STRUCTURAL ANALYSIS OF PORE OF ACTIVATED CARBON FIBERS BY USING HIGH RESOLUTION TRANSMISSION ELECTRON MICROSCOPY AND IMAGE PROCESSING

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Introduction

Activated carbon fibers (ACF's) are disordered materials, and the pore size of them is very small of a nano meter scale. High resolution (HR) transmission electron microscopy (TEM) is an effective method to analyze micro structure of carbon materials. However, TEM is generally qualitative and is difficult to be used for quantitative analyses. We have been studying the pore structure of ACF's by HR-TEM combined with image analysis [1,2]. In the present study, we discussed the relationship between the defocus value, which is one of the most significant parameters for the TEM observation, and the TEM image of ACF's.

Experimental

TEM images, especially of disordered materials like ACF's, are strongly depended on the defocus value (*delta f*) of the TEM at the high-resolution observation of nano meter scale [3]. In order to estimate the optimum *delta f* for the pore observation of ACF's, a mesophase pitch-based ACF, of which specific surface area (SSA) is 1280m^2/g measured by gas adsorption, is observed with various values of *delta f*'s using a 400kV acceleration voltage TEM (JEM4000EX, JEOL). The power spectra of the TEM images obtained by 2 dimensional (2D) first Fourier transform (FFT) were compared with the phase transfer functions of the TEM. The same sample was observed by using a 200kV acceleration voltage TEM (JEM2010FEF, JEOL), and the TEM images were compared to the images of JEM4000EX.

Results and Discussion

The phase transfer function cos(x)'s of the TEM for different *delta f*'s (*delta f*=-60nm, -150nm, and -250nm) are shown in Fig.1 (a), (b), and (c), respectively. The contrast of TEM image appears strongly when the absolute value of the phase transfer function is over 0.5. Since the intensity of brightness of the TEM image decreases with increase of spatial frequency, the first negative peak is important to form the TEM image. It is expected that the well contrast TEM images will be obtained at the range around the first negative peaks of the phase transfer function.

The TEM images of the same area for different *delta f*'s are shown in Fig.2. The bright parts are correspond to the pores of ACF's [4]. We interpret the observation that these three images look similar to one another (except for the scale of the images) suggesting the fractal nature of the pore structure. As a result of changing *delta f*'s, we can observe different size texture of the pores. The power spectra obtained by 2D-FFT were represented by graphs obtained by integration around their central points (see Fig.3 (a) – (c)) [1]. The insets of Figs.3 (a) – (c) are the power spectra. We think that the pore size distribution of the ACF is closely related to the integrated power spectrum. The integrated power spectra correspond to the phase transfer function shown in Fig.1. The range of pore size distribution shifts to the larger spacing range with decrease of *delta f*. The experimental results show that the pores of different range in the size can be selectively observed if we select the proper *delta f*.

Fig.4 shows the phase transfer function of JEM2010FEF at *delta f*=-150nm which is approximately equal to that of JEM4000EX at *delta f*=-90nm. The TEM image of the ACF sample observed by JEM2010FEF and its power spectrum are shown in Figs.5 and 6, respectively. The structure shown in the TEM image of JEM2010FEF is rather different from that observed by JEM4000EX. This difference seems to be caused by the difference of resolution of each TEM, however, the detailed information is unknown. There are some other parameters (for example, the spherical aberration constant: *Cs*, the chromatic aberration constant: *Cc* etc.), which affect the TEM image.

When we use a TEM of different type, careful discussion on the TEM image under consideration to these parameters is required.

Conclusions

As we predicted, the TEM image of ACF, which presents an amorphous structure, changes sensitively when the *delta f* shifts. The texture of ACF of different magnitude was selectively observable with consideration of the phase transfer function corresponding to the *delta f* and of power spectrum. It is expected that the images observed by different type TEM could be usefully compared each other when we choose suitable parameters.

