Engineering Notes

Processing Mechanical Test Specimens of Charred Solid Rocket Motor Insulation Materials

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DOI: 10.2514/1.39269

I. Introduction

THROUGHOUT the history of rocketry, the performance of rocket-powered devices has depended upon their ability to withstand the high temperatures and loads associated with their use. As the use of rockets has developed to require longer flights, the focus of research has shifted from developing the most efficient pressure vessel to improving the nozzle, insulation, and other time-sensitive materials [1]. Observed temperatures in modern solid rocket motors can reach as high as 4000°C, putting most structural materials at risk [2]. To allow the use of more standard materials for rocket casings, an effective thermal protection system is important. Typically, several layers of insulation are added between the solid fuel and casing, shielding the casing from the heat produced by the combusting fuel. Figure 1 shows a simple design for a cylindrical casing for a solid rocket motor.

Elastomeric compounds have been researched since the mid-tolate 1960s for applications as ablative insulation for solid rocket motors [3]. Ethylene propylene diene monomer (EPDM) rubber, found in automotive weatherstripping and some tennis shoes, is currently the standard choice for the matrix material, though new types of fillers are constantly being researched [2,4–7]. The strength of the insulation throughout the charring process is crucial to effectively designing solid rocket motors, as it impacts the amount and placement of insulation within the casing [8]. Effective modeling of these motor designs depends upon the accuracy of the material properties used in those models.

Typical methods for producing charred specimens of these insulation materials involve plasma torches, combustors, screening motors, and other complex methods [2–4,8–12]. In addition to being labor- and cost-intensive, these methods are sometimes ablative, which can make controlled-geometry specimens difficult to produce. The charred material is so fragile, it is nearly impossible to cut or machine test coupons from postchar insulation. The method described herein can be used to produce charred test specimens that are effectively identical to material taken from a screening motor and can be used on specimens machined from virgin insulation without significantly altering specimen geometries.

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II. Experimental

To properly explain the development of the char process details, it is easiest to follow its application for a particular project. Using a modified version of the ASTM-D3846-02 test standard (in which specimens are shorter and thicker than described in the test standard), specimens of several Aerojet insulation materials were tested to measure their shear strengths after charring [13]. In this method, two offset notches are cut into a specimen. A transverse force applied transverse to the notches creates a shear force (and hence shear stress) between the notch tips. Figure 2 shows the geometry and nominal dimensions for the specimens used for shear strength testing described next.

Because of the brittle nature of these materials after charring, specimens were machined from plates of virgin material. This was not simple either, because EPDM is very pliable and changes its shape if not properly restrained. The most effective way to do this was to use aluminum plates to clamp the panels in place, thus preventing the material from deforming during cutting. Notches were cut with a 0.635-mm-thick (0.025-in.) carbide blade in each side of the panel, after which a strip of notched material was trimmed off. Test specimens were cut from these strips using a box cutter. The basic process for machining these specimens is illustrated in Fig. 3.

Although this process was effective at producing specimens for this investigation, it was developed based on the cutting resources of a specific laboratory. Any procedure that can produce specimens with the proper notches and flat edges should be equally effective.

To prevent the material from oxidizing during the char, all specimens were to be charred under a predominately inert atmosphere of ultra-high-purity nitrogen gas. To create this atmosphere, specimens were loaded into a long alumina tube inserted into a Lindberg tube furnace. The ends were plugged with aluminum caps with holes drilled to allow nitrogen to be pumped in one side of the tube and carried out into a bubbler to verify positive pressure inside the tube. A quick check on the temperature of the end caps during furnace use showed that the aluminum was not seeing excessive temperatures. The caps were sealed into the tube using vacuum tape. To ensure good vacuum tape adhesion, it is recommended that the alumina tube extend at least 6 in. from either end of the tube furnace. Specimens were placed onto an alumina panel and left freestanding in the furnace during char.

