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Enhancement of the TEM image contrast and elemental mapping using EFTEM combined with cryo-TEM for particles in aqueous solution

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Applications of energy filtering and elemental mapping using energy filtered TEM (EFTEM) for multi-component particles, such as emulsions and vesicles, in aqueous solution is attractive because chemical information and enhanced image contrast can be obtained. Because of the film thickness and possible contamination during the process on bright field cryo-TEM imaging techniques, inherently poor image contrast by the multi-component particles in vitrified water film has been an issue to be resolved. To overcome these hurdles, zero-loss filtered imaging and elemental mapping using EFTEM are being increasingly applied for these materials. However, relatively little work has been done on the filtering images and elemental mapping of the multi-component particles in solution. One of the reasons for this is that the required electron dose needed for elemental mapping easily exceeds the allowable amount e- dose for cryo-TEM of frozen hydrated samples. Because of this, elemental mapping using EELS is usually done for freeze-dried samples. (1, 2)

An emulsion is a stable mixture of two or more immiscible components and one of the components forms fine particles held in suspension by small amounts of surfactants. For the morphological investigation of emulsions, cryo-TEM has been commonly used to visualize the morphological distribution. (3-5) In the study of the formation of multi-component emulsions forming either an acorn shape or a multi-lobed shape, the information of the component distribution in the emulsions is also essential to understand and to design the process to get the desirable ultimate properties of the emulsions. Elemental mapping using EFTEM coupled with Cryo-TEM for the emulsions in vitrified water was possible, although beam damage on the sample due to relatively high e- dose resulted in bubbling and dehydration.

Recent results of energy filtered silicone/poly(methyl methacrylate) (PMMA) emulsions having acorn shapes in aqueous solution are presented in Figures 1 and 2. Energy filtering for emulsions in aqueous solution proved to enhance the image contrast to resolve finer detail in the images, as shown in Figure 1. The zero-loss energy filtering resulted in aberration reduced image contrast. The filtered image (Fig. 1a) with lower beam dose intensity revealed a more highly resolved image than the unfiltered one (Fig. 1b). In the elemental mapping using EFTEM, although beam induced bubbling and dehydration due to multiple exposures for the elemental mapping occurred, elemental mapped images of Si, O and C for silicone/PMMA emulsions were successfully obtained, as shown in Figure 2. The Si and C mapped images clearly indicate the acorn structure having a PMMA polymer joined with a silicone polymer within the emulsion. These data were collected on the JEOL 2100F FEG TEM/STEM interfaced with Gatan 2002 GIF and Digital Micrograph data acquisition and control in Dow Corning at 200 KeV. The collection angle was 1.56 mrad.

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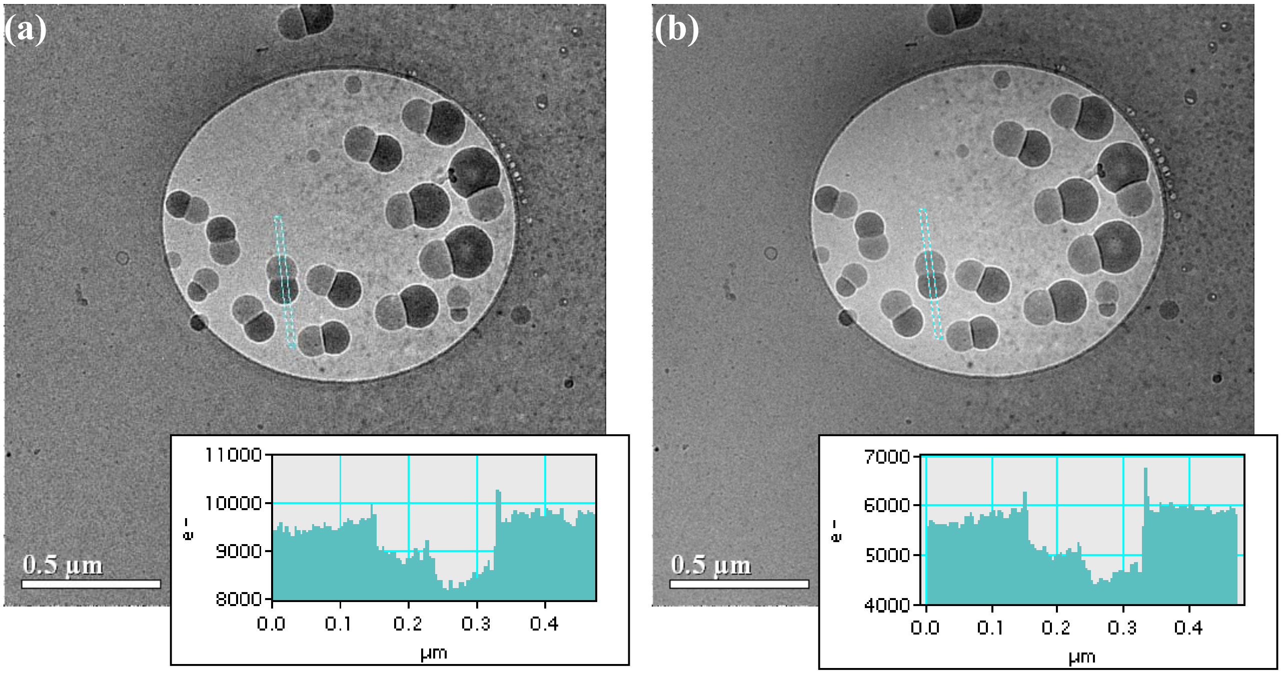

