}} \qquad (2)$$

where σ_{af} = macroscopic applied stress at which failure occurs, β = rate of stress application, and K = constant. Thus, the constant n, which is a measure of the stress-corrosion susceptibility for a given glass, can be evaluated experimentally from the slope of a log-log plot of failure stress vs loading rate such as Fig. 2. The n values for various silicate glasses are given in Table II.

Wiederhorn and Bolz[10,11] measured the dependence of crack velocity in many silicate glasses on stress in distilled H_2O. Crack velocity, which depends on chemical composition, fits the empirical equation:

$$v = v_0 \exp(-E^* + bK_I)/RT \qquad (3)$$

where v_0, E^*, and b are empirical constants and K_I is the stress intensity factor, which is proportional to the stress at the crack tip. Wiederhorn and Bolz[11] further showed that the crack ve-

Table II. Comparison of Dynamic Fatigue and Crack Velocity Data

Glass	Surface condition	n	Ref.
	Dynamic fatigue data		
Soda-lime silicate	Acid-etched and abraded	13.0	7
Soda-lime silicate	Abraded	16.0	8
E-glass*	Pristine fiber	27.0	19
Fused quartz*	Abraded	37.8	Present study
Borosilicate	Abraded	27.4	Present study
	Crack velocity data		
Soda-lime silicate		16.6	11
Aluminosilicate I*		27.4	11
Fused silica*		36.1	11
Borosilicate		34.1	11

*E-glass and aluminosilicate I can be compared because they have similar compositions; this is also true for fused quartz and fused silica.

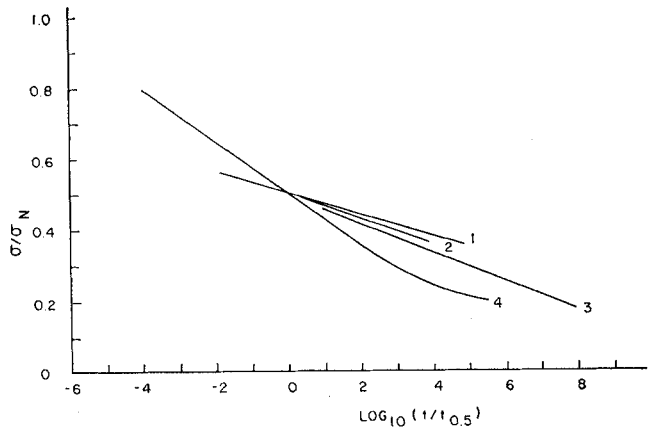

Fig. 5. Universal fatigue curves of (1) acid-etched soda-lime silicate glass, present study; (2) E-glass fibers, data from Ref. 23 in which liquid N strength of 820,000 psi, given in Ref. 19, was used; (3) fused SiO_2 fibers, data from Ref. 22; and (4) abraded soda-lime silicate glass, Ref. 15.

locity data represented by Eq. (3) agreed quite well with the stress corrosion theory developed by Hillig and Charles[5] and with the static fatigue data for abraded soda-lime silicate glass and fused silica fibers.

It would be extremely valuable to be able to relate the fundamental crack velocity parameters, E^* and b, to the empirical stress corrosion constant (n) determined from the sensitivity of fracture strength to loading rate; however, an analytical solution to this problem is not known. Alternatively, the crack velocity data can be used to measure n independently since, according to Eq. (1), a log-log plot of the crack velocity data vs stress intensity factor should be a straight line with the slope n. This procedure was followed using Wiederhorn's original data,[20] which fitted a log-log plot well, especially at the higher velocities. The results of these calculations are given in Table II.

The n values computed from dynamic fatigue tests and those from crack velocity data agree reasonably well both qualitatively and quantitatively. This agreement suggests that crack propagation by stress corrosion is important in the failure of glass under dynamic loading. Also, these results show that the dynamic fatigue technique can be used to determine the relative susceptibility to stress corrosion of silicate glasses, since the slope of the plot of strength vs loading rate correlates with stress corrosion resistance. This conclusion is true for both abraded and pristine glasses.

(2) Static Fatigue

Figure 3 shows that at any given load duration the strength increases with decreasing rh (decreased availability of H_2O to the glass surface). The similarity in the slopes of these curves indicates that the data could fit a single UFC (Fig. 4). These results show that the rate at which static fatigue occurs (as measured by the characteristic duration, $t_{0.5}$) depends on the availability of H_2O; however, the basic interaction between glass and H_2O that reduces the strength (as measured from the slope of the static fatigue curve) does not. Similar static fatigue results were observed for abraded soda-lime silicate glass[21] and pristine E-glass fibers.[4] Thus, it can be concluded that the rate of static fatigue depends on relative humidity but that the basic mechanism of static fatigue does not. This conclusion agrees qualitatively with Wiederhorn and Bolz's formulation[11] of the UFC from basic crack velocity data; however, they deduced that the value of $t_{0.5}$ should be inversely proportional to rh. The experimental dependence of $t_{0.5}$ on rh is much greater than this, demonstrating a deficiency in the stress corrosion model.[5]

