MiniSShot

Ameron Fiberglass/epoxy Pipe – Experimental Measurement of Glass Transition Temperature

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Introduction

Ameron Dualoy 3000/L fiberglass pipe (in short Ameron) has been successfully used as the MiniSShot motor casing. This type of tube was chosen for several reasons including availability, standard production, heat resistance and high strength to weight ratio. These commercially available pipes are manufactured in sizes ranging from 2-16 inch (50-400cm) nominal diameter (see table 1). This same type of pipe is also being considered for the DoubleSShot and ExSShot motor casings.

Ameron 3200 series pipes are rated by the manufacturer as class 200 fire protection and general industrial services pipes. These pipes are used in many standard applications and are rated by the manufacturer for 200 psig static working pressure (@150ºF)and ultimate RT burst pressure ranging from 1200 to 3200 psig (see table 2). According to the manufacturer brochure the tube structure is designed so that there is an external epoxy layer on top of the filamented epoxy. This protects the filamented area. The tube (at least 60% by weight) is also constructed of a continuous glass fiber and epoxy in a dual angle pattern to obtain an optimal pressurized strength. Based on the manufacturers MSDS, it is believed that the epoxy matrix is bisphenol-F based epoxy/novolac resin The pipe has an internal 0.025" protective resin liner.
The pipe is factory inspected to make sure it suffers from no defects such as pinholes, resin starved areas, delaminations, foreign inclusions, indentations or bubbles. Pipes are routinely quality control tested

Table 1.

Nominal Pipe Size		Pipe Outside Diameter[1]		Pipe Inside Diameter		Wall Thickness Total		Wall Thickness Structural	
(in)	(mm)	(in)	(mm)	(in)	(mm)	(in)	(mm)	(in)	(mm)
2	50	2.38	60	2.22	56	0.080	2.1	0.060	1.5
3	80	3.50	90	3.33	85	0.085	2.2	0.065	1.7
4	100	4.51	115	4.33	110	0.090	2.3	0.070	1.8
6	150	6.65	169	6.41	163	0.120	3.0	0.100	2.5
8	200	8.61	219	8.31	211	0.150	3.8	0.125	3.2
10	250	10.78	274	10.44	265	0.170	4.3	0.145	3.7
12	300	12.71	323	12.31	313	0.200	5.1	0.175	4.4
14	350	14.45	367	14.03	356	0.210	5.3	0.185	4.7
16	400	16.52	420	16.06	408	0.230	5.8	0.205	5.2

1) Typical outside diameters of 2 through 12-inch pipe are within API, ASTM and ANSI fiberglass and steel pipe dimensions.

Table 1: Ameron tube sizes [source: Ameron product PDF file]

Table 2.

Nominal Pipe Size		Static Pressure Rating		Ultimate[1] Internal Pressure				Ultimate[2] Collapse Pressure			
				Pipe		Coupling[3]		80°F 27°C		150°F 66°C	
(in)	(mm)	(psig)	(bar)	(psig)	(bar)	(psig)	(bar)	(psig)	(bar)	(psig)	(bar)
2	50	200	13.6	3200	215	3200	215	145	10.0	125	8.6
3	80	200	13.6	2400	160	2400	160	50	3.4	45	3.1
4	100	200	13.6	2000	135	2000	135	40	2.8	35	2.4
6	150	200	13.6	2000	135	1500	100	35	2.4	30	2.1
8	200	200	13.6	1200	80	800	55	25	1.7	21	1.4
10	250	200	13.6	1200	80	800	55	18	1.2	12	0.8
12	300	200	13.6	1200	80	800	55	12	0.8	9	0.6
14	350	200	13.6	1200	80	800	55	10	0.7	7.5	0.5
16	400	200	13.6	1200	80	800	55	10	0.7	7.5	0.5

1) Quality control minimum
2) For vacuum service above ground in sizes 8 inches and above consult Ameron.
3) Pronto-Lock and Pronto-Lock II mechanical coupling

Table 2: Ameron tube performance [source: Ameron product PDF file]

Goal

The goal of this experiment is to obtain an estimate of the Glass Transition Temperature (Tg) of the type of Ameron tubing used for the MiniSShot rocket motor. The Glass Transition Temperature is the point where the behavior of a polymer will fairly rapidly change from glassy to rubbery. This change in behavior is evidenced by a sharp decline in elastic modulus (stiffness) as the ambient temperature is increased. This is a result of the molecular chains of the polymer having enough free movement to slide past each other when a force is applied. Ironing of synthetic fabrics is a common, everyday example of heating a polymer beyond its Glass Transition Temperature to achieve a useful function (eliminating wrinkles).

