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### Abstract

The elastic constants, strength, fracture toughness, slow crack growth parameters, and mirror constant of a high lead glass supplied as tubes and funnels were measured using ASTM International (formerly ASTM, American Society for Testing and Materials) methods and modifications thereof. The material exhibits lower Young's modulus and slow crack growth exponent as compared to soda-lime silica glass. Highly modified glasses exhibit lower fracture toughness and slow crack growth exponent than high purity glasses such as fused silica.

# Introduction

The Exobiology on Mars program is a series of European Space Agency missions to understand if life ever existed on Mars. NASA is contributing scientific, engineering, and technical expertise to international efforts to explore the Red Planet. NASA's participation in the 2018 Exobiology on Mars Rover mission includes providing critical elements to the premier chemical analysis instrument on the rover, the Mars Organic Molecule Analyzer (MOMA). Some of the MOMA components are very brittle and require reliability analysis. This report provides results of mechanical testing and fractography of a high lead glass used in the MOMA system.

#### **Test Materials**

The test material was readily available as system subcomponents in the geometry of tubes and funnels with a curved spout. The cross section of the tubes, shown in Figure 1, consisted of six trapezoidal cavities arranged to form a central hexagon. Various regions of the tubes and funnel were either abraded, unabraded or metallized, thereby creating multiple surface finishes and flaw populations. Elastic modulus and strength tests were perform with the tubes, whereas slow crack growth and inert strength tests were performed with the funnels. With the exception of the test geometries, the tests nominally conformed to ASTM International standards C1289, C1368, and C1421.

#### **Mechanical Properties**

#### **Elastic Modulus**

The elastic modulus was measured using impulse excitation in accordance with ASTM International C1259 with the exception of the previously described cross section. In order to account for the influence of cross section, the differences in inertia and density as determined via finite element analysis (FEA) were used in the calculations. The measured Young's modulus is  $E = 58.5\pm0.6$  GPa, which is much lower than that of soda-lime silicate, but comparable to that of other high lead glasses (Refs. 1 and 2). Figure 2 shows the results as a function of bulk density. Because of the different sections of the specimens, it is likely that the apparent variation in bulk density is a result of geometric effects rather than real material density variation.

#### **Fracture Strength**

The fracture strength,  $S_{f}$ , was measured in four-point bending with 10 and 20 mm spans. The strength was calculated from

$$S_f = \frac{Mc}{I} \tag{1}$$

where M is the applied moment, c is the distance to the surface from the neutral axis, and I is the moment of inertia of the cross section as estimated with FEA. The resultant Weibull parameters are summarized in Table 1 and plots are show in Figures 3 and 4 for all of the data and the data censored into groups by fracture location: in the abraded region, the metallized region, or the unabraded region. Failure typically occurred in the abraded region and the Weibull statistics are similar for all fracture locations. Although the Weibull distribution provides an adequate fit, the log normal distribution provides a better fit, Figure 5.

#### **Fracture Mirror Constants**

The fracture mirror radii associated with the mist and hackle regions were measured on thirty of the strength specimens. Figures 6 to 9 show example fracture surfaces, which were frequently asymmetric. Fracture was the result of surface damage, shown in Figure 10, in the form of scratches, checks, and gouges. Because the strengths were narrowly distributed, the mirror constants were estimated point wise from, rather than by curve fitting of, the function

$$S_f = \frac{A_i}{\sqrt{r_i}} \tag{2}$$

where  $A_i$  is the particular boundary constant and  $r_i$  is the corresponding boundary radius. The resultant mirror-mist and mist-hackle constants were  $A_M = 1.4 \pm 0.1$  and  $A_H = 1.6 \pm 0.1$  MPa $\sqrt{m}$ .

#### **Fracture Toughness**

Fracture toughness was estimated from the size of the failure origins on fracture strength specimens exhibiting relatively symmetric mirror patterns along with the Newman-Raju equations (Ref. 3). Use of a stress intensity factor coefficient solution of a cracked rod (Ref. 4) gave similar results. The measured value of  $K_I = 0.57 \pm 0.03$  MPa $\sqrt{m}$  is similar to that of the other highly alloyed glasses (Ref. 5) shown in Table 2 (0.50 to 0.75 MPa $\sqrt{m}$  in air).

#### **Slow Crack Growth**

Slow crack growth properties were measured by method ASTM International C1368 with the exception of the specimen used: instead of standard beams or disks, funnels were tested by applying a bending moment via loading at the edge of the funnel mouth, thereby allowing the inherent crack growth mechanisms to be exhibited. Failure typically occurred in the abraded region of the funnel stem, as shown in Figure 11. The symmetry and cantilever curl exhibited on the fracture surface imply that simple bending was achieved in the tests, Figure 11(c). The resultant fracture strength is shown in Figure 12 and the crack velocity curves are shown in Figure 13 for the power and exponential functions, given respectively as

$$v = \frac{da}{dt} = A_1 K_I^{n_1} = A_1^* \left[ \frac{K_I}{K_{IC}} \right]^{n_1}$$
(3)

and

$$v = \frac{da}{dt} = A_2 \exp\left(n_2 K_I\right) = A_2 \exp\left(n_2^* \frac{K_I}{K_{Ic}}\right)$$
(4)

where v, a, and t are crack velocity, crack size, and time, respectively. Constants A and n are the material/environment dependent SCG parameters, and  $K_I$  and  $K_{IC}$  are, respectively, the Mode I stress intensity factor and the critical stress intensity factor or fracture toughness. The parameters are summarized in Table 3. The MOMA glass exhibits similar cracks growth parameters as Corning 0120, another high lead glass.