The temperature used for the charring process was 1100° C, as determined based on consultations with Aerojet, with a 20-min hold time. From these discussions, it was believed that the organic content of the EPDM should be fully reacted at this temperature. Initial charring attempts used heating rates on the order of 20° C/min, which was the upper limit of the furnace capability. This procedure produced specimens that were unfit for testing due to extreme swelling and crumbling. Analysis of the furnace procedure revealed that the temperature ramp needed to be controlled at a relatively slow rate to allow volatile compounds enough time to diffuse from the material without cracking or rupturing the specimens.

The final furnace procedure was set to a ramp rate of 1°C/min until reaching 1100°C, at which point specimens were held for 20 min before the furnace was shut off and the specimens were allowed to cool. The cooling rate was not controlled, but the nitrogen atmosphere was maintained until the furnace reached 300°C to insure no oxidation could occur. Analysis of the charred specimens showed that the geometry was effectively preserved, though there was some volumetric shrinkage and, in some specimens, small amounts of dimensional warping observed.

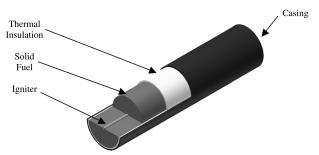


Fig. 1 Simple cylindrical solid rocket motor casing.

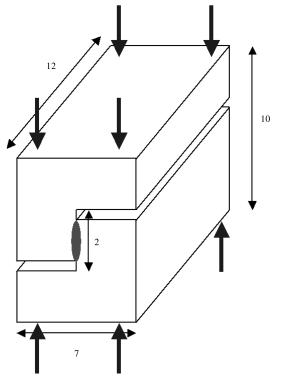


Fig. 2 Shear strength test specimen geometry (all dimensions in millimeters).

III. Example Results

As mentioned earlier, this furnace method was developed to measure the shear strengths of three charred Aerojet insulation materials. Notched specimens, as shown in Fig. 2, were tested in compression on an Instron 5867 frame with a crosshead speed of 0.025 mm/ min. The results of these tests are reprinted in Fig. 4, with the error bars representing 1 standard deviation, and in Table 1 [14].

In these data, the loading direction is in relation to the processing direction of the material, which can cause some orientation of the constituents and thus affect the insulation's mechanical properties. The number in italics and parentheses denotes the number of specimens tested for each orientation.

Despite the scatter common to brittle material failure, the results show that each material has its own shear strength. The furnace procedure therefore produced specimens that preserved the individuality of each type of insulation and even showed the sensitivity to loading direction in the highly aligned ARI-2750. Indeed, microstructural analysis through a scanning electron microscope showed that each material was morphologically distinct after charring [14].

To ensure that the material produced by this char method was representative of what occurred in the field, the microstructure and elemental composition of the ARI-2750 charred in this procedure was compared with those of the same material charred in one of

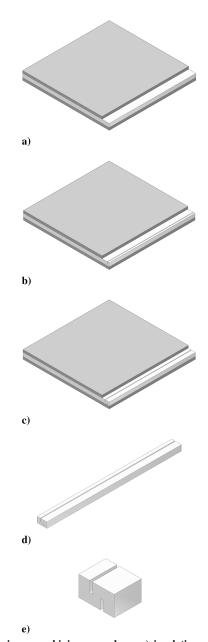


Fig. 3 Specimen machining procedure: a) insulation panel between aluminum plates; b) first notch machined into insulation; c) insulation inverted, second notch machined; d) strip machined from panel; e) specimens cut to length from strip.

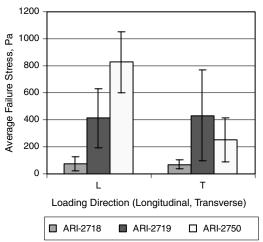


Fig. 4 Average shear strength measurements.

Table 1 Shear strength data

Material	Average shear strength	
	Longitudinal	Transverse
ARI-2718	$75.8 \pm 51 (13)$	69.5 ± 31 (6)
ARI-2719	$412 \pm 220 (11)$	$432 \pm 340 (15)$
ARI-2750	$826 \pm 230 (11)$	$237 \pm 160 (14)$

Aerojet's insulation screening motors. No significant differences, microstructural or compositional, were observed, verifying the furnace-charred material's use as a substitute method for producing charred insulation.

