TEXTURE CHARACTERIZATION OF COKE PARTICLES

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INTRODUCTION

The graphitizability and the crystalline structure perfection of high-temperature carbons depend on the degree of crystallites preferred orientation (i.e. the texture) in volumes of several micrometers in diameter. For evaluation of the micro-texture, optical and electronic microscopy and some other methods sensitive to the crystallites' anisotropy are usually used[1-3]. In the present work a quantitative characterization of the texture in cokes and other carbon materials is discussed.

GENERAL CONSIDERATION

The texture of carbon materials depends on size of volumes under consideration. The smaller the volume, the higher the texture. Crystallites spatial arrangement in coke particles, especially in needle cokes, is not random. It turns out that the preferred orientation of neighbor crystallites in cokes does not have even an axial symmetry and can be conditionally represented by a general ellipsoid with the crystallite basal planes being mostly oriented along the major X axis and the normals to the layers being mostly oriented along the minor Z axis (X>Y>Z). Depending on a macrosample forming conditions (extrusion or molding), coke derived carbons may reveal a cylindrical (needle) or plane-like texture. In this respect the coke ability to form probe samples with a maximum possible texture of cylindrical or plane-like types is an important microtexture index.

The average crystallite texture measured for the particles of selected sizes is properly characterized by the texture parameters $\langle \sin^2\theta \rangle$ and $\langle \sin^2\alpha \rangle$, where θ and α are the angles between the normals to the carbon layers and axes z and x, respectively. The angle brackets mean averaging over the particle (or sample) volume. Sometimes it is useful to describe the coke particle texture by the parameter $\langle \sin^2\theta \rangle$ and by 2D parameter $\langle \sin^2\phi \rangle$, where ϕ is the angle between the x-axis and projection of the normal to the carbon layer on the plane (x,y). It is just these parameters which are often used in descriptions of polycrystal macroscopic properties (thermal and electrical conductivity, coefficient of thermal expansion, etc.). For isotropic volumes $\langle \sin^2\theta \rangle = 2/3$, for macrosamples with plane-like texture it lies between 0 and 2/3, and for ones with cylindrical or needle texture the parameter $\langle \sin^2\alpha \rangle$ is between 2/3 and 1.

EXPERIMENTAL RESULTS AND DISCUSSION

In the present paper the texture characteristics of needle and regular cokes are presented. Prior to study the cokes were heat-treated at 2200 °C. Texture parameters were determined on particles of the following fractions: (+2-4), (+1-2), (+0.5-1), (+0.2-0.5), (+0.1-0.2), (+0.05-0.1), (+0.032-0.05), and (-0.032) mm. The particle size for each fraction was conventionally characterized by the average diameter value $\langle D \rangle = (D_{max}+D_{min})/2$, where D_{max}, D_{min} are the maximum and minimum clear sizes of sieves which limited the particles sizes. Probe samples having the maximum possible texture of both cylindrical and plane-like types were prepared for each fraction. The general technique for such a preparation was described in the paper [4]. The average texture parameter for particles in the probe sample was determined from diamagnetic susceptibility measurements, which are traditionally used for texture evaluation [5]. The microtexture parameter in the volumes of about 3-5 μ size across on coke particles of fraction (+20-40) μ was evaluated using electron spin resonance (ESR) investigations followed by a mathematical analysis of the ESR spectra.

The influence of the average particles diameter on the texture parameters $\langle \sin^2\theta \rangle$ and $\langle \sin^2\alpha \rangle$ as indexes of coke ability to form needle and plane-like textures, respectively, for all investigated cokes are presented in Fig.1 and Fig.2. As seen, a highly developed crystallite texture for the needle cokes (cokes 1 and 2) remains in particles up to several millimeters in size, whereas for the regular cokes (cokes 3 and 4) the particles of sizes more than 100 μ are practically isotropic. For smaller particles of both types of cokes the texture is clearly expressed, however for the needle cokes it is remarkably higher. Besides, for small particles (15 - 100 μ) the plane-like texture is always less expressed than needle-like texture of the same cokes. A large difference in the plane-like texture in volumes of about 5 to 20 μ size across tells about different graphitizabilities of the cokes.

