POSTER

STRUCTURE CHARACTERIZATION OF C/C COMPOSITE COMPONENTS BY ELECTRON SPIN RESONANCE

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INTRODUCTION

Structure peculiarities of carbon materials (CM) at various structure levels can be studied by the electron spin resonance (ESR). In particular, it gives the opportunity of characterization of CM texture in microvolumes which are "run over" by a spin during the spin-lattice relaxation time [1]. Characteristic sizes of such microvolumes are equal to some microns.

The purpose of the present work is the development of a ESR spectrum analysis method which can make it possible to characterize the structure perfection and the microtexture features of components in multicomponent CM including C/C composites.

RESULTS AND DISCUSSION

The general view of an experimental ESR spectrum of disperse CM sample can give the information on the type of crystallite arrangement in microvolumes. The shapes of ESR spectra and corresponding paramagnetic absorption intensity distributions can be easily calculated for CM having the ideal texures of various types. If there are several non-interacting components having different texture types in a material, the ESR spectrum is a superposition of the absorption lines related to the components.

A method of mathematical processing of ESR spectra of this kind have been developed for analyzing the structure of multicomponent CM. At the first step, the paramagnetic absorption intensity distribution over the resonance magnetic field is calculated from the experimental ESR spectrum by numerically solving the corresponding integral equation. Then, contributions of the components having different texture types can be separated from the total distribution and analyzed.

Fig.1(a) shows a spectrum of a C/C composite which was derived from a PAN-based carbon fiber (CF) heat-treated at 1600°C and of a medium-temperature coal-tar pitch. The final heat treatment temperature (HTT) of the composite was 2400°C. The curve c in Fig.1 shows the calculated absorption intensity distribution which corresponds to the spectrum. Contributions of components having plane and cylindrical texture types are conventionally shown by dashed lines in Fig.1. The fraction of the component having the texture of plane type was evaluated by a mathematical processing of the distribution obtained. It was of about 35% by mass in the composite. This value agrees well with the matrix content which was evaluated by a direct weight method. Therefore, the matrix does not "inherit" the filler texture in the composite under consideration.

On the other hand, shapes of the ESR spectrum and the corresponding absorption intensity distribution in a composite which was obtained by treatment

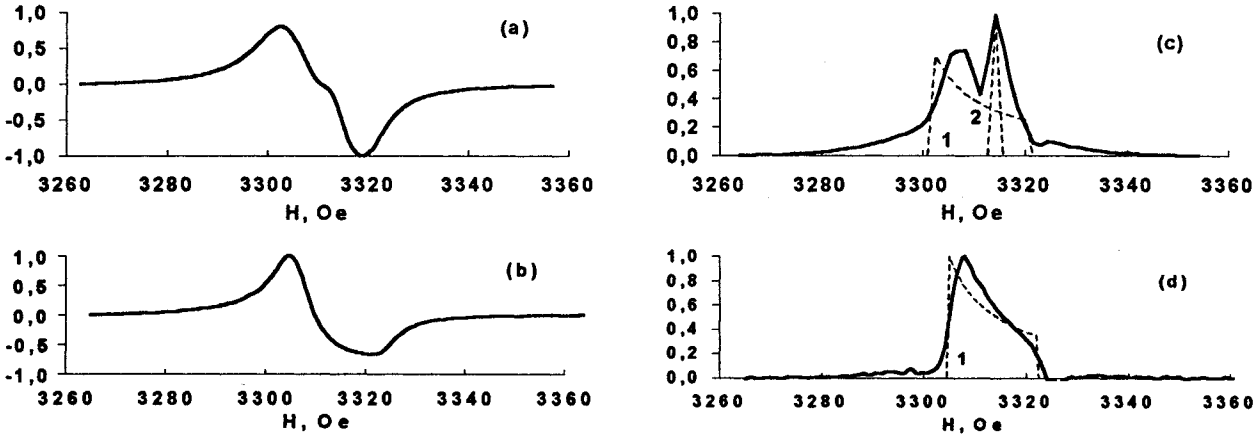

Fig.1. ESR spectra (a, b) and corresponding paramagnetic absorption intensity distributions (c, d) of C/C composites derived from coal tar pitch and PAN-based CF. HTT of CF: 1600 (a, c) and 2800°C (b, d). Dashed curves: contributions of the components having plane (1) and cylindrical (2) texture in microvolumes.

at 2400°C on the basis of the same pitch and PAN-based CF heat-treated at 2800°C are typical of CM having the cylindrical texture in microvolumes (Fig.1 b,d). Thus, interaction of the binder and the filler results in the formation of a matrix with the cylindrical type crystallite arrangement.