All of the funnel test specimens used to estimate SCG parameters failed in the abraded region of the constant moment section. However, it is noteworthy that during testing in air, several specimens failed at low stress due to large, spurious flaws located in the metallized section of the spout. When testing in dry nitrogen, the problem was exacerbated and failure frequently occurred in the metallized region. The spurious flaws can only be eliminated via inspection or proof testing.

## **Summary**

The strength, fracture toughness, slow crack growth parameters and Young's modulus of a high lead glass were measured. The high lead glass exhibits lower fracture toughness and is more susceptible to slow crack growth than soda-lime silicate. In general, glasses modified with oxides such as PbO and BaO exhibit lower fracture toughness and are more susceptible to stress corrosion than high purity glasses such as fused silica (n = 40) (Ref. 5).

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Failure	Number	Average	Standard	CV,	Weibull	$\sigma_{\theta}$
location	tested	S <sub>f</sub> (MPa)	deviation	percent	т	MPa
All	40	61.3	5.3	9	14.1	63.5
Unabraded	6	61.1	8.0	13	10.6	74.5
Metallized	6	61.6	5.2	8	14.0	71.5
Abraded	28	61.2	4.9	8	14.8	64.8

TABLE 1.—STRENGTH STATISTICS OF SPIRAL TUBES

TABLE 2.—FRACTURE TOUGHNESS (MPa√m) OF GLASSES (REF. 5)

Material	Environment			
	Air (%RH/°F)	Dry N <sub>2</sub>		
MOMA	0.57±0.03 (30/74)	0.69±0.01		
Corning 0120	0.50±0.02 (34/76)	0.67±0.02		
Electro-Glass 2164	0.61±0.05 (32/73)	0.74±0.03		
Schott S8061	0.64±0.01 (23/73)	$0.72 \pm 0.02$		
Schott S8070	1.57±0.03 (60/73)	1.90±0.03		
Schott 8330	0.61±0.04 (60/73)	$0.72 \pm 0.04$		
Soda lime silicate	0.75±0.04 (35/73)	0.80±0.01		
Ba-doped	0.72±0.002 (23/73)	0.76±0.01		
Silica, 7980	0.73±0.04 (45/75)	0.77±0.02		

TABLE 3.—SUMMARY OF SLOW CRACK GROWTH (SCG) PARAMETERS FOR GLASSES (REF. 5)

Material and relative	п	В	A	$n_2$	$A_2$	# Tested
humidity		MPa <sup>2</sup> ·s	m∕s (MPa√m)⁻n	(MPa√m) <sup>−n</sup>	m/s	
MOMA, 30% RH	19.5±2.1	2.5	$1.9 \times 10^{+1}$	51.5	2.3×10 <sup>-16</sup>	25
0120, 95% RH	17.0±3.1	0.6	$2.4 \times 10^{+1}$	49.2	5.3×10 <sup>-15</sup>	36
0120, 2% RH	23.2±5.3	4.3	$2.8 \times 10^{+1}$	61.1	4.2×10 <sup>-19</sup>	30
2164, 95% RH	12.9±1.1	6	2.3×10 <sup>-1</sup>	36.7	1.7×10 <sup>-13</sup>	65
2164, 2% RH	22.1±3.9	39	3.0×10 <sup>-1</sup>	36.6	1.7×10 <sup>-16</sup>	48
S8070, 95% RH	19.8±2.6	60	4.9×10 <sup>-9</sup>	16.1	3.9×10 <sup>-16</sup>	25
S8070, 2% RH	25.0±3.9	3,079	2.6×10 <sup>-12</sup>	17.1	2.8×10 <sup>-19</sup>	25
8330, 95% RH	17.1±1.3	5	5.6×10 <sup>-1</sup>	40.8	6.4×10 <sup>-15</sup>	25
8330, 3% RH	24.5±3.9	19	$8.0 \times 10^{-1}$	38.1	1.6×10 <sup>-16</sup>	30
8330, 1% RH	30.0±3.6	2,855	2.1×10 <sup>-2</sup>	38.3	1.8×10 <sup>-18</sup>	30
Soda Lime Silicate, H2O	20.1±0.9	5	7.5×10 <sup>-1</sup>	45.1	8.6×10 <sup>-17</sup>	30
7980, Fused Silica, H2O	39.3±3.2	0.00013	1.1×10 <sup>+7</sup>	88.3	1.2×10 <sup>-24</sup>	50



Figure 1.—Fracture cross section of strength test specimens.



Figure 2.—Dyanmic modulus and density.



Figure 3.—Weibull distribution for all the data, as determined with rank regression.



Figure 4.—Weibull distributions censored by fracture location.



Figure 5.—Log normal distribution for all the data.



Figure 6.—Fracture origin in SN – 0035.



Figure 7.—Fracture origin in SN – 0039.



Figure 8.—Fracture origin in SN – 0040.



Figure 9.—Fracture origin in SN – 0031.



Figure 10.—Surface damage on tubular strength test specimens.



Figure 11.—Slow crack growth test specimen: (a) finite element analysis of test configuration, (b) fractured test specimen, and (c) and (d) fracture surfaces.







Figure 13.—Crack velocity of high lead glasses as a function of stress intensity for (a) power law and (b) for the exponential law.