

**NBSIR 87-3681**

# **Application of Optical Image Analysis to Quantitative Microstructure Characterization of Composite Materials**

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December 1987

Sponsored by:

**U.S. Army Materials Technology Laboratory  
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**U.S. DEPARTMENT OF COMMERCE, C. William Verity, *Secretary***  
**NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director***



October 1, 1987

CONTRACT REPORT TO: Dr. James McCauley  
Army Materials Technology Laboratory

FROM: Dr. James F. Kelly  
National Bureau of Standards

TIME PERIOD: This report covers work done under contract with the Army  
Materials Technology Laboratory during FY1987.

Classification Code: 21702020 64-8929 P728102.13 2588 S19129  
Cost Code: 7 TY00K5X475XD

PROJECT TITLE: Standards for Quantitative Microstructural Characterization  
of Composites and Advanced Structural Ceramic Materials

PROJECT SCOPE: The ultimate objective of this project is to define standard procedures for numeric description, by image analysis (optical and SEM), of the microstructural features relevant to mechanical properties of organic matrix composites and advanced structural ceramic materials. The effort in this initial year of the project at NBS concentrated on two aspects of image analysis characterization: the system calibration procedure to satisfy accuracy and precision requirements, and the specific details of a microstructural analysis applied to a single composite specimen. For this work, AMTL provided a calibration test slide containing a number of geometric figures of specified dimensions and a prepared composite specimen of unidirectional graphite fiber reinforced polymer, GFRP; Sample I.D. No. 4-7 AS4-4 10A.



Report Title: Application of Optical Image Analysis To Quantitative  
Microstructure Characterization of Composite Materials

Abstract

Proper specification of the microstructure of organic matrix composites is necessary for control of their mechanical properties. A study of the requirements for quantitative microstructure characterization using an image analysis(IA) system is reported. System calibration procedures are described with a comparison between two commercial IA systems. The requirements for a standard reference material for second phase area fraction are presented and a numerical procedure is developed to describe the inhomogeneity in second phase dispersion throughout the matrix.

IMAGE ANALYSIS SYSTEM CALIBRATION

There are two aspects of measurement accuracy using an optical microscope or SEM based image analyzer.

1. Calibration of the pixel size. This can be done with either a length or an area standard. This calibration standard must cover a sufficient length(area) such that the pixel resolution is insignificant. This limitation should be explicitly defined in any proposed handbook of recommended procedures. e.g. A standard length extending at least 100 pixels would be required for 1% precision.





2. Threshold setting. Each pixel in the image area is interpreted by the computer as either off or on, based on whether the measured intensity level at the pixel location exceeds the threshold intensity level. Materials, such as the GFRP samples, exhibiting good optical contrast between the separate phases, can be clearly separated between the bulk matrix and reinforcing phase. The difficulty in setting the correct threshold level arises from an uncertainty in precisely defining the boundary regions or fiber perimeters. For large features, such as the calibration disk, this isn't a problem since the number of perimeter pixels are few compared to the total intersected in making either a diameter or area measurement. However, for features the size of the graphite fibers (approx. 7 $\mu$ m), the perimeter pixels may represent 20 to 25 per cent of the fiber area. This large uncertainty means that the proper calibration of pixel size only guarantees an accurate measure of large features.

These observations point out the need for an area fraction standard reference material which is similar to the test specimen in the following characteristics:

1. Total second phase area fraction
2. Size distribution of second phase features
3. Contrast between phases

#### TEST SLIDE CALIBRATION

A comparison of the calibration between the NBS Omnicon system and the AMTL



Quantimet system was obtained by analyzing the AMTL test slide after calibrating with the Omnicon test slide.

The image analysis system utilized in these analyses was an Omnicon\* 3500, manufactured by Bausch and Lomb\*. A calibration slide provided with this unit was used to define the pixel dimensions. The procedure was to select for imaging a circular disk of known diameter which covered a sufficient number of pixels to achieve good precision in the calibration. At the maximum system magnification of approximately 770X, and a screen size of 22cm by 16.5cm, a 100um diameter disk extends over more than 300 pixels. This calibration is checked each time the system is initialized. Typical variation in calibration of about 1 % was observed on a day to day basis.

