RECENT DEVELOPMENTS IN ELECTRON TOMOGRAPHY AND FIB/SEM SERIAL SECTIONING for 3D MORPHOLOGICAL ANALYSIS IN POLYMER COMPOSITES.

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Introduction: In order to satisfy the increasing demands for high-performance polymeric materials, polymers are often mixed with inorganic or metal components. In such composite materials, often called polymer composites, the polymers usually constitute the matrix whereas the metal or inorganic compounds are mixed to achieve certain additional properties, such as mechanical, electrical, or optical properties. Three-dimensional (3D) structures of such composite materials are essential to understand their properties.

Electron tomography [1], alternatively called 3D TEM, has been used not only to visualize the morphologies but also to predict properties together with computer simulation techniques [2]. A forcused ion beam (FIB) equipped with scanning electron microscopy (SEM) has also been used to investigate 3D morphologies of polymer composites. While electron tomography has higher resolution than FIB/SEM serial sectioning, FIB/SEM for short, observable volume is much larger for FIB/SEM compared with electron tomography.

3D morphological observations under stretching in nanometer scale resolution: Recent main interest in electron microscopy is to go beyond the 3D imaging, adding one more dimension. In polymer composites, deformation can be a key "dimension". Namely, it is particularly important to observe *in-situ* morphologies under (uniaxial) stretching. Recently, we have developed a 3D tomography holder for in-situ tensile deformation for polymeric materials in transmission electron microscope (TEM) [3]. Unlike the existing stretching holders, the newly-developed holder has some unique futures: the drifting of field of view for TEM observations becomes minimum because both ends of (microtomed) thin sections are stretched simultaneously at the same stretching rate. Another essential feature of the newly-developed holder is that we can achieve much larger strain can be applied to the specimen than the predecessor holder does: the largest strain achievable with this tensile deformation holder is, in principle, about 50 (when the initial specimen length is 20 μm). Moreover, the tensile holder allows us to tilt the specimen up to 60 ~ 75°for tomographic reconstruction.

The tensile deformation tomographic holder was used to observe deformation processes of rubber composite, in which nano-scale fillers dispersed in polymeric matrix. The morphological changes of rubber composites as well as the fracture processes under stretching were directly observed at nanometer scale. It was successfully observed that the aggregates were deformed under extension, and then the nano-voids formed around the aggregates.

Super-resolution for asymmetric resolution of FIB-SEM using AI with deep learning: In FIB-SEM, the specimen surface is stripped by an ion beam and imaged by an SEM installed orthogonally to the FIB. The lateral resolution is governed by the SEM, while the depth resolution, i.e., the FIB milling direction, is determined by the thickness of the stripped thin layer. Thus, in most cases, the lateral resolution is superior to the depth resolution. In polymeric systems, the depth resolution is typically on the order of 10 nm (most likely 20 nm), so the application of FIB/SEM is limited to morphologies with rather large characteristic length. [4].

Here, a new approach based on an image-processing or deep-learning-based method for super-resolution of 3D images with such *asymmetric resolution* is proposed [5]. In this way, it became possible to restore the depth resolution to achieve symmetric resolution. It was found that this this new methodology was effective not only to restore the depth resolution but also to considerably reduce the observation time of polymer composite materials. Details will be given at the conference time.

References:

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