209
-12/69
AEC RESEARCH AND DEVELOPMENT REPORT

805
Y-1664
Chemistry

MASTER

STRUCTURAL STUDIES OF CARBON FOAM WITH A
SCANNING ELECTRON MICROSCOPE

C. R. Schmitt
A. L. Coffey, Jr.
J. M. Schreyer

UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

operated for the ATOMIC ENERGY COMMISSION under U. S. GOVERNMENT Contract W-7405 eng 26

UNION
CARBIDE

OAK RIDGE Y-12 PLANT
P. O. Box Y
OAK RIDGE, TENNESSEE 37830

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

Printed in the United States of America. Available from Clearinghouse for Federal
Scientific and Technical Information, National Bureau of Standards,
U.S. Department of Commerce, Springfield, Virginia 22151
Price: Printed Copy \$3.00; Microfiche \$0.65

— LEGAL NOTICE —

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

- A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Date Issued: February 27, 1969

Document Y-1664

Chemistry
TID-4500

UNION CARBIDE CORPORATION
Nuclear Division

Y-12 PLANT

Contract W-7405-eng-26
With the US Atomic Energy Commission

STRUCTURAL STUDIES OF CARBON FOAM WITH A
SCANNING ELECTRON MICROSCOPE

C. R. Schmitt
A. L. Coffey, Jr
J. M. Schreyer

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, expressed or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission, or employee of such contractor, to the extent that such employee or contractor of the Commission, or employee of such contractor prepares, disseminates, or provides access to, any information pursuant to his employment or contract with the Commission, or his employment with such contractor.

Oak Ridge, Tennessee

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Document Y-1664Chemistry
TID-4500

Distribution:

Air Force Scientific Command

Meeks, S. J.

Atomic Energy Commission-ORO

Keller, C. A.

Oak Ridge Gaseous Diffusion PlantJordan, R. G.
Vanstrum, P. R.Paducah

Winkel, R. A.

Y-12 PlantBallenger, H. F. (2)
Bell, B. B.
Bernander, N. K.
Briscoe, O. W.
Burkhart, L. E.
Coffey, A. L., Jr (5)
Evans, G. W.
Hemphill, L. F.
Horde, G. W. (2)
Kahl, K. G.
Keith, Alvin (2)
McLendon, J. D.
Mitchel, G. W.
Phillips, L. R.
Schmitt, C. R. (5)
Schreyer, J. M. (5)
Wesley, R. L.
Y-12 Central Files (5)
Y-12 Central Files (Route)
Y-12 Central Files (Y-12RC)

In addition, this report is distributed in accordance with the category Chemistry, as given in the "USAEC Standard Distribution Lists for Unclassified Scientific and Technical Reports", TID-4500.

ABSTRACT

A scanning electron microscope was used for the direct examination of structural features and surface topography of low-density carbon foams and thermosetting resins from which these foams were made. Hole and indentation-type structural defects were found which were more prevalent in the weaker foams examined.

CONTENTS

SUMMARY	5
INTRODUCTION	6
STUDIES OF THE CARBON FOAM	7
Experimental Work	7
Discussion	14
ACKNOWLEDGEMENTS	23
REFERENCES	24

SUMMARY

The scanning electron microscope (SEM) was found to be a valuable tool for the direct examination of structural features and surface topography of low-density carbon foams and carbonaceous polymers from which these foams are made.

Samples of low-density carbon foam (0.05 and 0.10 gm/cc), which showed relatively low and high compressive strengths, were examined to determine any structural defects or abnormalities in the cell walls that might account for differences in their compressive strengths. Scanning electron photomicrographs taken both parallel with and perpendicular to the direction of foam rise disclosed some definite structural abnormalities in the weak foam in that the cell walls contained a significantly greater number of holes or small rounded areas of indentation or warpage in the surface, which probably act as stress risers. Scanning electron microscope studies on the low-density, polyurethane-furan resin foam from which the carbon foam was made showed that the hole and indentation-type structural defects were present in the foam while still in the uncured condition, thus indicating that process variables in the formulation, mixing, and curing operations would need better control in order to obtain a stronger foam.

