Fractal structure of carbon fibers

S. G. Bogdanov, É. Z. Valiev, and A. N. Pirogov
Institute of Metal Physics, Ural Branch of the Russian Academy of Sciences, 620219, Ekaterinburg, Russia

(Submitted 25 May 1992; resubmitted 5 August 1992)
Pis'ma Zh. Eksp. Teor. Fiz. **56**, No. 5, 254–256 (10 September 1992)

Small-angle neutron scattering by a cloth sample made of carbon fibers reveals that the fibers have a structure typical of bulk and surface fractals. The bulk fractal dimension D and the surface fractal dimension D_s are the same, $D \simeq D_s \simeq 2.7$, within the experimental errors. These results can be explained in a natural way if it is assumed that the fibers of the cloth consist of fractal clusters with a dimension $D \simeq 2.7$ and a radius $R_c \simeq 200$–400 Å which are formed from carbon particles of radius $r_0 \simeq 18$ Å.

Carbon-fiber cloth, which is a sorbent with a well-developed porous structure, is used widely in medicine, the chemical industry, and other fields. Sorption methods are customarily used to analyze the structure of carbon sorbents.[1] However, such methods are incapable of furnishing unambiguous data on the three-dimensional structure of the carbon skeleton of carbon sorbents. This information is necessary for reaching an understanding of the sorption properties of carbon sorbents and also for predicting new fields of application.

In this letter we are reporting an attempt to study the three-dimensional structure of carbon sorbents by small-angle neutron scattering.

The test sample was a cloth made of carbon fibers. The porous structure of the cloth was achieved by the standard procedure for preparing activated charcoal. The starting material was a cellulose nitrate cloth, which was impregnated with a fireproofing compound and then held at (200–300) °C. The carbonization (a heating at 600 °C with air excluded) was then carried out, and an activation in water vapor was carried out at ~850 °C.

The density of this carbon cloth was $\bar{\rho} = 0.48$ g/cm^3.

The small-angle neutron scattering was measured with instruments installed in horizontal channels of the IVV-2M reactor. Measurements over the scattering-vector interval $q = 0.006$–0.13 Å^{-1} were carried out on a scattering apparatus with a central wavelength $\bar{\lambda} = 4.5$ Å and $\Delta\lambda/\bar{\lambda} \approx 30\%$. The measurements in the interval $q = 0.09$–1.0 Å were carried out on an apparatus with a monochromatic neutron beam with $\lambda = 2.42$ Å. To determine the crystal structure of the cloth material, we recorded neutron diffraction patterns and x-ray diffraction patterns in the large-angle region. All these measurements were carried out at room temperature.

Experimental results. It follows from the results of the large-angle neutron diffraction that the carbon atoms form a hexagonal lattice with lattice constants $c \simeq 7.40$ Å and $a \simeq 2.55$ Å. The widths of the Bragg reflections correspond to a radius $r_0 \simeq 18$ Å

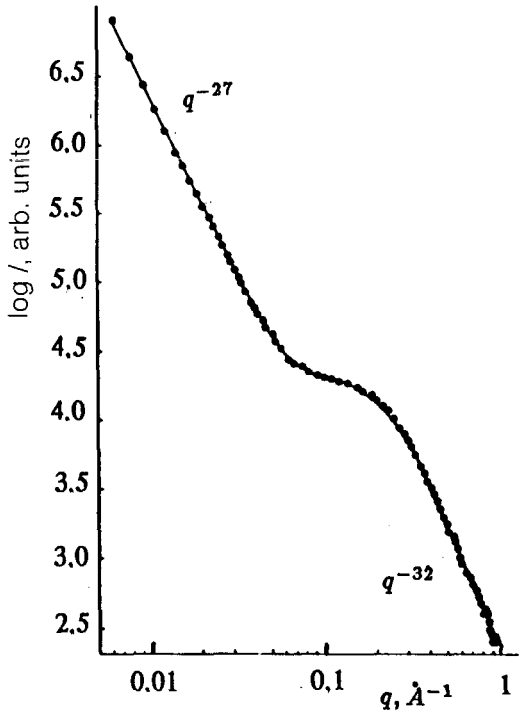

FIG. 1. Angular distribution of the intensity of scattered neutrons, in logarithmic scale.

for the coherent-scattering regions. It can be concluded from these results that the carbon fibers are formed by an ensemble of microscopic carbon particles with a radius $r_0 \simeq 18$Å and a density $\rho_0 \simeq 1.9$ g/cm^3.