References

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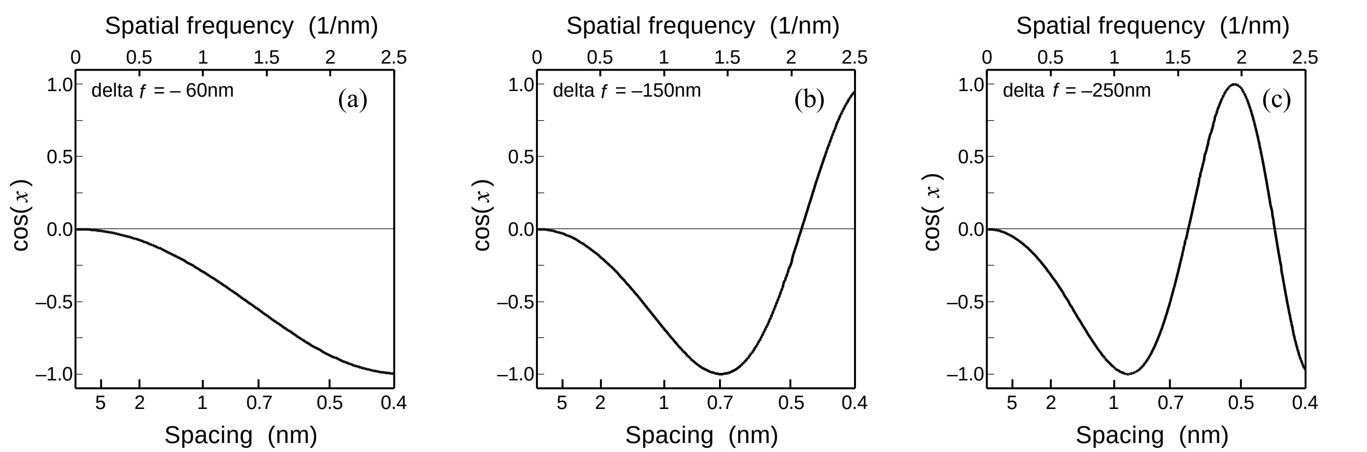
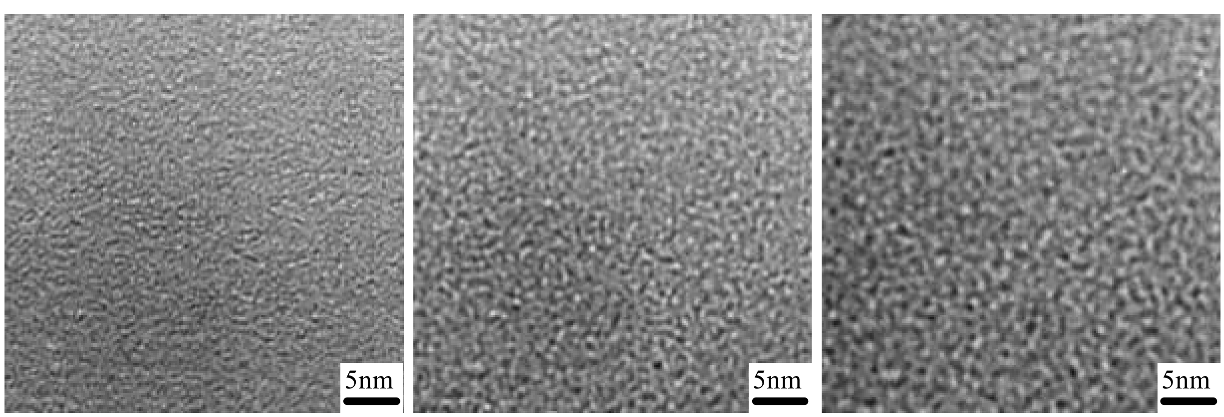
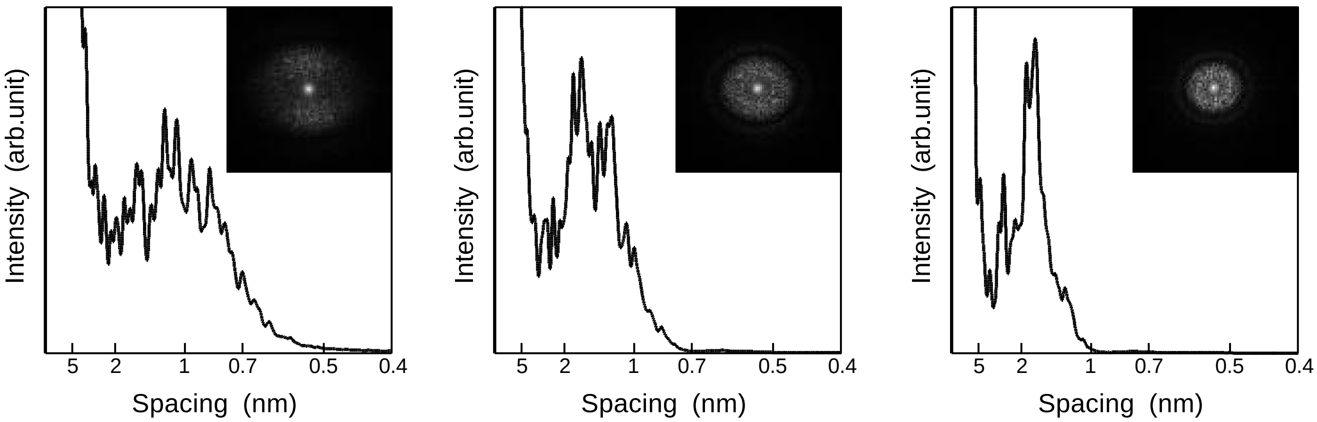