INTRODUCTION

The scanning electron microscope offers a new approach to morphological and surface topography studies of low-density structural materials such as carbon foams. This technique allows the direct examination of the structural units (struts) comprising the cells of the carbon foam and has considerable advantage over optical microscopy in that it has a wider range of viewing magnifications, has a depth of focus nearly 300 times greater, and has a resolution approaching 100 Å.

Since the strut surfaces and other physical characteristics of carbon foam make it difficult to examine in replicated form by standard, transmission-type electron microscopy, the use of the scanning electron microscope seemed to be the fastest and easiest method for the direct examination of structural features and surface topography of low-density carbon foams. An investigative program was, therefore, initiated to evaluate the suitability of the scanning electron microscope for revealing any structural defects or abnormalities in the cell walls of low-density carbon foams and polymeric foam, before coking, that might be related to the compressive strength of these foams.

STUDIES OF THE CARBON FOAM

EXPERIMENTAL WORK

Samples of low-density carbon foam (0.05 gm/cc) were examined with a scanning electron microscope to discern any structural defects or abnormalities that might be present in the cell walls. The foams were examined both parallel with (top view) and perpendicular to (side view) the direction of foam rise.

Scanning electron photomicrographs showing typical top and side views of the carbon foam taken at a magnification of 100X are presented in Figure 1. Also shown in Figure 1 are electron photomicrographs of a small protuberance of a cell wall (shown by a circle at 100X) which were made at magnifications of 300, 1,000, and 3,000X, and which show the increasing resolution of structural detail that is possible with this instrument.

Figure 2 shows six scanning electron photomicrographs of some typical "hole" and "crack"-type defects which were located in various samples of the 0.05 gm/cc carbon foams.

Two samples of 0.05 gm/cc carbon foam which showed an appreciable difference in compressive strengths (ie, 25 and 70 psi, respectively) were examined in an attempt to determine any structural defects or abnormalities in the cell walls that might account for differences in compressive strength. Scanning electron micrographs of the relatively weak foam taken at magnifications of 300 to 7,000X disclosed some definite structural abnormalities in that the cell walls contained a significantly greater number of indentations or small rounded areas of warpage in the surface. These abnormalities can be seen in Figure 3. The cell walls of the relatively strong 0.05 gm/cc carbon foam (as shown in Figure 4) were considerably smoother in appearance, although some of the indentations or warpage areas were also present.

Electron microscope scans of two other samples of carbon foam having a density of 0.10 gm/cc which also showed relatively low and high compressive strengths (ie, 214 and 289 psi, respectively) showed the same general structural differences in that a greater number of surface indentations or warpage areas in the wall structure was associated with the foam having the lower average compressive strength. Electron micrographs of the relatively weak and strong 0.10 gm/cc carbon foams at 500 to 3,000X magnification are presented in Figures 5 and 6, respectively.

Top View (parallel)

100X

Side View (perpendicular)

100X

Detail Views of Area "A"

(a) 300X

(b) 1,000X

(c) 3,000X

Figure 1. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM SHOWING RESOLUTION OF THE STRUCTURAL DETAIL.

(a) 300X

Hole Defect

(b) 1,000X

(c) 3,000X

Crack Defect

(a) 100X

(b) 300X

(c) 3,000X

Figure 2. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM SHOWING SOME TYPICAL STRUCTURAL DEFECTS.

Side Views

(a) 100X

(b) 300X

(c) 1,000X

(d) 3,000X

(e) 7,000X

(f) 10,000X

Figure 3. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM HAVING A LOW COMPRESSIVE STRENGTH.

Top Views

(c) 300X

(b) 300X

(c) 300X

Side Views

(a) 300X

(b) 300X

(c) 1,000X

Figure 4. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM HAVING A HIGH COMPRESSIVE STRENGTH.

