Voltage contrast imaging of carbon nanotube filler in rubber

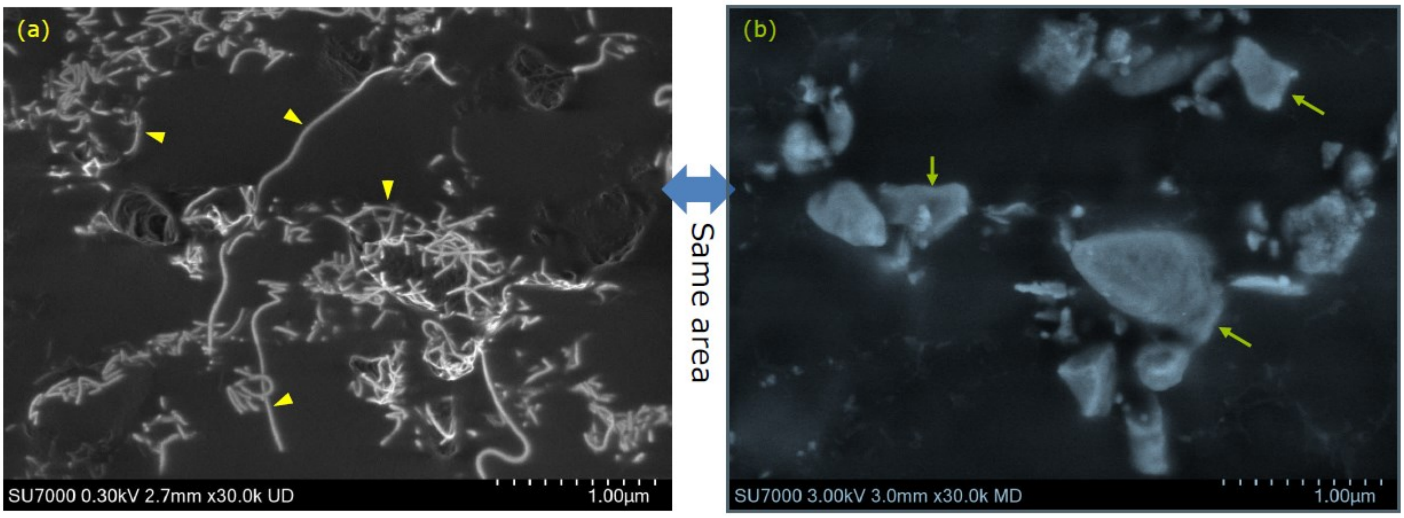
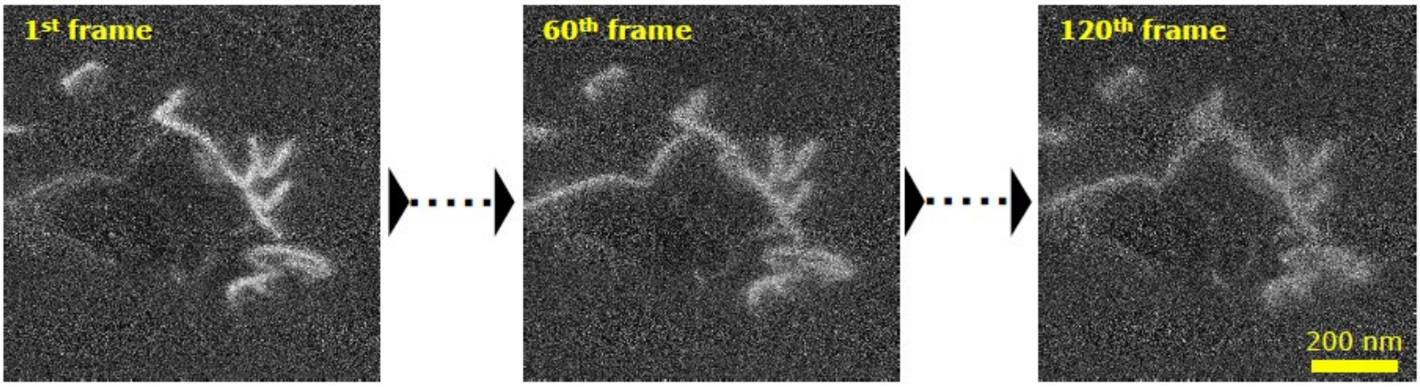


Figure 1. Voltage contrast image (a) and material contrast image (b) of rubber containing carbon nanotube filler
Instrument : SU7000 FE-SEM Accelerating voltage : (a) 0.3 kV, (b) 3 kV
Magnification : 30 kx Detector (signal) : (a) Upper detector (SE), (b) Middle detector (BSE)

Carbon nanotube (CNT) has been used as rubber fillers recently in addition to conventionally used carbon black and silica particles. The distribution of CNT in rubber is important because it affects the properties of the rubber. The small difference in average atomic number between the CNT and the rubber makes distinction of them difficult with enough difference in material contrast by BSE signal. On the other hand, voltage contrast (VC) by SE signal enables to distinguish them by the difference of surface potential changing their SE signal intensities. Fig. 1 shows an SE image (a) of the rubber taken at 0.3 kV and a BSE image (b) of the same area taken at 3 kV. CNT could be clearly observed as bright lines in the SE image (arrow heads in Fig. 1(a)). While in the BSE image, CNT was invisible; only silica particles were observed brightly (arrows in Fig 1(b)).



