

FIBER DENSITY ANALYSIS BY DENSITY GRADIENT TECHNIQUE

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1. Introduction

Currently many diverse fluid displacement procedures are employed to measure carbon/graphite fiber density. Cross referencing of available fiber density data is difficult due to the use of different test fluids and the low precision associated with some techniques. Precise fiber density values are required for aerospace applications to insure final part density uniformity and to determine composite void fractions. The density gradient procedure described in this paper was developed at the Lockheed Palo Alto Research Laboratory (LPARL) in 1975 and subsequently transferred to the Hitco Corporation in Gardena, Calif. as a standard test procedure for carbon fabric CCA3-1641B. The procedure follows the general outline of ASTM-1505 with specific techniques employed to make it directly amenable to carbon/graphite fiber analysis. All of the precision and accuracy data presented was generated on experimental carbon fabric. The commercial fabric is used extensively throughout the aerospace industry with phenolic resin to produce ablative composite hardware.

2. Experimental

Specific equipment and procedures not specified in the ASTM-1505 procedure are presented.

Column Preparation, Ref. Figure 1

Benzene, $\rho = 0.8794$ gms/cc and 1,1,2,2, Tetrabromoethane (TBE), $\rho = 2.9638$ gms/cc are the fluids used to produce a gradient column with the appropriate density range. Specific materials employed are as follows:

- o Pyrex tube, heavy wall with closed end, 160 cm high x 2.54 cm I.D.
- o Basket, cylindrical shape 2 cm high, fabricated from ≈ 40 mesh copper screen, soft soldered.
- o Chain, 1/8" round bead stainless steel, ≈ 7 cm diameter x 15 cm long drum attached.
- o Motor speed control, Minarik model SL-14.
- o Glass density floats, SGA D-1190 from SGA Scientific Inc., 737 Broad St., Bloomfield, N. J.
- o Meter tape, 150 cm long purchased from any yardage store, double back taped to column exterior.

Aluminum components must be avoided due to chemical attack by TBE. A cork is used to seal off the column when not in use and a slotted cork used during test operations to reduce fluid evaporation. The continuous siphon procedure, per ASTM Method B, is used to "build the column". Liquid flowing into the column is directed to flow down the bead chain at a rate of 1 cm column rise

per minute. A disc of #1 filter paper is placed in the basket to reduce splashing. The basket is continuously raised to maintain its level consistent with the rising fluid height. The column is filled to within 5 cm of the top and 2 cm of pure benzene is introduced to "cap off" the column. The column is mounted in a fume hood to prevent excessive exposure to the benzene vapor. No temperature control is provided; as the laboratory temperature changes the calibration float beads adjust accordingly.

Specimen Preparation

Care is exercised at all times to insure against fiber bundle break up. Carbon fabric is sampled at specific locations with a 1/2" or 5/8" cork borer tool. Location identity is maintained by removing cord segments from the disc side or sides. All cord cuts follow the warp or fill direction to prevent frayed edges. Yarn samples are about 10 cm long. Sizing is removed from the specimens by exposure to hot MEK for epoxy compatible sizing and water for PVA. Residual moisture and solvent is removed by oven drying for a minimum of 2 hours at 105°C. All specimens must be dried prior to testing. Some carbon fibers with type 1 isotherm pores may require a longer drying time at temperature and under vacuum to remove all of the residual moisture. After drying, specimens are introduced directly into a beaker with pure benzene and subjected to a 5 minute ultrasonic agitation. The conditioned specimens are introduced directly into the column without allowing the benzene to dry out. Fabric discs are simply dropped in and for yarn samples 3-2 cm lengths of sample yarn are cut above the column and allowed to drop into the column. A timer is initiated at introduction and 5 minute interval density values are measured until equilibrium is approached.

3. Discussion

The development of fast and precise data by this technique requires insight into 3 factors which greatly influence fiber response. These factors are surface debris, residual moisture and the internal pore fill rate. Surface debris is occasionally present on low fired carbon fiber and is primarily residual inorganic compounds driven out of the fiber during pyrolysis. This material is removed by the 5 minute benzene ultrasonic operation. Residual moisture is always present in carbon or graphite fibers and values, up to 4% by weight, have been measured on as received carbon fabric. The water molecule is very small approx. 3.5Å diameter, and has a propensity for penetrating deep into the pore

fiber structure. If the moisture is not removed prior to testing the water molecules will supplant normal air void areas which cannot be penetrated by the benzene molecule, which is about 5.9Å in diameter. Since water is insoluble in benzene it is also possible that residual water molecules will be pushed into smaller void areas by the penetrating benzene. Residual fiber moisture will induce higher fiber density values. Using the experimental procedure outlined and a 30 minute test time, 16 specimens of varied experimental carbon fabric were tested at LPARL and the Hitco Corporation to determine the test procedure precision. The standard deviation of difference between laboratories was 0.00208 gms/cc with the observation error standard deviation of 0.00147 gms/cc. This relates to a 68% confidence level to ± 0.00147 gms/cc and essentially 100% confidence at $\pm .00441$ gms/cc.