It is important to note that besides the large anisotropy of coke particles along the major X and minor Z axes there is also a remarkable texture anisotropy in the basal plane (x,y). The data presented in Fig.3 show the dependence of the texture anisotropy in the plane (x,y) on the sizes of the selected particles. As seen, only the coke 3 has almost an isotropic coefficient of texture in the plane and its value is practically independent of the particle sizes. Physical properties of these coke particles are also axially symmetrical for all the sizes.

CONCLUSION

Many macroscopic properties and quality of polycrystalline carbons and graphites are very sensitive to microtexture of the cokes as raw materials. The new approach to the texture estimation of the cokes makes it possible to establish useful quantitative interrelations between their texture parameters on various levels and many operation properties of coke derived materials including electrodes graphites, structural carbons and graphites, etc.

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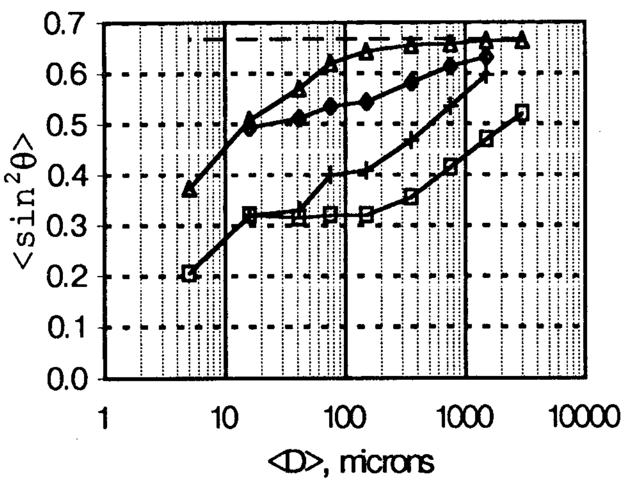

Fig. 1. Plane-like texture parameter vs. particle size .

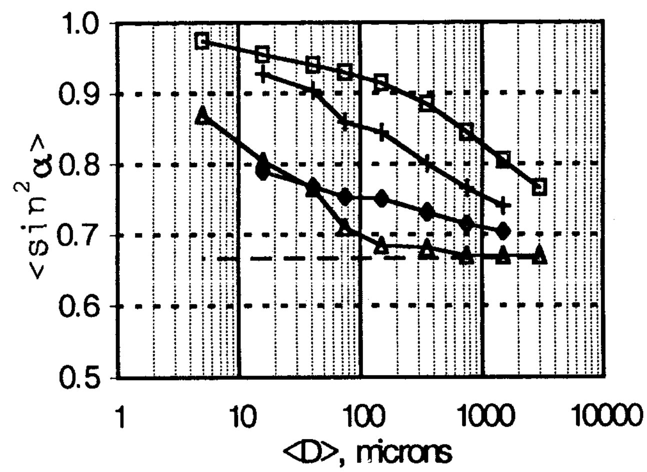

Fig. 2. Needle-like texture parameter vs. particle size.

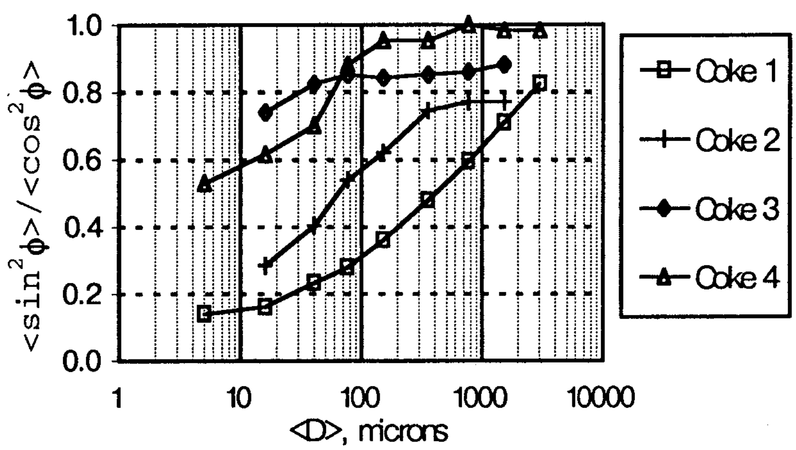

Fig. 3. Texture anisotropy parameter in coke particles basal plane.