The area of each of the twelve circular discs on the AMTL test slide were measured using the highest microscope magnification (40X objective lens). It required six fields to cover the twelve disks. The analysis was repeated twice at each of three threshold settings designated low, medium, and high. The results of these analyses are given in Table I. The Feret diameters of the 12 disks were measured in two perpendicular directions. The 40X lens and a medium threshold setting were used for these measurements.

For comparison the results of AMTL measurements provided by John Ricca and Rebecca Jurta are included as Table III. As can be seen in these tables, the mean values of the NBS measurements differ by less than 1% from those made by AMTL for both area and diameter. This should give us confidence

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\* OMNICON is a registered trademark of Artek Systems Corp.



that an excellent calibration of the pixel size can be made using a similar test slide specimen or a standard stage micrometer often provided with the optical microscope.

#### MICROSTRUCTURE CHARACTERIZATION OF GFRP SAMPLE 4-7 AS4-4 10A

The unidirectional graphite fiber reinforced polymer composite specimen is a rectangular section cut and polished by AMTL from a tensile specimen with the graphite fiber axes perpendicular to the cutting plane. The composite is approximately 25mm by 1.4mm. Figure 1 shows photomicrographs of the sample which demonstrate two important features of the microstructure: 1. There is little variation in the diameters of the graphite fibers and 2. The area fraction or concentration of fibers is not homogeneous.

It seems unlikely that small variations in the fiber diameter distribution would have any significant affect on mechanical properties, thus it should be sufficient to measure a few fiber diameters as verification that the mean diameter is within an acceptable range. The more critical issue is how to accurately measure the mean fiber area fraction and obtain a meaningful measure of the local area fraction distribution.

The mean fiber area fraction was measured by manually setting the threshold for fiber detection to a level which gave a satisfactory visual display on the Omnicon monitor. Namely, a setting such that the fiber image areas appear intensified, but not the matrix areas. As discussed earlier, this procedure is done without calibration since no appropriate area fraction reference standard is available. However, the threshold setting is reproducible and should yield results which differ by a constant factor



from area fraction measurements made on the same material using a different threshold.

The Omnicon 3500 was programmed to measure the fiber area fraction in the current field of view, then translate the microscope stage to an adjacent field for measurement until a matrix of 300 fields (75 X 4) were completed. Each field covers an area of 295um by 220um, thus about 55% of the sample is covered by the 300 fields. The program stores the 300 individual area fraction results and calculates the minimum, maximum, mean and variance of the data. These data are then output via the RS232 printer port to the input port of an MS-DOS PC and stored on disk. This is a more convenient format for printing, plotting, or further data analysis. The results for two replicate 300 field analyses are presented in Table IV.

The most striking feature of the microstructure, the inhomogeneity in fiber concentration, has no well defined measure. Measurement of area fraction over a large section of the sample gives us a reproducible measure of the fiber area fraction, but the variability from field to field is a function of the field size. The occurrence of areas with fiber area fractions significantly less than the mean are likely to be of critical importance in the resultant mechanical properties of the composite material. One measure, which I would propose for correlation with mechanical properties, is the variance of the fiber area fraction distribution measured at several field sizes. As the field size is decreased the increase of this variance is an indicator of inhomogeneity of fiber distribution. Ultimately, as the field decreases to encompass only a few fibers we would expect an increased variance due simply to counting statistics. However, at intermediate field





sizes which still cover a large number of fibers, the variance in fiber distribution will increase more rapidly with decreasing field area for an inhomogeneous dispersion than for a homogeneous one. Measurements covering the same region on the GFRP specimen were using three different field sizes. The results are presented in Table V.

The change in variance seen in Table V expresses the increased variability in area fraction from field to field for the reduced field areas. When the measured field size is large enough to average over many of the fiber depleted regions, the measured variance in area fraction is low. As the field area is decreased to where a single depleted region greatly reduces the area fraction, we see the significant increase in the variance of the area fraction distribution, while the mean area fraction remains essentially constant.

The distribution of fiber area fraction for measurements made with three different field sizes( full field, 1/4 field, 1/16 field) are shown in Figures 2-4. The total sample area analyzed is the same for each of the three distributions.

