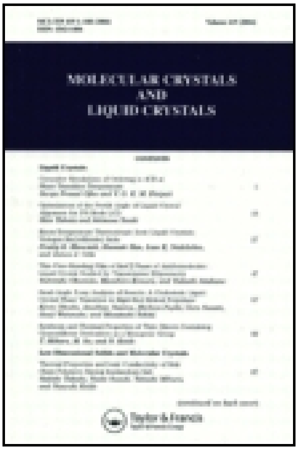

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Application of Image Analysis for TEM Image of Acceptor Graphite Intercalation Compound

Takashi Miyazaki [a], Kyoichi Oshida [a], Morinobu Endo [b] & Mildred S. Dresselhaus [c]

[a] Nagano national college of technology, Nagano, 381-8550, Japan

[b] Shinshu University, Nagano, 380-8553, Japan

[c] Massachusetts Institute of Technology, Cambridge, Massachusetts, 02139, USA

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Application of Image Analysis for TEM Image of Acceptor Graphite Intercalation Compound

TAKASHI MIYAZAKI[a], KYOICHI OSHIDA[a], MORINOBU ENDO[b] and MILDRED S. DRESSELHAUS[c]

[a]*Nagano national college of technology, Nagano 381–8550, Japan,* [b]*Shinshu University, Nagano 380–8553, Japan and* [c]*Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, USA*

A new approach to study of micro texture and structure of graphite intercalation compounds (GICs) is shown using high resolution transmission electron microscopy (TEM) combined with image analysis and fuzzy reasoning. As a result of application of the frequency analysis using 2-dimensional (2D) fast Fourier transform (FFT) to the $CuCl_2$-GICs, a characteristic power spectrum pattern called streak, which is similar to an electron diffraction pattern, is obtained. The images correspond to the specific frequencies are reconstructed by 2D inverse FFT (IFFT). Then the stage structure of $CuCl_2$-GICs is investigated.

Keywords: acceptor-graphite intercalation compounds; transmission electron microscopy; image analysis; fuzzy reasoning; first Fourier transform

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 $CuCl_2$s. An 00*l* lattice fringe image obtained from $CuCl_2$-GIC 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

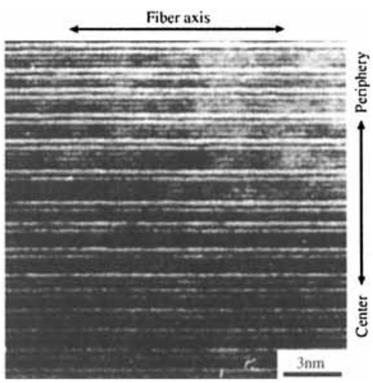

FIGURE 1 The digitized image from TEM picture of $CuCl_2$-GIC.

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* lattice planes) for the purpose of analyzing the frequency distribution along the *y*-axis (perpendicular to the 00*l* 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

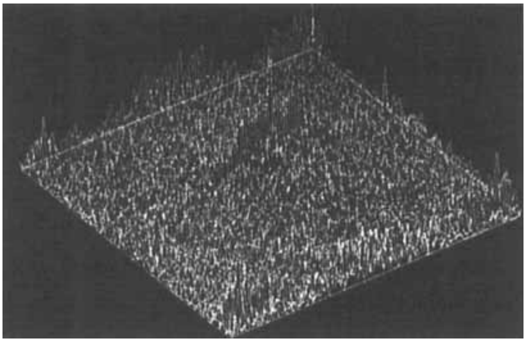

FIGURE 2 The power spectrum obtained from the TEM image of $CuCl_2$-GIC shown in Fig.1.

by fuzzy reasoning [4]. 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 obtained by membership functions of the height and width of the peaks. The membership functions are defined by the following formulas:

$$\mu_G(x) = 2x-1 \quad (1)$$

$$\mu_N(x) = 1-|2x-1| \quad (2)$$

$$\mu_B(x) = 1-2x \quad (3)$$

in which x is the degree of the height or the width. Equation 1 is used in the case of the degree of the height or the width is over 0.5, and equation 3 is used when it is under 0.5. Equation 2 is used with equation 1 or 3. 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.