The UFC of acid-etched soda-lime silicate glass is compared to those of other silicate glasses in Fig. 5. Static fatigue curves for E-glass were also determined by Otto[4] and by Schmitz and Metcalfe.[24,25] Since the latter curve had a slightly greater slope than that determined by Hollinger *et al.*[23] and since Otto's curve had a slightly smaller slope, the curve determined by Hollinger *et al.* was chosen as representative of the static fatigue curve for E-glass fibers. Figure 5 shows little if any differences in susceptibility to static fatigue among the high-strength silicate glasses despite large differences in chemical composition. Also, the susceptibility (slope of the UFC) for abraded soda-lime silicate glasses is about twice as great as that for the pristine glasses. These results cannot be explained by the stress corrosion model[5] and contrast with those for dynamic fatigue, suggesting either that stress corrosion is altered in static fatigue of pristine glasses or that the basic mechanism of strength deterioration in static fatigue differs for pristine glasses. Two explanations are possible in line with this reasoning: (1) The kinetics of the stress corrosion mechanism and the local environment at relatively perfect surfaces may differ considerably from those found at the tips of surface cracks or (2) the results may reflect differences in the basic interaction of H_2O with relatively perfect and severely damaged glass surfaces.

Wiederhorn[1] suggested that stress corrosion behavior differs between silicate glasses because the hydroxyl ion concentration at crack tips is governed by the buffering nature of the glass surface. Buffering reactions would be expected to be more effective for deep cracks than for shallow cracks on relatively perfect surfaces because of the small volume of solution at the crack tip and the long diffusion distance necessary to equalize bulk and crack-tip environment. Evidence that the general chemistry within the stress corrosion crack differs considerably from that of the bulk solution has been given for the stress corrosion cracking of metals.[26,27]

Metcalfe and Schmitz[25] proposed that the mechanism of static fatigue in E-glass fibers is ion exchange between alkali metal ions in the glass and hydrogen ions from either absorbed moisture or aqueous solution that generates tension in the surface layer. This process is the reverse of that commonly used to strengthen glass chemically. These workers based their proposal on observations that tests in aqueous solutions showed that acidic solutions of low pH were more detrimental to strength than alkaline solutions. Addition of Na^+ ions to an acidic solution reduced the detrimental strength effects, and strength lost in acid could be restored by subsequent immersion in a solution containing Na^+ ions. Variation of the Na content of E-glass from 5 to 0.031% did not change the basic mechanism of strength loss. Metcalfe and Schmitz also noted the similarity of the static fatigue curves of fused silica and E-glass fibers and thought that it might result from trace amounts of Na in the fused SiO_2.

To determine which process is responsible for the strength loss in the static fatigue of pristine glasses will require additional data on Na^+–H^+ exchange and more detailed knowledge of the stress-corrosion process at the advancing edge of the stress corrosion crack. It is entirely possible that stress corrosion and Na^+–H^+ exchange occur simultaneously. The dominant process would depend on the nature of the flaws and the local environment at the crack tip.

V. Conclusions

(1) Dynamic fatigue data for silicate glasses can be compared favorably with fundamental crack velocity data using the dynamic fatigue theory of Charles.

(2) Static fatigue curves of acid-etched soda-lime silicate glass determined at 74°F in 50 and 100% rh can be represented by one universal fatigue curve. The results also showed that the rate at which static fatigue occurred was enhanced by an increase in the availability of H_2O.

(3) The universal fatigue curve for acid-etched soda-lime silicate glass does not lie on that for abraded soda-lime silicate glass; the acid-etched glass is less susceptible to static fatigue.

(4) The static fatigue susceptibilities, as measured from the universal fatigue curve, of pristine silicate glasses (soda-lime, E-glass, and fused silica) differ only slightly.

(5) The stress corrosion model cannot account for the static fatigue of pristine silicate glasses. This inadequacy is probably related to differences between the fatigue processes that occur at the tips of deep cracks as opposed to shallow cracks in relatively flaw-free surfaces.

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Structure and Mechanical Properties of Codeposited Pyrolytic C-SiC Alloys

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Alloys of pyrolytic carbon and silicon codeposited in beds of fluidized particles were investigated. X-ray diffraction measurements revealed only two phases, turbostratic C and β-SiC. Since the C crystallites are oriented randomly, the C was macroscopically isotropic, with apparent crystallite sizes in the range found for pure C deposited under similar conditions. Transmission and scanning electron microscopy showed that the SiC was generally distributed as small particles which tended toward larger particles and continuous laminations at high SiC concentrations. The fracture stress and Young's modulus were measured in 3-point bending; they increased continuously with increasing SiC content from values for pure isotropic carbons to values for chemically-vapor-deposited SiC.

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I. Introduction

CARBONS obtained by chemical vapor deposition, generally called pyrolytic carbons, have been studied extensively.[1] These materials, obtained from the decomposition of a hydrocarbon gas either near a heated stationary substrate or in a heated bed of fluidized particles, generally differ from graphite in that there is no order between adjacent layer planes. The term "turbostratic" has been used to describe this structure. The carbons deposited on a stationary substrate have flexural fracture strengths of 10 to 30 ksi and are highly anisotropic because of preferred orientation of the crystallites. The carbons deposited on a bed of fluidized particles have a wide range of structures and properties, depending on the deposition conditions. The present work was concerned particularly with the macroscopically isotropic carbons de-