Figure 1. The phase transfer functions for different *delta f*'s of JEM4000EX.



(a) delta *f* = -60nm (b) delta *f* = -150nm (c) delta *f* = -250nm

Figure 2. The TEM images for different *delta f*'s of JEM4000EX.



(a) *delta f* = -60nm (b) *delta f* = -150nm (c) *delta f* = -250nm

Figure 3. The insets power spectra for different *delta f*'s of JEM4000EX are integrated around the center points of them.

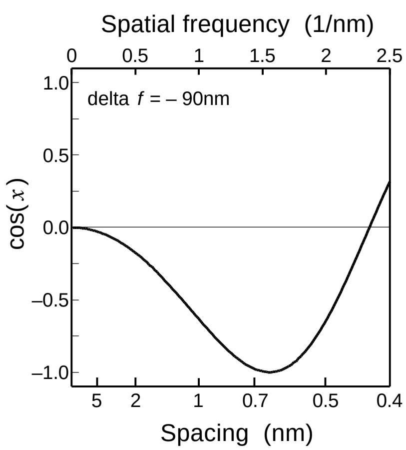


Figure 4. The phase transfer function for *delta f*=-90nm of JEM2010FEF. The value of *delta f* approximately corresponds to that of JEM4000EX at *delta f*=-90nm.

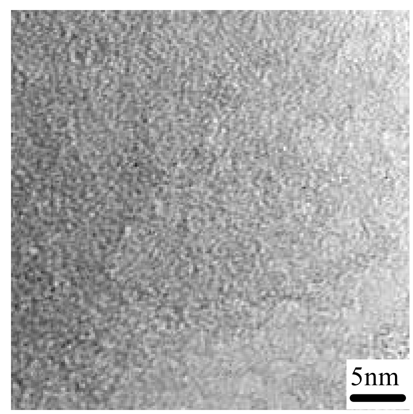


Figure 5. The TEM image for *delta f*=-90nm of JEM2010FEF.

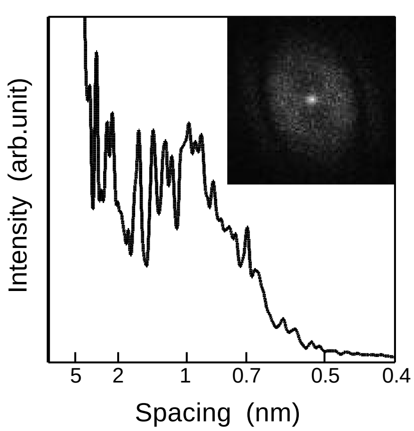


Figure 6. The inset power spectrum *delta f*=-90nm of JEM2010FEF is integrated around the center point of it.