#### SUMMARY

Consideration of the use of automated image analysis in the microstructural characterization of uniaxial graphite fiber reinforced polymer matrix composites has led to the following observations:

1. There is a need for a standard reference material for second phase area fraction calibration of image analyzers. This reference material



should be similar to the test materials in total area fraction, individual feature area, and second phase/matrix contrast.

2. A measure is needed to quantify the inhomogeneity in fiber distribution. One possible such measure, the change in variance of the area fraction distribution with changing field size was demonstrated.

3. A coordinated program of mechanical testing and microstructural analysis is needed to establish the correlation between mechanical properties and any measure of microstructural inhomogeneity.



TABLE I

MEASURED AREAS\* OF 12 CIRCULAR DISKS ON QUANTIMET TEST SLIDE

THRESHOLD	TEST	MINIMUM	MAXIMUM	MEAN	MEDIAN	STND. DEV.
LOW	1	8447	8548	8481	8460	24.6
LOW	2	8438	8534	8474	8460	25.5
MEDIUM	1	8326	8380	8349	8350	14.5
MEDIUM	2	8290	8404	8355	8350	28.5
HIGH	1	8235	8321	8273	8267	23.6
HIGH	2	8236	8302	8262	8254	14.5

\* All areas are in  $\mu\text{m}^2$ 

TABLE II

MEASURED FERET DIAMETERS\* OF 12 DISCS ON QUANTIMET TEST SLIDE

FERET DIRECTION	MINIMUM	MAXIMUM	MEAN	MEDIAN	STND. DEV.
X AXIS	103.7	104.6	104.2	104.4	2.3
Y AXIS	103.0	104.6	103.9	104.0	2.3

\* Diameters are in  $\mu\text{m}$ .

X Axis is taken parallel to long direction of test slide.

TABLE III

AMTL MEASUREMENTS OF DIAMETERS AND AREAS OF THE 12 DISKS

PARAMETER	MINIMUM	MAXIMUM	MEAN
FERET X	102.4	104.1	103.3
FERET Y	102.4	104.7	103.2
AREA	8270	8486	8362



TABLE IV

FIBER AREA PERCENT RESULTS FOR 300 FULL FIELD ANALYSES

RUN #	MINIMUM	MAXIMUM	MEAN	STND. DEV.
1	19.5	63.1	44.2	7.0
2	21.7	61.9	44.6	7.4

TABLE V

FIBER AREA PERCENT RESULTS FOR VARYING FIELD SIZE

FIELD FRACTION*	FIELDS	MINIMUM	MAXIMUM	MEAN	VARIANCE
1 X 1	18	35.6	53.5	46.5	22.
1/2 X 1/2	72	25.6	57.0	43.6	61.
1/4 X 1/4	288	11.4	64.8	44.2	146.