IV. Conclusions

The method described herein for producing test specimens of charred insulation material addresses many of the problems associated with traditional methods. Using a nitrogen-flooded tube furnace with a slow heating rate, premachined pieces of reinforced EPDM insulation were charred and successfully tested to measure the shear strengths of the charred materials. Charred materials from this furnace were virtually identical to material recovered from a fired test motor and tested to prove that each material charred would demonstrate unique properties and morphology.

The results from this work show that this furnace procedure can be used to produce test specimens for a variety of mechanical tests, allowing a large number of specimens to be produced with minimal effort and minimal effect on specimen geometry. It is hoped that future application of this method will improve the general understanding of the charring process in these materials as well as the ability to successfully model that process and its effect on material properties.

Acknowledgments

The authors would like to thank Fred Davidson, John Sparks, Doug McPherson, and Kent Hennessey from Aerojet for their support and advice throughout this project and for donating the materials used to validate this procedure. They also appreciate the assistance of David Berry of the Virginia Polytechnic Institute and State University Materials Science and Engineering Department for his help in developing the actual furnace setup.

References

- Umholtz, P. D., "The History of Solid Rocket Propulsion and Aerojet," AIAA TP 99-2927, 1999.
- [2] Koo, J. H., Stretz, H., Bray, A., Weispfenning, J., Luo, Z. P., Blanski, R., and Ruth, P., "Nanostructured Materials for Rocket Propulsion System: Recent Progress," AIAA TP 2003-1769, 2003.
- [3] Bradley, W., "Internal Insulation for Multiple Restart Motors," AIAA TP 68-500, 1968.
- [4] Crump, J. K., and Amy, A. T., "Flight Amplified Erosion of Head End Internal Insulation," AIAA TP 91-1851, 1991.
- [5] Annicelli, R. A., "Next Generation EPDMs for Auto, Wire and Cable," *Rubber World*, Feb. 2004, pp. 15–17.
- [6] Caveny, L. H., Geisler, R. L., Ellis, R. A., and Moore, T. L., "Solid Rocket Enabling Technologies and Milestones in the United States," *Journal of Propulsion and Power*, Vol. 19, No. 6, 2003, pp. 1038–1066. doi:10.2514/2.6944
- [7] Canaud, C., Visconte, L. L. Y., Sens, M. A., and Nunes, R. C. R., "Dielectric Properties of Flame Resistant EPDM Composites," *Polymer Degradation and Stability*, Vol. 70, No. 2, 2000, pp. 259–262. doi:10.1016/S0141-3910(00)00124-5
- [8] Schneider, P. J., Dolton, T. A., and Reed, G. W., "Mechanical Erosion of Charring Ablators in Ground-Test and Re-Entry Environments," AIAA TP 66-424, 1966.
- [9] Hender, D. R., Roots, A. L., and Litzinger, G. E., "Thermal Response Characterization of a Microballoon Loaded Silicone Rubber," AIAA TP 83-1472, 1983.
- [10] Spear, G. B., "The Role of Chemical Reactions and Mechanical Erosion in Aerothermal Modeling of External Insulations," AIAA TP 90-1868, 1000
- [11] Nelson, D. S., and Prince, A. S., "An Experimental Method for Evaluating the Char Tenacity of Fiber-Reinforced Insulation Materials," AIAA TP 94-3184, 1994.
- [12] Smith, S. L., Spear, G. B., and Cornwell, M. D., "Statistical Monte Carlo Prediction for Thermal Reliability," AIAA TP 94-3183, 1994
- [13] "Standard Test Method for In-Plane Shear Strength of Reinforced Plastics," ASTM Standard D 3846, American Society for Testing and Materials International, West Conshohocken, PA, 2002, www.astm. org.
- [14] Kyriakides, S. A., "Characterization of Shear Strengths and Microstructures for Charred Solid Rocket Motor Insulation Materials," M.S. Thesis, Materials Science and Engineering Dept., Virginia Polytechnic Inst. and State Univ., Blacksburg, VA, 2007.

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