Figure 1. TEM images of emulsions having acorn structure in vitrified water film with line profiles from the lined region in the images; (a) unfiltered image; (b) zeroloss filtered image

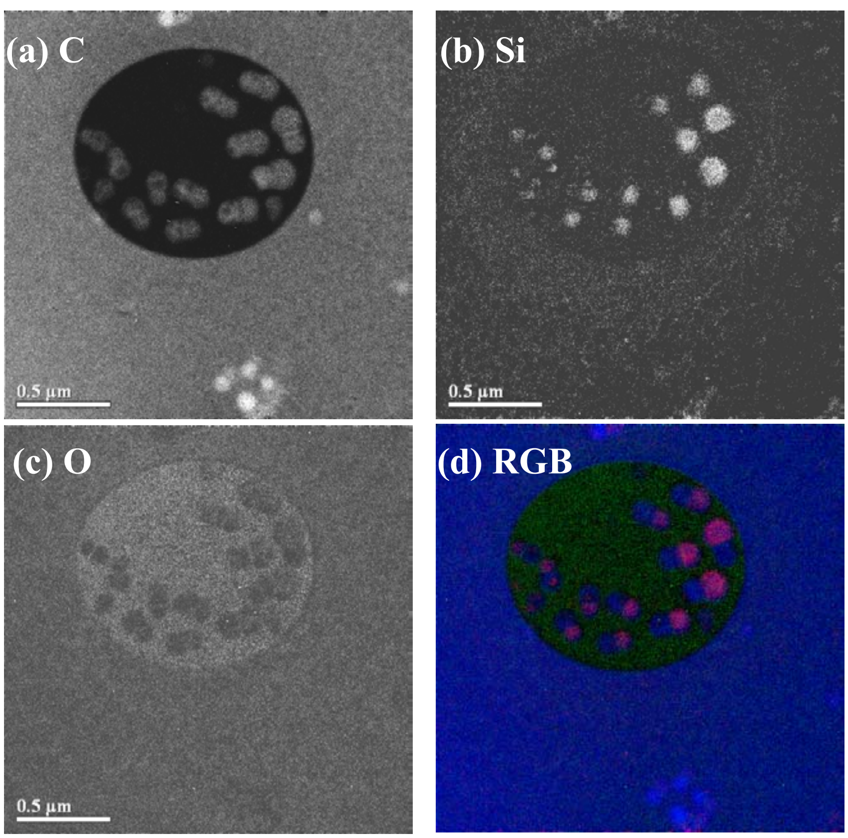

Figure 2. Elemental maps for acorn emulsions in vitrified water film shown in Figure 1: (a) C map; (b) Si map; (c) O map; (d) RGB image (red: Si, blue: C, green: O)

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