\* Full Field dimensions are 295um X 220um





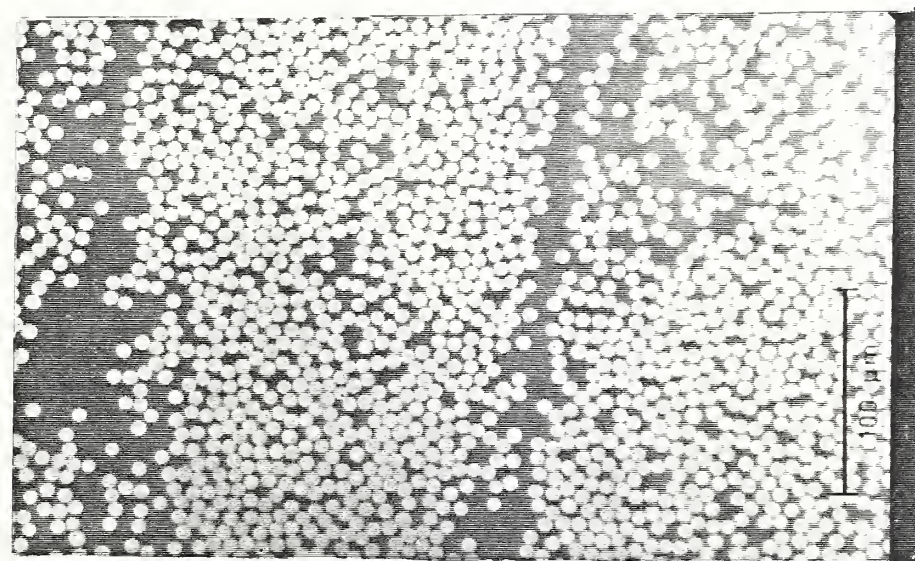
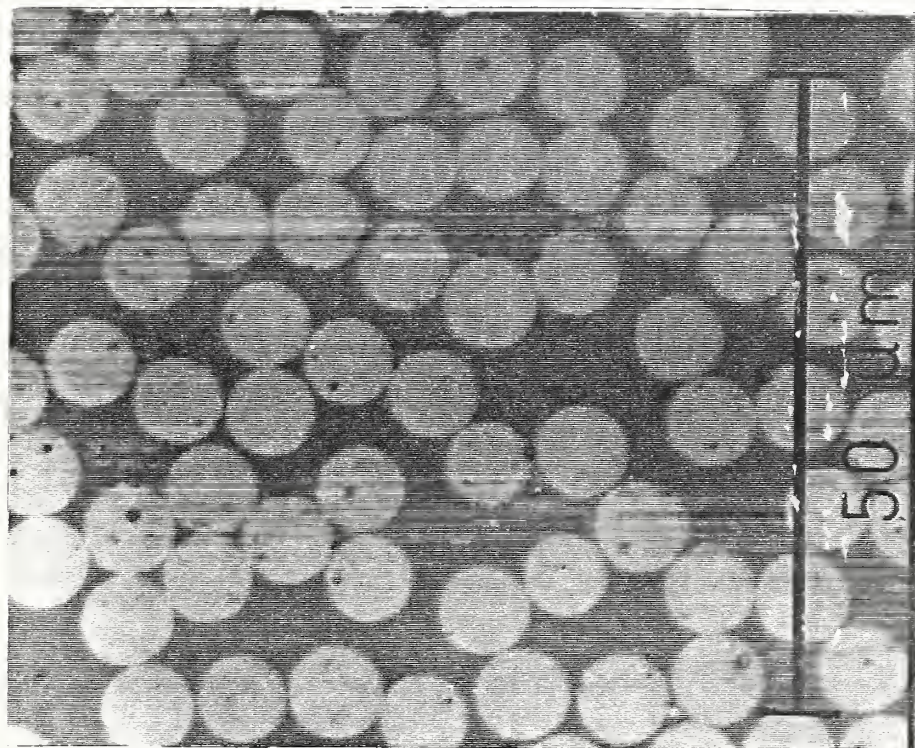


