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Precise Measurements of Transmission Attenuation in Mass-Thickness Contrast TEM Images

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Since material's properties are strongly influenced by density and distribution of localized structures, the capability to observe a large volume inside bulk, especially to observe a thick specimen should be important to make efficient and reliable analyses. Discussion on the maximum observable thickness is of growing importance also in recent research trends such as electron tomography of micron-sized materials [1] and in-situ observations in gas or liquid environments. For efficient designing of those experimental researches, it should be greatly helpful if pre-estimation of the maximum observable thickness by TEMs becomes possible. Thus, deep understanding of the transmission attenuation feature is one of the important issues to achieve estimation of the observable thickness. In the present study, the transmission attenuations were precisely analyzed using 200–3000 keV electron beams.

The transmission T for a polystyrene sphere was measured along the dashed line in Fig. 1(a) to compare with the sphere thickness as shown in Fig. 1(b). Then the relation between them was converted to the attenuation curve in the chart of thickness-$\log_{10}T$. Figure 2(a) shows the attenuation curves for 500–3000 keV electrons. If the kinematical approximation of electron scatterings is valid in a material, the transmission decays exponentially with increasing thickness (Beer's law) and appears on a straight line in the $\log_{10}T$ chart. Thus the attenuation curves with downward convex features in Fig. 2(a) are considered as a multiple scattering effect [1]. On the other hand, in the early stage shown in Fig. 2(b), the attenuation curves are not linear but slightly convex upward. This should be a quite unanticipated result which means breakdown of Beer's law even in the early stage of the transmission attenuation.

Based on these results, we proposed a function containing three parameters to express the nonlinear attenuation curves. Our mathematical model and other models proposed previously were compared with the experimental data measured in a 200-kV TEM and high-voltage electron microscopes at Osaka University over a wide range of conditions: acceleration voltage of 200–3000 kV, objective aperture radius of 0.85–30 nm^{-1}, and thickness of 0.25–10 μm [2]. As shown in Fig. 2(a) and (b), our model can excellently reproduce all of the measurements with a high degree of precision, although the other existing models are valid only in limited imaging conditions and/or in limited thickness ranges. Thus, a quantitative description of the transmission attenuation in normal TEM imaging conditions was clarified finally after more than a century since early researches using β-rays [3]. Figure 3 shows schematics of the overall shape of an attenuation curve and the corresponding evolution of the attenuation coefficient. The convex curvature in the early stage means that the attenuation coefficient μ has increased from the kinematical coefficient μ_k. Beyond the kinematical range, an "almost" linear attenuation appears with the linear attenuation coefficient μ_0, which is a mean value of μ(t) in the linear range. The characteristic behavior of the attenuation curves can be explained intuitively based on a simplified model of multiple scattering [2, 4].

References:

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[4] The authors are grateful to Prof. H. Mori for invaluable discussion and Mr. E. Taguchi, Mr. T. Sakata and Mr. T. Yasuda of Osaka university, and Mr. A. Ohsaki, Dr. S. Ohta, and Mr. S. Takakuwa of JEOL Co., Ltd., and Dr. S. Arai of Nagoya university for their assistance with the experiments. This work was partly supported by MEXT KAKENHI (grant number 26105009).

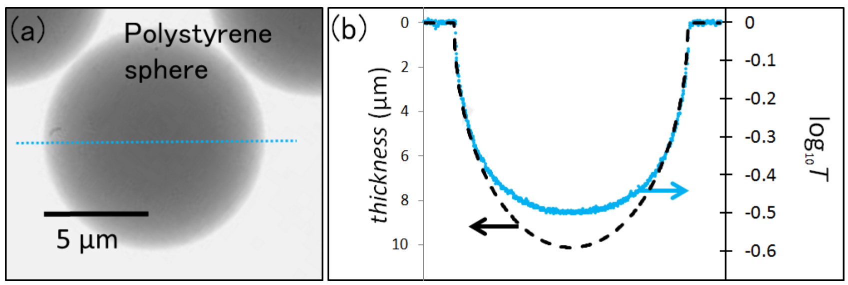

Figure 1. The procedure to measure the relation between the electron transmission and thickness. (a) TEM image of polystyrene spheres with diameters of about 10 µm taken at 3000 kV using an objective aperture with a radius of 17 nm^{-1}. (b) Comparison between the transmission T along the dotted line in (a) and projected thickness of the sphere calculated from the diameter.

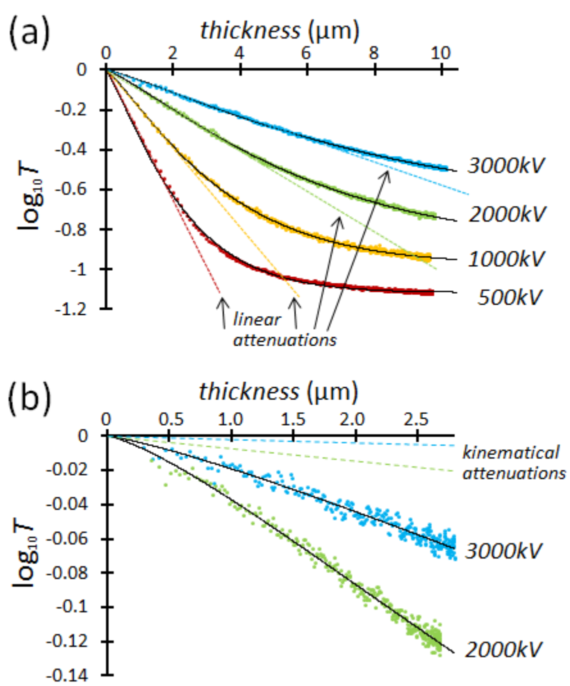

Figure 2. Transmission attenuation curves in polystyrene. (a) The overall shape and (b) the early stage of the attenuation. The dotted lines represent linear or kinematical attenuations. The solid curves show the fitting results by our model.

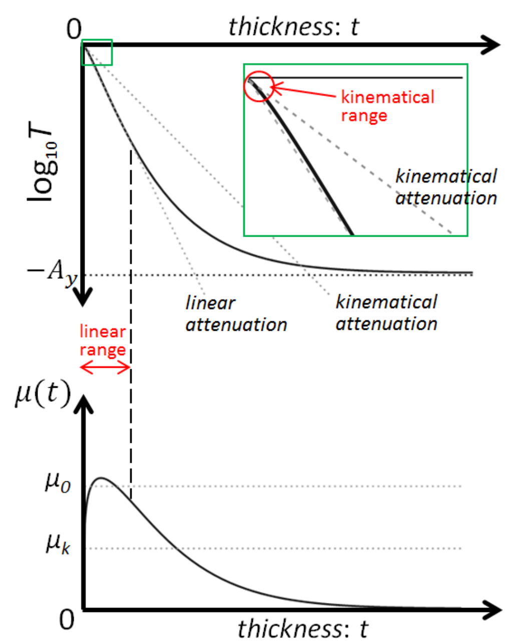

Figure 3. Schematic diagrams of the nonlinear transmission attenuation. In the lower panel, the attenuation coefficient is compared to linear and kinematical attenuations with stationary coefficients μ_0 and μ_k, respectively. The early stage is magnified in the inset.

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