Loss of stiffness that accompanies the transition from glassy to rubbery state is potentially detrimental to the mechanical strength of a glass reinforced polymer (particularly compression strength). As such, it is useful to know the value of Tg.

Once an experimental value of Tg is determined, it may then be compared to published Tg values for epoxy/novolac resins. The goal of this comparison is to support the assumption that the Ameron resin matrix is produced from this type of resin.

Materials and Methods

Ameron Dualoy 3000/L fiberglass pipe, 3.5” OD, nominal thickness 0.090” (0.065” structural + 0.025 epoxy liner on inside). Material (according to the manufacturer MSDS) appears to be glass filament reinforced bisphenol-F based epoxy/novolac resin.

A specimen was cut of dimensions 2.35 cm wide by radial length of 9.0 cm.
The sample was heated by immersion in oil using an electric deep fryer filled with approximately 1 liter of vegetable oil (oil depth approx. 7 cm). Temperature measurement was taken with a scientific mercury immersion thermometer rated to 500F.

Results

The specimen was placed into the oil as shown in Figure 1. Starting at a temperature of 240ºF, a hand-applied force was briefly applied to the specimen using a metal rod (Figure 2), and the response to the load observed. This was continued at 10ºF increments until the response was no longer elastic springback. The specimen buckled when force was applied at 340ºF. This is taken to be the Glass Transition Temperature (Tg). Results are shown in Table 3.

Temp [ºF\	[ºC]	Behaviour under applied load
240	116	Elastic
250	121	Elastic
260	127	Elastic
270	132	Elastic
280	138	Elastic
290	143	Elastic
300	149	Elastic
310	154	Elastic
320	160	Elastic
330	166	Elastic
340	171	Buckled

Table 3: Loading response of the Ameron tube specimen at varying temperatures.

Figure 1: Experimental setup with the sample immersed in the oil. Top view.

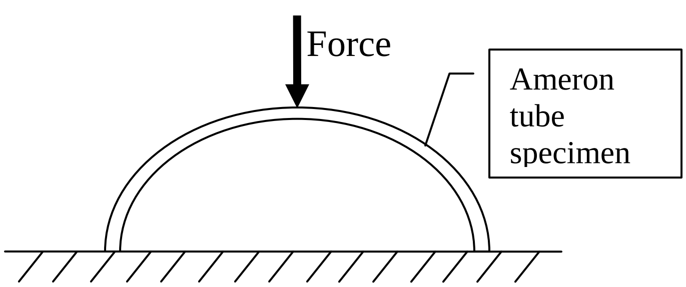

Figure 2: Method of applying force on the Ameron tube specimen.

Figure 3. An image of the tested Ameron tube specimen after buckling under the force during heating [left] compared to an untreated sample [right].

The results of the Tg measurement is consistent with published values of Tg for presumably similar epoxy compounds (e.g Dow D.E.R. 354, also a bisphenol-F based epoxy/novaloc resin). See Table 4.

Resin	Curing Agent	Tg, °F (°C)
D.E.R. 354	MTHPA	264 (129)
	Dicy	259 (126)
	NMA	315 (157)
	DETDA	273 (134)
	DDS	351 (177)
	DACH	270 (132)

Table 4:Glass transition temperatures (Tg) of resin formulas [Source: Dow Chemical Company datasheet “Epoxy Novolac Resins – High Temperature, High performance Resins”

Conclusions

A tentative Glass Transition Temperature (Tg) measurement was successfully obtained using this simple experimental setup. A comparison of the measured Tg value compares well to published values for epoxy/novolac resins.

Future plans

[Editor notes]
In order to accurately identify the Tg of this tube or similar tubes there are some changes that need to be done in the experimental setup, as follows.

1. Samples of the tube must be made in the same manner and in the same size.
2. Three samples will be inserted into the cold oil bath and warmed up to a single tested temperature, to avoid a heat shock.
3. After reaching the desired temperature and allowing some time to stabilize it a measured and constant force (e.g. a lead weight) will be used to test the buckling of the samples.
4. The force used should also be compared to the force needed to buckle identical samples at room temperature (i.e. how much force is needed at 340ºF (171ºC) to buckle the sample compared to the force needed at room temperature.
5. Since the preliminary test has shown clear results there is only a need to test the manufacturer listed Tg as well as the experimental shown Tg and a few more (say up to 30º) temperature points below thus testing a fairly small range of temperatures.
6. According to the manufacturers' data sheet the Ameron tube also tends to considerably expand while being heated. This expansion should also be measured and taken into account in the rocket motor design.
7. Other types of high temperature resin tube should also be tested in order to compare the results obtained in these tests to establish the best product for our needs.

Note: It would be best to first measure the force needed to buckle the Ameron samples at 171ºC and them use the same force every time during the Tg experimental measurements.