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UV Treatment of TEM/STEM Samples for Reduced Hydrocarbon Contamination

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Surface contamination due to hydrocarbons is frequently a limiting factor in TEM, scanning TEM (STEM) or SEM observations. Regardless whether the contamination arises from the specimen preparation such as rinse agents or FIB processing or just due to exposure to hydrocarbons in the atmosphere it is a long standing problem for anyone doing high-resolution imaging or analysis. The use of ultraviolet light (UV) as a treatment for removal has long been established [1,2,3]. In this paper we will expand on the use of a UV-light treatment using the commercially available Hitachi ZONE cleaner used for reducing contamination on sensitive samples for TEM, STEM and FIB and SEM analysis.

The UV treatment was tested using a variety of samples. A commercially obtained graphene sample, amorphous carbon holey film and a FIB prepared Li-polymer composite. These samples were shown to prevent high resolution imaging or analysis due to contamination when exposed to the electron beam in both high voltage TEM (amorphous carbon samples) and high voltage STEM (graphene and Li-polymer samples). The samples were then cleaned using the Hitachi ZONE TEM cleaning system and re-examined to determine the ability to obtain high resolution images or analysis [1].

Graphene sample was imaged in a Hitachi HD2700 SFE dedicated 200 kV STEM. It was found that the contamination prevented the STEM system from obtaining lattice images and that an analytical probe with 100 pA for about 30 s a contamination spot was produced. After 60 s of cleaning in the ZONE cleaner at a pressure of 10 torr the same area was re-examined, the lattice image was easily obtained and placing a analytical spot on the sample for 30 s did not result in noticeable contamination spot. Figure 1 shows the result: a contamination spot is clear in pristine sample but not present in ZONE-cleaned area. Figure 2 shows similar result on FIB-prepared sample of a Li-doped polymer, used in an electrolytic storage cell. The sample was examined in the STEM in darkfield mode. When first imaged with 50 pA probe the sample exhibited a high contamination rate, while after 120 seconds the contamination rate was greatly reduced and after ZONE cleaning 600 seconds was nearly eliminated.

A question arises on the collateral sample damage caused by the ZONE cleaner. Figure 3 shows a dependence of mass loss of a commercially purchased amorphous carbon film on exposure to UV-light in ZONE cleaner. The mass loss was measured using the contrast method described for example in [4]. Extensive (100 min) ZONE cleaning resulted in about 25% mass loss of the original carbon film. Figure 4 shows change in the apparent bright filed TEM contrast of the same sample in pristine form a) and b) exposed to UV light for 90 minutes. The rougher contrast in Fig. 4b) compared to a) suggests that in addition to moderate mass loss some redistribution or non-uniform mass thickness loss may be taking place.

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[5] The work was supported by Hitachi High Technologies Canada, NRC/NINT and IREQ.

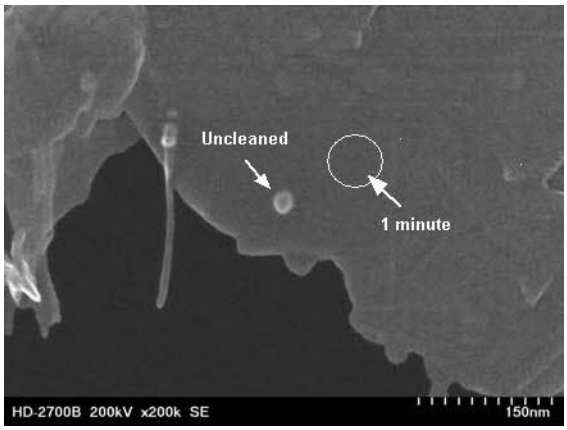

Figure 1. Graphene sample imaged in 200 kV dedicated STEM in secondary electron mode. The contamination build up in uncleaned (pristine) sample area is clearly visible. After 1 minute of ZONE cleaning no observable contamination was seen after the same exposure to electron beam. The exposure was 100 pA for 30 s with stationary probe for each area.

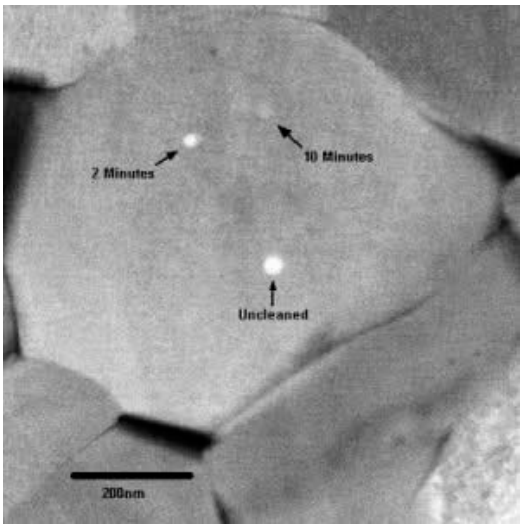

Figure 2. Li-doped polymer sample. The contamination resulting from stationary 50 pA probe positioned on the pristine (uncleaned), 2 min and 10 min ZONE cleaned sample are marked by arrows. Decreased contamination rate is obvious.

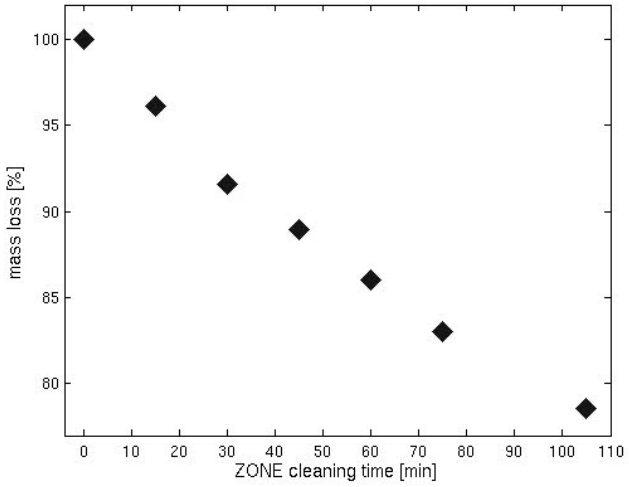

Figure 3. Mass loss resulting as function of exposure time to UV light in ZONE cleaner indicates that for typical cleaning conditions (less than 10 min) the mass loss should be less than about 5%.

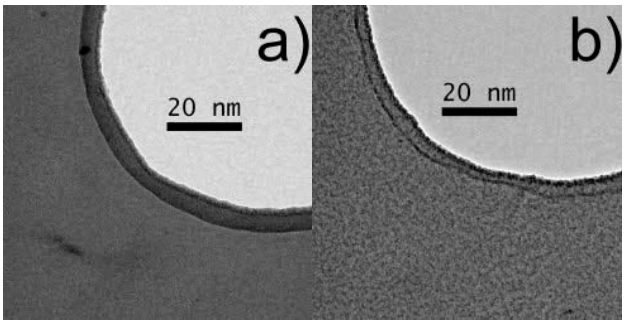

Figure 4 Bright field TEM of amorphous carbon pristine sample a) and b) sample exposed to UV light in ZONE cleaner for 90 min. The more pronounced contrast in b) compared to a) indicates non-uniform sample removal or redistribution of sample material during ZONE cleaning

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