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Standard Test Method for Obtaining Char Density Profile of Ablative Materials by Machining and Weighing[1]

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INTRODUCTION

The ablation characteristics of charring materials must be well known in order to design the material for a specific set of environmental conditions. The char density profile and the environmental conditions under which it was formed can provide useful information about the ablation performance.

A method of obtaining the char density profile from a charred sample of material is described in the following sections. Some chars are very friable and are easily broken before they can be measured. Other chars are relatively strong and can be handled with ease. The type of char density profile measurement described in this method is applicable if the char is strong enough to be machined without breaking.

1. Scope

1.1 This test method covers the determination of the char density profile of a charred ablator that can be used with the following limitations:

1.1.1 The local surface imperfections must be removed, and the char must be able to be machined off in a plane parallel to the char-virgin material interface before the density profiles can be determined.

1.1.2 The char must be strong enough to withstand the machining and handling techniques employed.

1.1.3 The material should have orderly density variations. The total thickness of the char and degradation zone must be larger than the machining thicknesses required.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Summary of Test Method

2.1 Density variation throughout a charred ablator material is determined by successively measuring, machining, and weighing a sample of known size to obtain the density of the material removed by machining.

3. Apparatus

3.1 The apparatus required for this method includes a laboratory balance capable of measuring to the nearest 0.0001 g, and a machining technique capable of removing material in increments as small as 0.025 mm (0.001 in.). For example, flat specimens can be machined with a surface grinder using a medium fine grit ceramic grinding wheel of a soft grade dressed to the proper contour. Cylindrical specimens can be mounted in a lathe and the char can be removed with a sharp carbide or diamond tip tool.

4. Sampling

4.1 The charred sample selected for machining and weighing should be taken from a representative section of the ablated specimen where the environmental conditions are well known, and where the surface is parallel to the char-virgin material interface. Where large sections are available, this condition is usually met. For small samples which have been exposed to varying environmental conditions along the length of the sample, the sample size will be smaller.

5. Test Specimens

5.1 A typical specimen size obtained from a channel, pipe flow, or rocket motor section may be 12.7 by 12.7 mm (0.5 by 0.5 in.), or 12.7 mm (0.5 in.) in diameter. The sample thickness is determined by the available thickness of material. Smaller or larger samples can be used depending upon the accuracy, weighing apparatus, and specimen size. Larger samples reduce edge effects. Excess virgin material should be eliminated, if possible.

5.2 The specimen is rough-cut out of the ablated section, and then machined so as to make the sides perpendicular and the front surface parallel to the char-virgin interface as shown in Fig. 1.

[1] This test method is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.08 on Thermal Protection.

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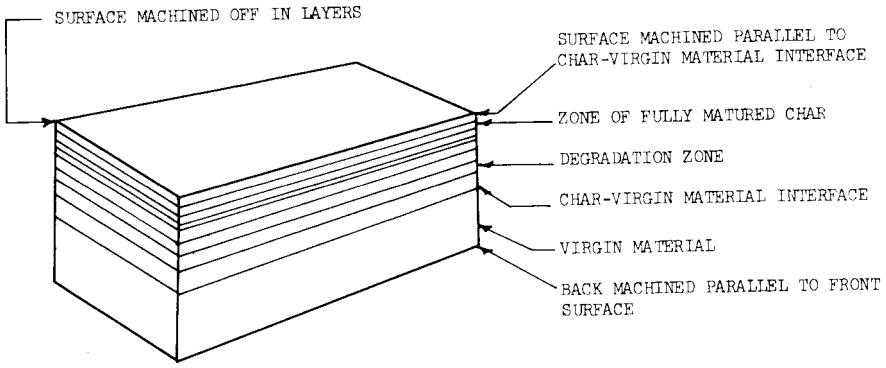

FIG. 1 Typical Specimen for Measuring Char Density Profile

5.3 For simplicity and ease of handling, the base of the sample (the backface or side opposite the charred surface) may be cemented to a thin piece of steel. This permits the sample to be handled with tweezers and facilitates the mounting of the sample on a magnetic chuck or other mounting jig for machining the surface from a fixed reference plane.