(a) Single frame images (1st, 60th, 120th frame)

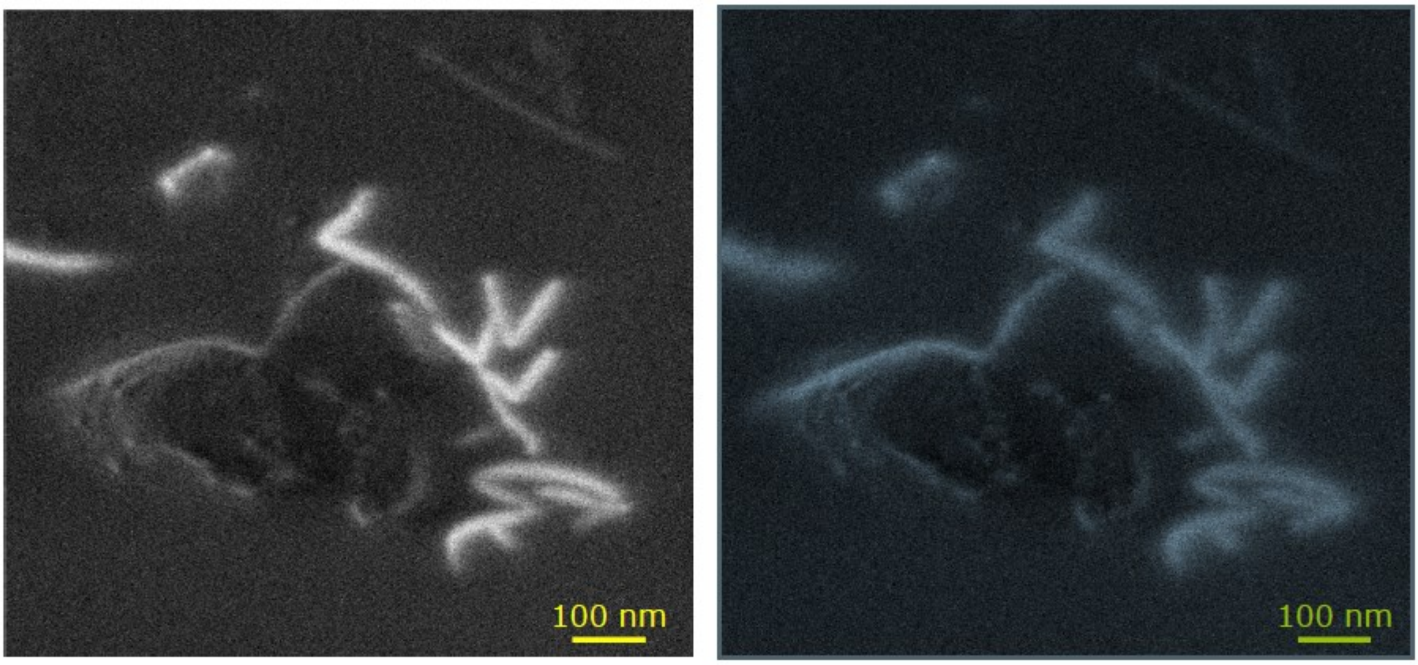


Fig. 2 VC images of CNT containing rubber taken by the SFC function
(b) Integrated image of 1st to 20th frames (c) Integrated image of 109th to 128th frames

VC imaging is effective to differentiate materials with different conductivities. In higher magnification observation, the VC tends to decrease due to surface potential saturation caused by increased electron irradiation per unit area (dosage) in addition to the contamination effect. Thus, optimizing the dosage to avoid the saturation is important for VC imaging. Fig. 2 shows VC images of the CNT containing rubber taken by the Serial Frame Capture (SFC) function. The SFC function saves all elemental frames with each frame selectable to integrate or not. In Fig. 2 (a), the contrast decreases as the number of frame increases. Fig. 2 (b) is an integrated image of 1st to 20th frames which are before the contrast decrease. The CNTs were clearly observed. Fig. 2 (c) is an integrated image of 109th to 128th frames with higher accumulated dosage. The contrast became unclear. As shown here, the SFC function enables to visualize the CNT distribution in rubber clearly by selectively accumulating images with integrated dosage that does not deteriorate the VC.

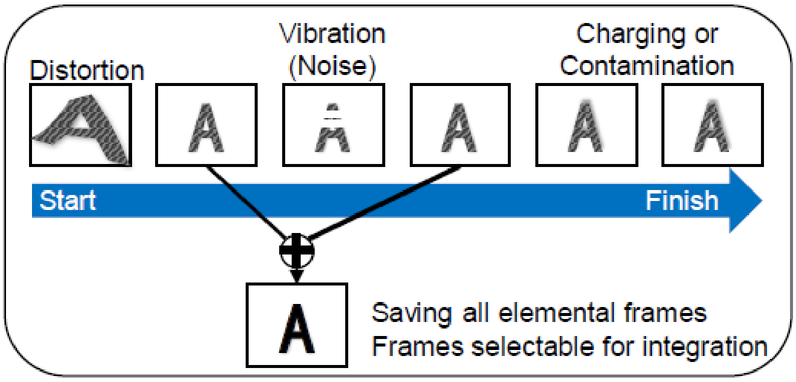
Instrument:	SU7000 FE-SEM
Accelerating voltage:	0.3 kV
Magnification:	100 k×
Detector (signal):	Upper detector (SE)
Specimen:	courtesy of High Impact Project from Malasian Rubber Board, funded under the 11th Malaysian Plan (RMK-11)

SU7000 FE-SEM

The SU7000 offers fast acquisition of multiple signals to address a variety of SEM needs, from imaging a wide field of view to visualizing sub-nanometer structures. The system simultaneously detects and displays up to six different types of signals. Moreover, it features a wide variety of observation techniques since it is optimized for large specimen sizes, variable pressure conditions, croygenic conditions and in-situ analysis.

Recommended configuration:

- SU7000 FE-SEM
- Serial Frame Capture



Schematic illustration of the Serial Frame Capture function

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