We can find specific frequencies corresponding to the periodicities in the original images by analyzing the graphs of the power spectrum. The specific frequencies in the power spectrum of $CuCl_2$-GICs were extracted and real space images were reconstructed by using a 2D IFFT. Since the original image of $CuCl_2$-GICs is thought to consist of the reconstructed images with different frequencies, the images were superimposed to analyze the staging structure of $CuCl_2$-GICs.

RESULTS AND DISCUSSION

Frequency Analysis

Figure 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 for 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 gray peaks and the light gray peaks were judged to be Normal and Bad, respectively. From this

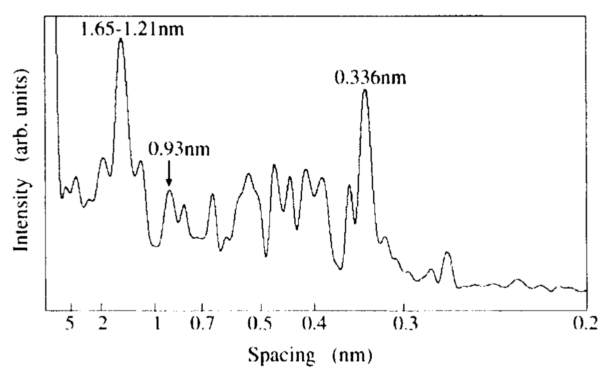

FIGURE 3 Integration of power spectra of Fig. 2.

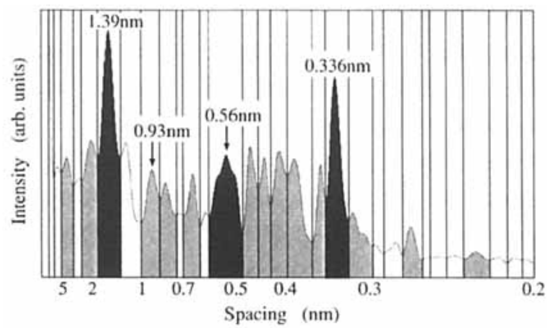

FIGURE 4 Judgment of the degree of the peaks obtained by integration of the power spectrum of Fig. 3 by fuzzy reasoning.

frequency analysis, it is clear that the stage structure of $CuCl_2$-GIC contains 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.

Reconstruction of the Image

Real space images were reconstructed by taking the 2D IFFT in order to verify the staging structure of the $CuCl_2$ VGCFs discussed above. The spcific frequency components of 0.336nm correspond to the 002 lattice fringe and 1.396 correspond to the stage-2 or stage-3 regions were extracted, and reconstructed real space images. The two images are easily superimposed upon each other by the 2D IFFT. The figure obtained by superimposing these two

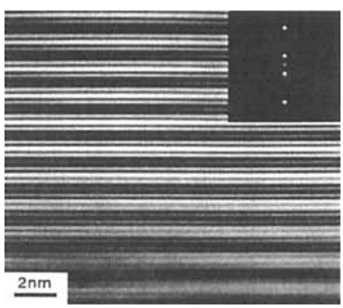

FIGURE 5 Image of the IFFT for the $CuCl_2$-GIC sample corresponding to the superposition two interlayer spacings, 0.336nm, and 1.396nm.

images is shown in Fig. 5. The inset to Fig. 4 shows the selected area pattern of the power spectrum that is used for the extraction of the specific frequencies in the image pattern. The central points of the power spectrum were also selected for maintaining the brightness of the real space image. In Fig. 4, we find that an image similar to the stage image of GICs can be constructed from two different frequencies corresponding to the high intensity peaks in the power spectrum. But the superimposed image (Fig. 4) differs slightly from the original image shown in Fig. 1, which may contain stage-1, -2, -3 regions. The other frequency components are hypothesized to contribute to the detail construction of the original image.

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 00*l* 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.

Acknowledgments

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