STRUCTURAL ANALYSIS OF ACCEPTOR GRAPHITE INTERCALATION COMPOUNDS BY TEM COMBIND WITH IMAGE PROCESSING

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Introduction

Many kinds of carbon-based materials have been contributing to develop the current engineering technology, which have been reinforced by analysis techniques of carbon structure. X-ray diffraction, various spectroscopic, and transport measurements have been widely used as quantitative techniques for analyzing carbon-based materials. However, it is difficult to attain atomic resolution from these results. On the other hand, whole and partial images of materials can be observed by means of transmission electron microscopy (TEM). The microscopic observation, however, is not quantitative by itself.

In this paper, we have studied the microstructures of acceptor GICs with $CuCl_2$ intercalants by means of microscopy combined with image analysis and fuzzy reasoning. We use a 2-dimensional (2D) fast Fourier transform (FFT) for our frequency analysis. From the analysis of the power spectrum obtained by the 2D FFT, we have extracted some specific frequencies and real space images associated with these frequencies by means of the 2D inverse FFT (IFFT). We have found that the stage structure of the GICs consists of specific components that can be separated by the fuzzy reasoning. The relationship between the electron diffraction streak patterns and the microstructure of the GICs is further analyzed here.

Experimental

Vapor grown carbon fibers (VGCFs) [1], which consist of a honeycomb network of concentrically stacked layers of graphene planes around the fiber axis, are used as precursor materials for GICs. Because of their small diameter (~1 μm), VGCFs serve as excellent host materials for the structural analysis of intercalation.

Well-staged intercalated regions can be observed directly in a fiber matrix of VGCFs intercalated with $CuCl_2$-GICs. An 00*l* lattice fringe image obtained from $CuCl_2$ is shown in Fig.1. The staging structure of the GICs is composed of regions of graphite stacking, mixed with stage-1, stage-2, and higher stage regions [2]. The fibers shown in Fig.1 are well-staged acceptor GICs.

TEM images of $CuCl_2$-GIC are processed by 2D-FFT techniques. A power spectrum of $CuCl_2$-GIC obtained from the result of 2D-FFT is shown in Fig.2. The central point corresponds to the brightness of the original TEM image. Its characteristic pattern is distributed in a line perpendicular to the 00*l* lattice planes of the TEM image. The intensity of the power spectrum is represented by graphs obtained by integration along the *x*-axis (parallel to the 00*l*

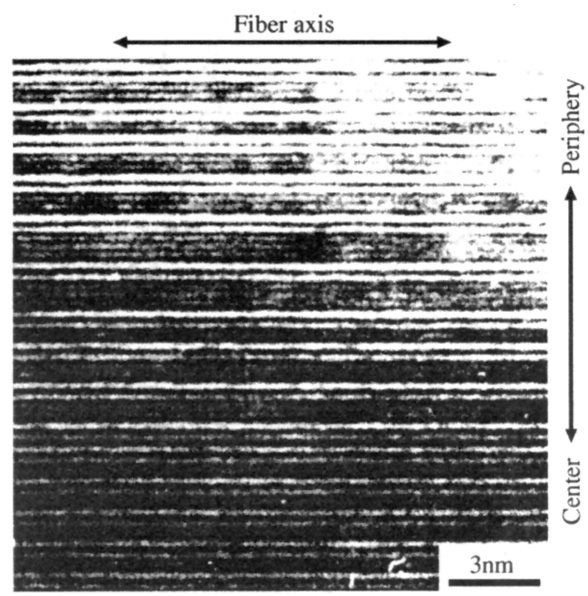

Figure 1. The digitized image from TEM picture of $CuCl_2$-GIC.

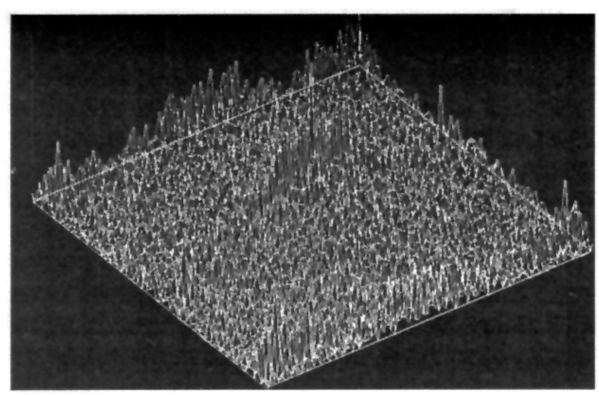

Figure 2. The power spectrum obtained from the TEM image of $CuCl_2$-GIC shown in Fig.1.

lattice planes) for the purpose of analyzing the frequency distribution along the y-axis (perpendicular to the $00l$ lattice planes) [3].