Figure 1. Optical Micrographs of GFRP Specimen



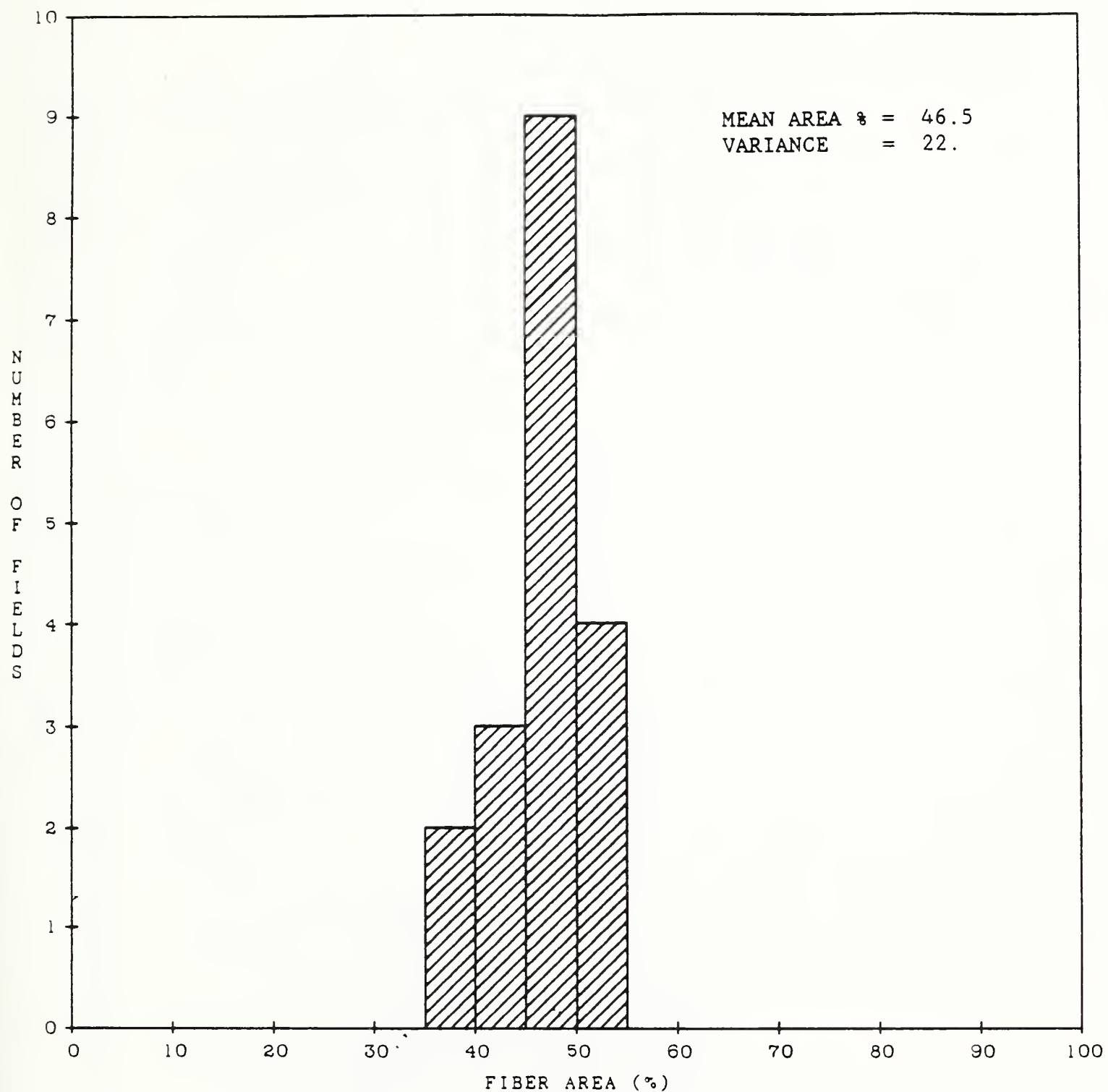


Figure 2. Area Fraction Distribution of Graphite Fibers.  
Field Area: Full  
Number of Fields: 18