Top Views

(a) 500X

(b) 1,000X

(c) 3,000X

Side Views

(a) 500X

(b) 1,000X

(c) 3,000X

Figure 5. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.10 gm/cc) CARBON FOAM HAVING A LOW COMPRESSIVE STRENGTH.

Top Views

(a) 500X

(b) 800X

(c) 2,000X

Side Views

(a) 500X

(b) 1,000X

(c) 3,000X

Figure 6. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.10 gm/cc) CARBON FOAM HAVING A HIGH COMPRESSIVE STRENGTH.

Scanning electron photomicrographs of the uncoked, low-density foams that were made from a polyurethane-furan resin formulation are contained in Figure 7. As shown, the top row center and right photomicrographs at 100 and 300X, respectively [Views (b) and (c)] clearly show the presence of membranous organic material comprising the windows of the foam cells. The bottom row left and center photomicrographs of Figure 7 taken at 300 and 700X, respectively [Views (d) and (e)] show an intersection of three struts which shows the structural "hole" or "hole defect" already present before the coking treatment. Examination of other struts shown in Figure 7 also reveal that some indentations are in existence before coking.

Scanning electron photomicrographs of typical polyurethane-furan resin foam (0.05 gm/cc) that had been coked at temperatures of 650, 800, and 1,000° C are given in Figures 8 through 11. These foams show the same surface indentation and hole-type defects.

Scanning electron photomicrographs of a 0.05 gm/cc carbon foam obtained from a commercial vendor are seen in Figure 12. This foam exhibited a very low compressive strength (ie, approximately 20 psi), and a large number of "holes" or structural defects in the cell walls of this foam are readily apparent in the photograph.

DISCUSSION

The scanning electron microscope and its use has been described and reviewed by Thornton⁽¹⁾ and others.⁽²⁻⁵⁾ Basically, the SEM operates by focusing a beam of electrons from a heated tungsten filament onto a small area of the specimen. When the electrons strike the surface, they cause the sample to emit low-voltage secondary electrons in amounts determined by the surface structure. These electrons are detected and collected by a scintillation/photomultiplier system. The resulting electrical pulses are used to modulate the brightness of a cathode-ray tube screen which is scanned by synchronism with the electron probe that scans the specimen so that a visible image of the surface is displayed. Photographs in the form of Polaroid pictures can then be taken of areas of interest over a wide range of viewing magnifications.

The scanning electron photomicrographs shown in Figures 1 through 8 were obtained with a JEOLCO, Model JSM-2 stereoscanning microscope. As shown in Figures 2 - 8, surface irregularities in the cell walls of the carbon foams become very apparent when viewed at magnifications of 3,000X. Although higher viewing magnifications were available and tried, it was concluded that 50 to 3,000 magnifications were sufficient to reveal all significant structural

(a) 45X

(b) 100X

(c) 300X

(d) 300X

(e) 700X

(f) 1,000X

Figure 7. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY POLYURETHANE-FURAN RESIN FOAM BEFORE COKING.

(a) 300X

(b) 300X

(c) 1,000X

(d) 1,000X

(e) 1,000X

(f) 1,000X

Figure 8. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM COKED AT 650° C.

(a) 300X

(b) 300X

(c) 300X

(d) 1,000X

(e) 1,000X

(f) 1,000X

Figure 9. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM COKED AT 800° C. (Top Views)

(a) 300X

(b) 300X

(c) 300X

(d) 500X

(e) 1,000X

Figure 10. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM COKED AT 800° C. (Side Views)

Top Views

(a) 100X

(b) 500X

(c) 500X

Side Views

(a) 300X

(b) 500X

(c) 1,000X

Figure 11. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM COKED AT 1,000° C.