In order to analyze the power spectrum quantitatively and objectively, the characteristics of the samples are extracted from the resulting power spectrum by fuzzy reasoning. We use the degree of peak in the power spectrum to extract the specific space frequency. The degree of the peak is obtained by fuzzy reasoning. All the peaks in the power spectrum except the lowest frequency peak are normalized on the basis of the highest peak and the widest peak. Both the height of the highest peak and the width of the widest peak are set to 1. Then the fuzzy values are defined by membership functions of the height and width of the peaks. The peaks are classified into 3 groups of the degree of peak (Good (G), Normal (N) and Bad (B)) by the mini-max method and the elastic center method of fuzzy reasoning.

Results and discussion

Fig.3 shows the distribution of interlayer spacing obtained by integration along the perpendicular to the line pattern of the power spectrum in Fig.2. The graph shows strong correlations between the intensity of the power spectrum and the spatial frequency. The peaks of the integrated power spectrum shown in Fig. 4 are classified into three groups (G, N, B) by fuzzy reasoning. The degree of peak of the three peaks of 1.39nm, 0.56nm and 0.336nm, shown in the black color in Fig. 4, was estimated to be Good. The results of the degree of peak of the gray peaks and the light gray peaks were Normal and Bad, respectively. From this frequency analysis, it is clear that $CuCl_2$-GIC indicates evidence of stage-1 (the interlayer repeat distances (IRD): 0.93nm), stage-2 and stage-3 regions (IRD: around 1.39nm), and graphite stacking (IRD: 0.336nm), but the presence of the stage-1 region is not evident in the TEM image.

Real space images were reconstructed by taking the 2D-IFFT in order to verify the staging structure of the $CuCl_2$-GIC discussed above. As a result of these analyses, it is clear that the lattice fringe image of the $CuCl_2$-GIC (Fig.1) consists of certain images which correspond to specific frequencies that are revealed by analysis of the power spectrum, where stage-2 and stage-3 images are shown to be dominant in the $00l$ graphite lattice fringe image.

Conclusions

The staging structure of $CuCl_2$-GICs is revealed through the frequency analysis of the TEM image using image processing and fuzzy reasoning. The image processing method is practical and useful for further analysis of changes in the structure within a very small area. We also expect that this approach will also be useful for analysis of structural characteristics of other carbon materials.

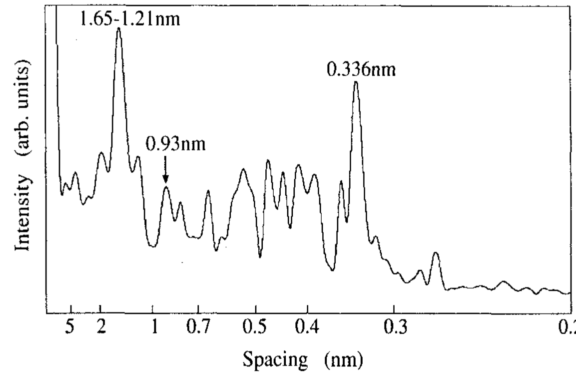

Figure 3. Integration of power spectra of Fig.2.

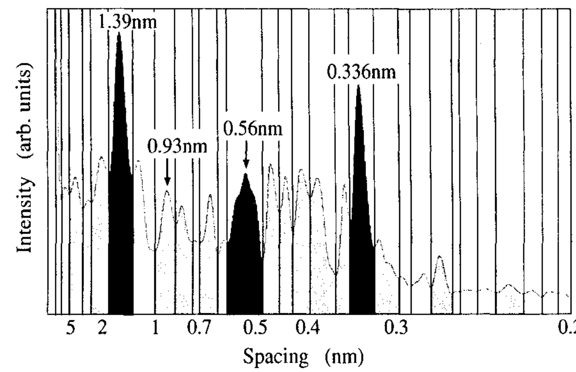

Figure 4. Judgment of the degree of the peaks obtained by integration of the power spectrum of Fig.3 by fuzzy reasoning.

References

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Acknowledgments

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