The spatial relationship & composition of functional components within polymer materials is theorized to control bulk properties such as conductivity [1,2,3], however, characterizing 3D structure within a bulk material is not always easy due to the “soft” nature of the materials and similarities in filler components and bulk composition. Similar material investigation of components within bulk material are achieved by traditional FIB-SEM, slice and view techniques where the focused ion beam is used to cut through a bulk material and an electron beam of the SEM is used to characterize the composition and structure within successive slices of a 3D volume. After the slice images are obtained a 3D model can be generated and analyzing the elucidated structural relationship is possible.

Typically FIB-SEM 3D characterization is done with hard materials where the technique is capable of ~4nm resolution in x/y/z directions creating a symmetrical voxel for 3D reconstruction. “Soft” polymeric materials, however, exhibit various milling and imaging artifacts when traditional methodologies are used requiring the development of new low dose imaging and milling procedures to be used for 3D volume and (S)TEM sample preparation. In Fig. 1, images showing the effects of standard (33.6 nC/µm²) and low dose (7.57 nC/µm²) techniques on shrinkage, melting and general structural damage is compared as applied to extraction of a (S)TEM lamella from a bulk polymer material. This illustrates the operational conditions will not add artifacts for the 3D data set acquisition which was subsequently performed. A conductive rubber developed in Mexico (patent application MX/a/2013/014435) was tested by this technique to see if it is possible to image the component materials in the bulk to understand the inter particle spacing and composition.

Investigation of the conductive rubber was done with an automated slice and view procedure on the FEI Versa 3D DualBeam and slice-image data was processed with FEI Aviso Fire software to yield particle orientation and spacing. This data appears to align with the basic models presented in the textbook by Milton [1]. This new low dose technique appears suitable for analyzing composite soft materials and should be applied to additional samples.

References:
Fig. 1: Damage occurs in polymeric materials when traditional FIB milling conditions are used that result in a high ion dose (top image). Reducing the focused ion beam dose (by over 75%) eliminates damage to the sensitive polymeric materials (lower image).