The measured fiber density accuracy was determined by fabricating phenolic matrix composite hardware from the same experimental fabric. Figure 2 presents the measured fiber density versus final composite density with a theoretical line based on a 37½ weight % matrix. The theoretical composite densities were calculated from the following volume balance equation:

$$g_{c\rho c}^{-1} = g_{r\rho r}^{-1} + g_{f\rho f}^{-1}$$

where g = weight % of c-composite,
 r -resin and f -fiber
 ρ = density gms/cc respectively.

Some scatter of fabricated composite density data can be expected due to inprocess variables which will alter the matrix volume fraction. Phenolic resin staging prior to cure, degree of debulking, bleeder cloth application and other small variables can induce slight changes in the final part matrix volume fraction. The theoretical calculations however assume a constant matrix/fiber fraction with no void content. The relationship demonstrated in Figure 2 appears to indicate two significant results; the measured fiber density data is accurate and the inprocess variables which can alter the matrix volume fraction are minimal.

4. Conclusions

The density gradient technique has demonstrated excellent precision and accuracy for measuring carbon fabric density. The test appears amenable to the entire range of carbon/graphite fabric and yarns which are currently being produced. The simplicity and precision demonstrated by this technique indicate it should be considered as the bases for an industry wide test procedure.

5. Acknowledgements

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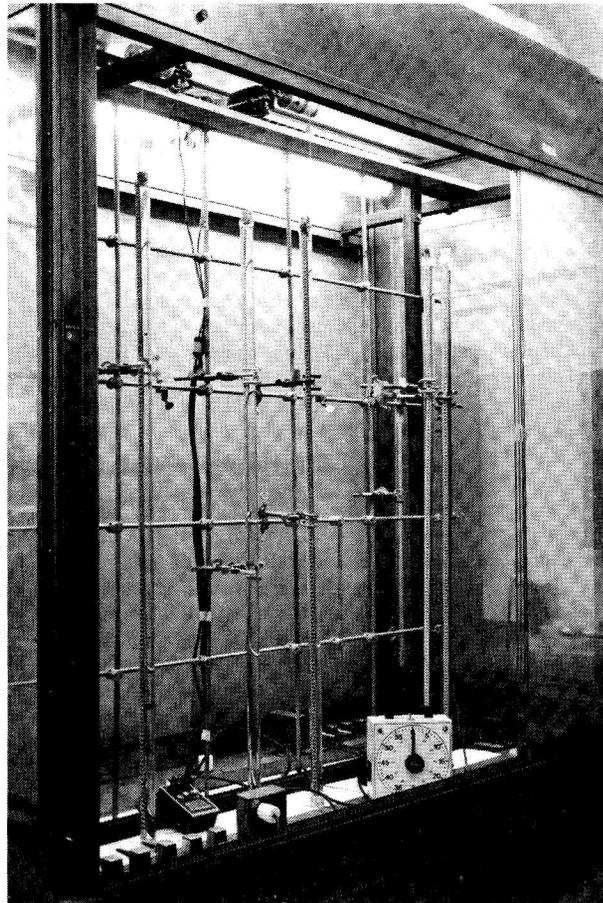


Figure 1

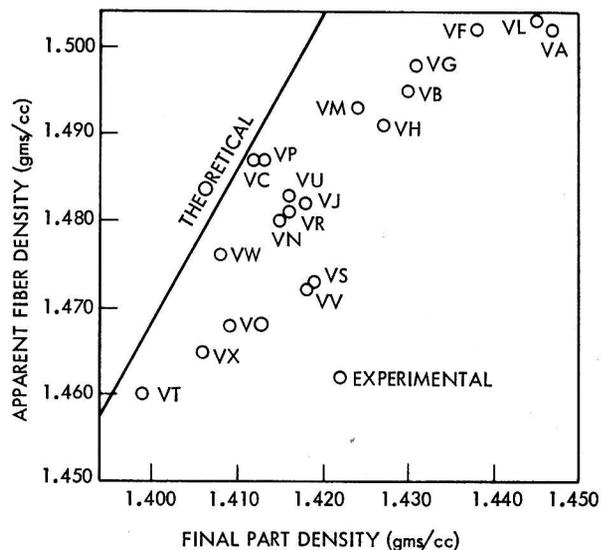


Figure 2