A partial "inheritance" of the filler texture by the matrix is illustrated in Fig.2. It shows the absorp tion intensity distribution in a laboratory-made C/C composite with HTT 2400°C. It was made of PAN-based CF (HTT 2800°C) and phenolic resin. Contributions in components having cylindrical (i.e. CF) and isotropic (i.e. resin-derived matrix) textures can be clearly seen in Fig.2. The cylindrical-texture component fraction (about 44%) is close to the CF content in the composite (47% by mass).

As in the case of Fig.2, there is an additional peak on the distribution curve (marked as A). It is associated with a small matrix portion (about 5% by mass) which is modified by the filler-matrix interaction. This modified matrix has the texture which is similar to that of the filler, but the perfections of the crystallite alignment and structure are lower than those of the filler.

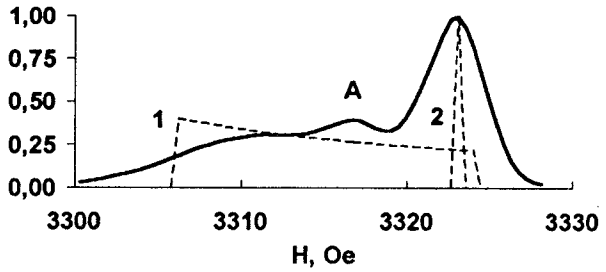

Fig.2. Paramagnetic absorption intensity distribution of a C/C composite derived from phenolic resin and disperse PAN-based CF. Dashed curves: contributions of the components having plane (1) and isotropic (2) texture in microvolumes. A - an additional peak.

Quantitative characteristics of the component structure perfection can be obtained by an additional analysis of the mathematically separated contributions to the experimental ESR spectrum of a multi-component CM.

As an example, the results of ESR spectra analysis of an industrial C/C composite are described. Prior to the ESR measurements, the sample was conditioned to exclude the "oxygen effect" [2]. The absorption intensity distribution mainly consists of a superposition of two contributions: those of components having cylindrical and isotropic texture in microvolumes (Fig.3). They are associated with the fibrous filler and isotropic matrix, respectively. The g-factor values of the filler crystallites are close to those of high-modulus PAN-based CF having HTT of about 2500°C [3]. At the same time, the g-factor values of the matrix crystallites and the localized spin concentration in the matrix coincide with the typical values of non-graphitizable CM with HTT of about 2300-2400°C.

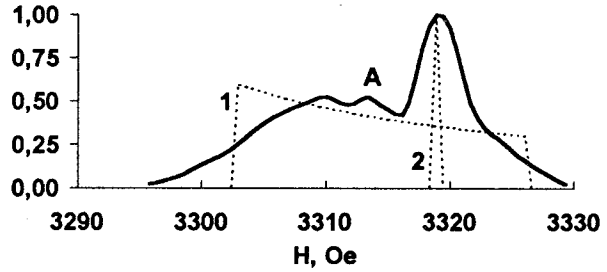

Fig. 3. Paramagnetic absorption intensity distribution of a commercial C/C composite. Designations are the same as in Fig. 2.

As in the case of Fig.2, there is an additional peak (marked as A) on the distribution curve shown in Fig.3. The peak can be associated with a thin matrix layer which is in the immediate contact with the CF surface. The texture of this layer is similar to that of CF.

After an additional treatment of the composite at 2800°C, the crystallite g-factor values of the matrix and filler became close to those of non-graphitizable CM and PAN-based CF, respectively, heat-treated at this temperature [3]. The matrix content was estimated by the analysis of the ESR spectrum of this sample. It is equal to about 10-15% by mass.

Thus, it may be concluded that the investigated C/C composite was derived from a non-graphitizable resin as a binder and PAN-based CF heat-treated at about 2500°C. The final HTT of the composite was about 2300-2400°C.

CONCLUSIONS

Thus, the devised approach to the ESR data analysis gives the possibility to characterize the nature of the components in C/C composites, to evaluate the content and heat treatment temperature of the components and, to estimate the filler effect on the matrix structure.

REFERENCES

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3. A.S. Kotosonov, S.A. Kupriyanov, 20th Conf. on Carbon, Santa Barbara, USA, 1991, P. 542.