6. Procedure

6.1 *Sample Preparation*:

6.1.1 Take the dimensions of the specimen with micrometers or other suitable measuring devices to the nearest 0.025 mm (0.001 in.), and record. Weigh the specimen on a balance to the nearest 0.0001 g and record the weight. If the sample has not been prepared with parallel sides, take additional measurements to determine the surface area being removed. Section 6.4 has additional comments on moisture content and chemical reactions of some chars with atmospheric constituents.

6.1.2 Take care to prevent breaking off any of the char during the measuring or weighing operations.

6.2 *Machining*:

6.2.1 If the char-virgin material interface is not parallel to the front surface, then machine the latter parallel to the interface.

6.2.2 It is preferable to have a grinding machine, or lathe set up specifically for this machining operation so as to leave the settings untouched between weighings. For a surface grinding machine, advance the grinding wheel dressed flat to ±0.0127 mm (0.0005 in.) in 0.025-mm (0.001-in.) increments until a suitable amount has been removed. This is usually about 0.254 mm (0.010 in.). After the surface has been cleaned up (when the machining process has resulted in a continuous surface all over the specimen) allow the specimen to stabilize at the selected relative humidity and temperature conditions before measuring and weighing. For consistency, remove the maching dust after each machining operation with about 34 kPa (5 psi) air through a nozzle held about 25 mm (1 in.) from the machined surface.

6.3 *Dimensional Measurements*:

6.3.1 The increment of material removed between weighings is usually 0.254 mm (0.010 in.) depending upon the total char thickness and the uniformity of the charred specimen. If the char is relatively uniform in density down to the char-virgin material interface, increase the machining increments. If the char-virgin material interface is sharply defined, take smaller machining increments. Exercise extreme care when making the dimensional measurements with a micrometer to avoid crushing the char.

6.4 *Weight Measurements*:

6.4.1 Maintain the specimen at constant humidity conditions (ideally dry) during the machining, measuring, and weighing operation. A freshly charred ablator has a chemically active surface like that of activated charcoal. Previous experience indicates equilibrating the specimen to 50 % relative humidity and 20°C (70°F) dry should yield good results.[2] Make a separate measurement on the char to determine how much moisture is contained at this relative humidity.

6.4.2 Some chars have chemically active species which are unstable in atmospheric conditions. Special handling techniques would be required for these chars and this is beyond the scope of this test method.

6.4.3 Repeatedly machine, measure, and weigh the specimen until the char and the char-virgin material interface has been removed, and the density approaches the virgin material density.

7. Calculation

7.1 Determine the average density of the material removed using the weight and dimensional changes as follows:

$$\rho = (W_1 - W_2)/A(H_1 - H_2) \quad (1)$$

where:

ρ = average density, kg/m^3,
W_1 = weight before machining, kg,
W_2 = weight after machining, kg,
A = surface area of specimen face, m^2,
H_1 = height of specimen before machining, m, and
H_2 = height of specimen after machining, m.

8. Report

8.1 Report the density calculated for each machining step at the point mid-way between the height measurement for each increment removed. Report the density on a moisture-free basis using the measured relative humidity and temperature conditions taken when weighing the specimen. Report the average relative humidity and temperature conditions. If the moisture content of the char has not been determined as a function of

[2] Liston, E. M., "Experimental and Analytical Studies of Radiation-Only Pyrolysis of Model Char-Forming Polymers", Contract NAS-7-341, SRI Report No. 19, Final Report 1968.

relative humidity and temperature then report these conditions and the time of equilibration at these conditions before weighing.

9. Precision and Bias

9.1 The precision resulting from the machining and weighing technique described here is inversely proportional to the height removed between weighings and the accuracy of the measuring device used to determine the dimensions between weighings. An error of less than 3 % can be achieved where the height removed is on the order of 0.254 mm (0.010 in.) and the sample is not smaller than 12.7 mm (0.5 in.) in length and width, or diameter. The precision is also dependent upon the moisture content of the char and the variation in relative humidity and temperature during machining and weighing.[3]

9.2 The bias of this method is dependent upon the friability of the sample, the density gradient, the size of the specimen, the uniformity of the char, the moisture content of the char, and the variation in relative humidity.

10. Keywords

10.1 ablation; char density; density profile

[3] Hiester, N. K., and Clark, C. F., "Comparative Evaluation of Ablating Materials in Arc Plasma Jets," NASA CR-1207, 1968.