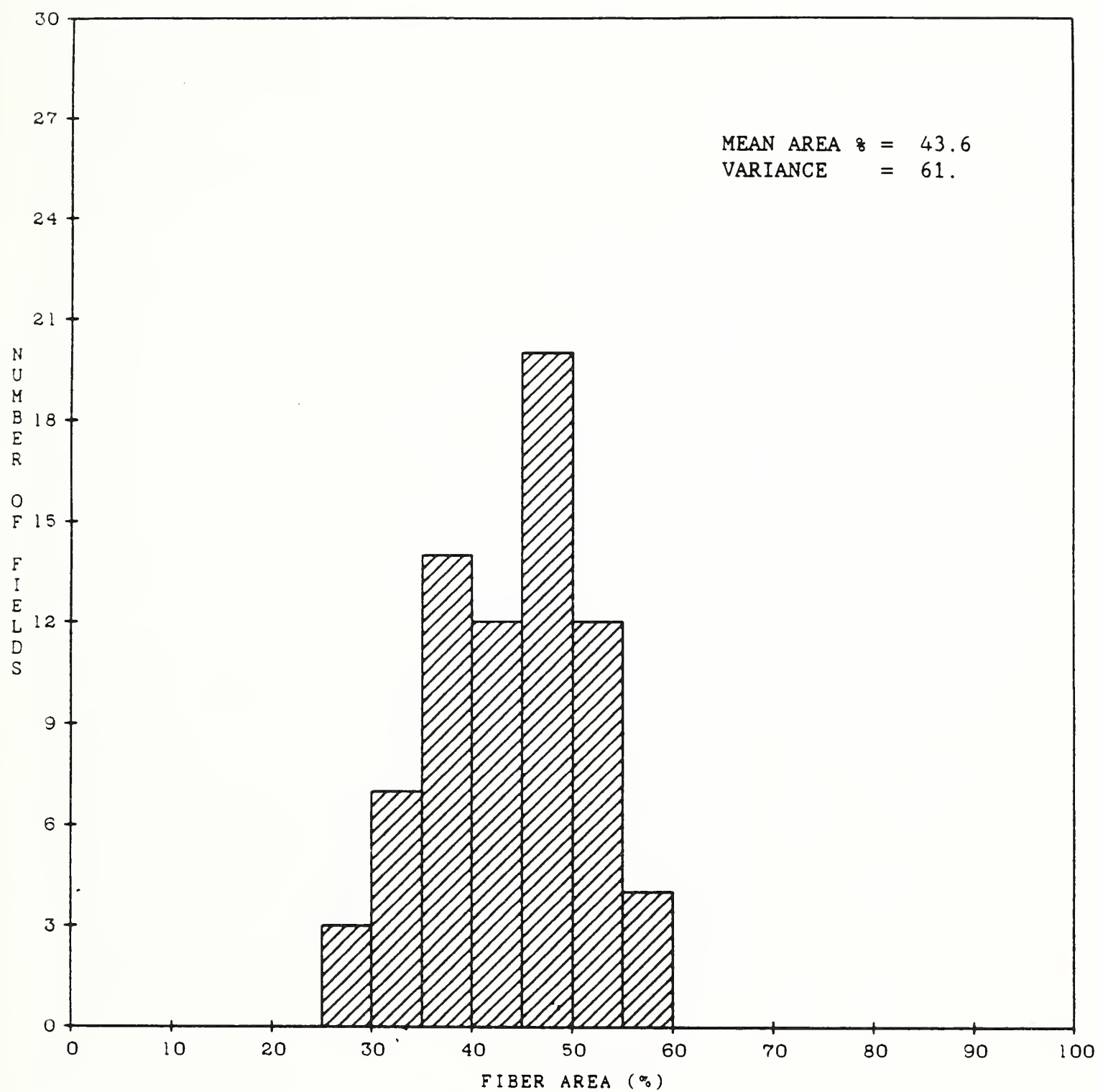


Figure 3. Area Fraction Distribution of Graphite Fibers.  
Field Area: 1/4 Full  
Number of Fields: 72