Figure 1 shows the intensity of the small-angle neutron scattering, I, versus the wave vector $q = 4\pi \sin\theta/\lambda$ in logarithmic scale. We see two regions in which $\log I$ is a linear function of $\log q$: $q = 0.006$–0.02 and 0.4–1.0 Å^{-1}. In these regions we have

$$I \propto q^{-n} . \tag{1}$$

At small values of q the exponent is $n = 2.7 \pm 0.1$, and at large values of q it is $n = 3.2 \pm 0.1$.

The scattering law in (1) is typical of fractal structures.[2] For the scattering region $q = 0.006$–0.02 Å^{-1}, which corresponds to large distances, we have $n = D$, while for the region $q = 0.04$–1.0 Å^{-1} (small distances) we have $n = 6 - D_s$, where D and D_s are the dimensions of the bulk and surface fractals, respectively. For the carbon fiber which we studied we thus find $D \simeq D_s \simeq 2.7$.

To explain this result we consider a model of a fractal cluster consisting of particles of radius r_0 and density ρ_0 (Ref. 3). The number of particles in a sphere of radius $r \gg r_0$ is given by

$$N(r) = (r/r_0)^D, \qquad 1 < D < 3. \tag{2}$$

From (2) we find the average mass density of the material in a sphere of radius of r to be

$$\rho(r) = \rho_0 \left(\frac{r_0}{r}\right)^{3-D}, \tag{3}$$

and we find the surface area of the particles in a sphere of radius r to be

$$S(r) = r_0^2 \left(\frac{r}{r_0}\right)^D. \tag{4}$$

Equations (3) and (4) constitute the definitions of bulk and surface fractal properties at distances $r \gg r_0$ (Ref. 4). The dimensions which determine the bulk and surface fractal properties are the same, as they are for our test sample. It can therefore be concluded that the carbon fibers are formed from fractal clusters.

Real fractal clusters have a finite size R_c. The size of the cluster in our case could in principle be determined from that value (q_x) of the wave vector at which the scattering intensity deviated from the $q^{-2.7}$ law as $q \to 0$. However, we could not follow this procedure because of the finite resolution of our apparatus. All that we can conclude from the results of the small-angle scattering is $R_c > q_{\min} \simeq 170$ Å. The size R_c can be estimated from expression (3) by setting $\rho(R_c) = \bar{\rho}_B$, where $\bar{\rho}_B$ is the average density of a fiber, $\bar{\rho}_B \simeq (1.5 + 2)\bar{\rho}$, and $\bar{\rho}$ is the density of the cloth. From (3) we then find

$$R_c = r_0(\rho_0/\bar{\rho}_B)^{1/(3-D)} \simeq 18\left(\frac{1.9}{0.96 - 0.72}\right)^{3.33} \text{Å},$$

$$R_c \simeq (175 - 450)\,\text{Å}.$$

It thus follows from the results of our measurements that a carbon fiber consists of fractal clusters and that in the size interval 18 Å $< r <$ 175–450 Å it has both bulk and surface fractal properties. At a scale $r >$ 175–450 Å, the fiber is homogeneous on the average.

The model which we have proposed can also be used to evaluate the sorption characteristics of a carbon cloth. We know from Ref. 3 that in the size region $r_0 < r < R_c$ the size distribution of the pores is $f(r) \propto r^{-D+2}$ for fractal clusters, and the maximum pore size is on the order of R_c. An estimate of the specific surface area of the carbon cloth from the formula $S = 3/(\rho_0 r_0)$ yields $S \simeq 900$ m^2/g. This figure agrees in order of magnitude with the result found by sorption methods.

We also carried out a study of the small-angle neutron scattering of some activated charcoal samples obtained from fruit kernels. The results of these measurements showed that carbon sorbents of this sort, like the carbon filaments, have fractal properties. One might thus expect a fractal structure to be an inherent characteristic of a wide class of carbon sorbents.

[1]M. M. Dubinin, *Carbon Sorbents and Their Use in Industry*, Nauka, Moscow, 1983, p. 100.
[2]D. Schaefer and K. Kefer, in *Proceedings of the Sixth Trieste International Symposium on Fractals in*

Physics, ICTP, Trieste, Italy, 1985.
[3]B. M. Smirnov, Usp. Fiz. Nauk **152**, 133 (1987) [Sov. Phys. Usp. **30**, 420 (1987)].
[4]B. M. Smirnov, Usp. Fiz. Nauk **149**, 177 (1986) [Sov. Phys. Usp. **29**, 481 (1986)].

Translated by D. Parsons