(a) 100X

(b) 300X

(c) 300X

(d) 400X

(e) 1,000X

(f) 2,000X

Figure 12. SCANNING ELECTRON PHOTOMICROGRAPHS OF LOW-DENSITY (0.05 gm/cc) CARBON FOAM (Vendor A) HAVING A LOW COMPRESSIVE STRENGTH.

and surface detail for the carbon foams examined. The small angular particles which are especially prevalent in Figures 5 and 6 are actually fragments of the carbon foam which were chipped off in preparing the sample for scanning studies.

A summary of the physical properties and some formulation variables associated with the low-density carbon foams as scanned in Figures 3 through 6 is given in Table 1. The compressive-strength and modulus-of-elasticity values shown in Table 1 are average values representing test results of six individual foam specimens cut from various representative locations of the foam casting.⁽⁶⁾

Table 1
PHYSICAL PROPERTIES OF CARBON FOAMS USED FOR THE
SCANNING ELECTRON PHOTOMICROGRAPHS SHOWN
IN FIGURES 3 THROUGH 6

Foam Strength Designation	Bulk Density (gm/cc)	Compressive Test Orientation(1)	Compressive Strength(2) (psi)	Compressive Modulus of Elasticity (psi x 10 ³)	Ratio of Furan Resin/Urethane
Strong	0.05	Parallel	72	2.8	0.55
Strong	0.05	Perpendicular	40	2.3	0.55
Weak	0.05	Parallel	26	1.0	0.55
Weak	0.05	Perpendicular	24	1.2	0.55
Strong	0.10	Parallel	289	16.2	0.55
Strong	0.10	Perpendicular	209	7.1	0.55
Weak	0.10	Parallel	214	7.4	1.00
Weak	0.10	Perpendicular	136	5.8	1.00

(1) Orientation relates to the direction of the foam rise.

(2) Used unbonded platens and a crosshead speed of 0.05 inch/minute.

Since the polyurethane-furan resin foam before coking is a rigid, organic solid structure, it presented some difficulty for SEM studies. It is a poor conductor of electricity, and, when examined directly, the scanning electron beam created a high potential electron charge. By applying a thin coating of gold (ie, approximately 100 Å thickness) to the foam using a vacuum evaporation technique, the scanning electron photomicrographs that are shown in Figure 7 were readily obtained. The use of vapor-deposited gold for eliminating the buildup of an electron charge on the surface of a sample for SEM studies has also been reported by Stoffey.⁽⁷⁾

The presence of "hole" and indentation defects in the foam while still in the uncoked condition (Figure 7) is important in that it indicates that process variables in the formulation, mixing, and low-temperature oven-curing steps are of primary importance and need to be controlled better. Without the scanning

electron microscope studies, considerable effort could have been needlessly expended on controlling the heating and coking cycles of the foam which would have been to no avail in eliminating these already existent structural defects.

ACKNOWLEDGEMENTS

The authors extend grateful acknowledgement for the assistance of O. Heller who obtained several of the electron photomicrographs shown in this report and V. B. Campbell who provided technical information concerning some of the foam specimens examined.

REFERENCES

- (1) Thornton, P. R.; Scanning Electron Microscopy, Applications to Materials Device Science; Chapman and Hall, Ltd, London (1968).
- (2) "Scanning Electron Microscopy/1968", Proceedings of a Symposium on the Scanning Electron Microscope, The Instrument and Its Applications, April 30-May 1, 1968; IIT Research Institute, Chicago, Illinois (April 1968).
- (3) Oatley, C. W., Nixon, W. C., and Pease, R. F. W.; "Scanning Electron Microscopy", Advanced Electronics and Electron Physics, 21; Academic Press, Inc, New York, New York (1965).
- (4) Minkoff, I.; "Applications of the Scanning Electron Microscope in Materials Science", Jour of Materials Science, 2, pp 388 - 394 (1967).
- (5) Oatley, C. W.; "Looking for Structures", Industrial Research, 10, pp 62 - 65 (July 1968).
- (6) Campbell, V. B.; Personal Communication, Chemical Engineering Development Department, Y-12 Plant.
- (7) Stoffey, D. G.; Chemical and Engineering News, pp 60 - 61, September 23, 1968.