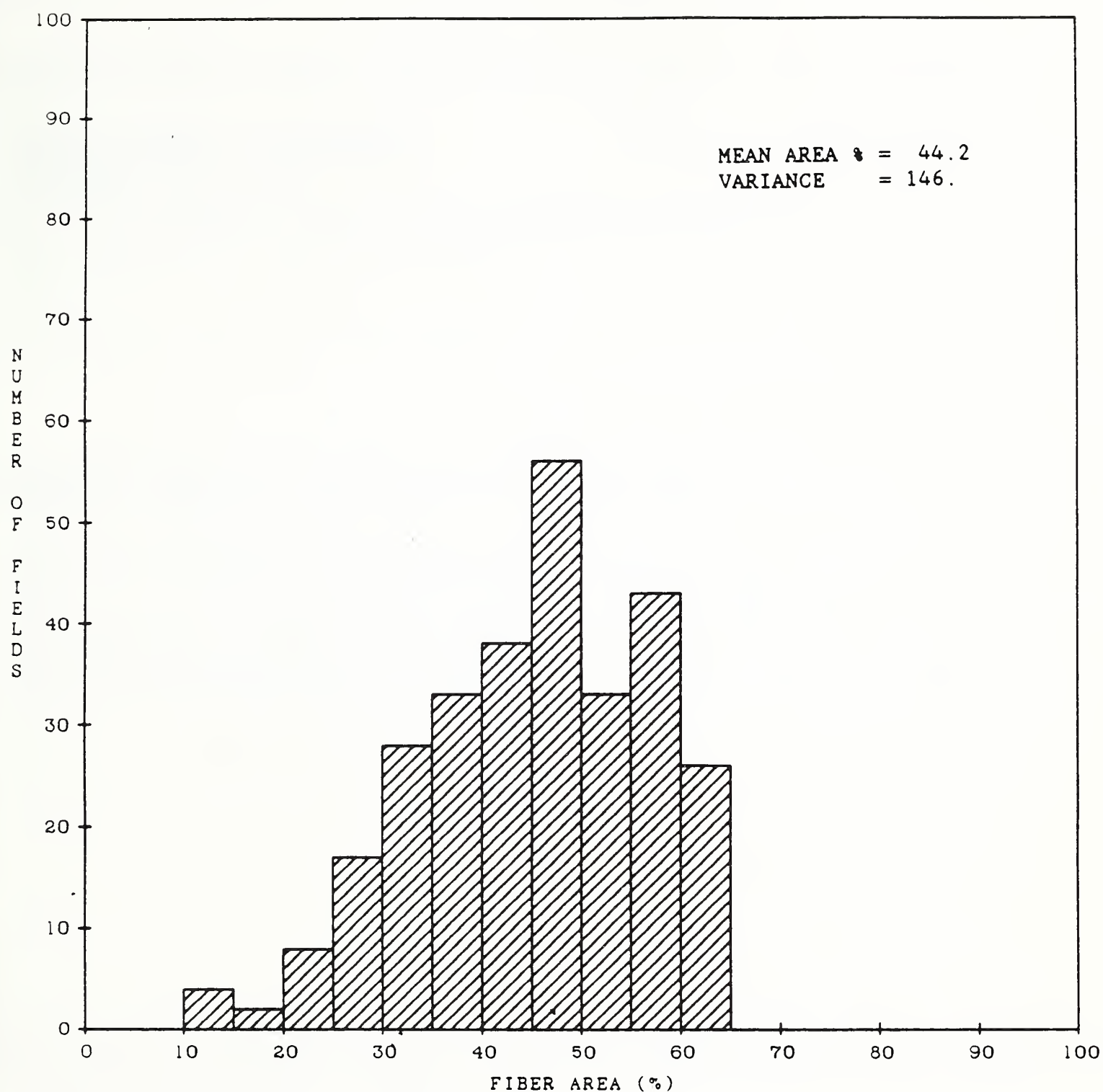


Figure 4. Area Fraction Distribution of Graphite Fibers.  
Field Area: 1/16 Full  
Number of Fields: 288





U.S. DEPT. OF COMM. <b>BIBLIOGRAPHIC DATA SHEET</b> <i>(See instructions)</i>	1. PUBLICATION OR REPORT NO. NBSIR87-3681	2. Performing Organ. Report No.	3. Publication Date December 1987
4. TITLE AND SUBTITLE Application of Optical Image Analysis to Quantitative Microstructure Characterization of Composite Materials			
5. AUTHOR(S) James F. Kelly			
6. PERFORMING ORGANIZATION <i>(If joint or other than NBS, see instructions)</i> NATIONAL BUREAU OF STANDARDS U.S. DEPARTMENT OF COMMERCE GAITHERSBURG, MD 20899		7. Contract/Grant No. 8. Type of Report & Period Covered FY87	
9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS <i>(Street, City, State, ZIP)</i> U.S. Army Materials Technology Laboratory Watertown, MA			
10. SUPPLEMENTARY NOTES  <input type="checkbox"/> Document describes a computer program; SF-185, FIPS Software Summary, is attached.			
11. ABSTRACT <i>(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)</i> Proper specification of the microstructure of organic matrix composites is necessary for control of their mechanical properties. A study of the requirements for quantitative microstructure characterization using an image analysis (IA) system is reported. System calibration procedures are described with a comparison between two commercial IA systems. The requirements for a standard reference material for second phase area fraction are presented and a numerical procedure is developed to describe the inhomogeneity in second phase dispersion throughout the matrix.			
12. KEY WORDS <i>(Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)</i> area fraction; calibration; characterization; composite; graphite fiber; image analysis; microstructure; optical			
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