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Investigating the Causes of Electrostatic Charging of Phase-contrast Apertures

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As has been reviewed recently [1], many strategies have been proposed for producing contrast within in-focus images of phase objects in electron microscopy. While promising results have been obtained for several of these alternatives, inconsistent and variable patterns of charging, described operationally as "patch potentials", tend to ruin the long-term performance of most such devices. We find that the problem of patch potentials occurs even though devices are heated at >300 °C during use in order to avoid the buildup of polymerized, carbonaceous contamination. We have previously reported, however, that patch potentials can initially be avoided on thin-film titanium apertures by coating them with evaporated (graphitic) amorphous carbon [2].

Evaporated-carbon coatings nevertheless change their behavior with aging, as do thin carbon-film phase plates, becoming susceptible to charging in the area(s) where they are exposed to the strongest amount of electron irradiation. As a result, a "permanent" patch of electrostatic potential is produced at a point where the unscattered electron beam is intentionally allowed to touch the edge of an aperture, as is illustrated in Figure 1. Although the materials-property changes that occur spontaneously during aging are not yet understood, we began to suspect that they involve a change in susceptibility to knock-on damage by high-energy electrons. More specifically, we hypothesize that changes occur in the contact potential and/or work function of such damaged areas relative to the as-made material. Recognizing that knock-on damage in graphitized carbon specimens appears to be annealed in real-time when irradiated at ~700 °C [3], we investigated whether the development of radiation-induced patch potentials could also be prevented in (graphitic) evaporated carbon films. Encouragingly, as is shown in Figure 1, this appears to be the case.

Heating phase-contrast devices (apertures) significantly above 300 °C can itself introduce new problems, however, especially if the device is fabricated from more than one chemical element. In the case of a tulip aperture fabricated from thin-foil titanium, for example, the surface of the titanium metal is unavoidably coated with ~3 nm of oxide upon exposure to air, and there also is a currently-uncharacterized amount of gallium implanted near to milled edges and possibly contaminating the surface. While problems that initially may be caused by the oxide and the gallium can be "covered up" by coating with evaporated carbon, heating at a very high temperature might increase the mobility of different elements; promote reaction between different elements; and cause separation (delamination) between different layers. Unfortunately, we do see new patch potential effects appear when heating carbon-coated titanium devices heated above ~400 °C, as is shown in Figure 2. Current research is thus proceeding in two different directions: (1) the use of "monolithic" graphitic or (doped) diamond materials (i.e. eliminating the titanium + oxide component) or (2) improving the adhesion of evaporated carbon to the titanium oxide layer.

References:

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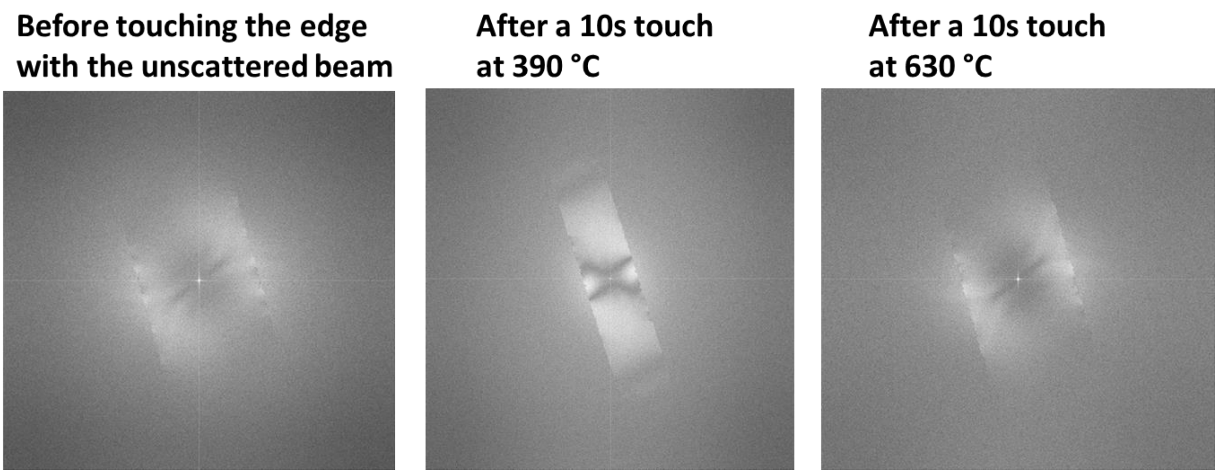

Figure 1. Fourier transforms (FFTs) of images of an amorphous thin-carbon specimen. Departures from the pattern that is expected on the basis of defocus and spherical aberration must be due to the effects of unwanted electrostatic potentials. In this example a straight knife edge, milled into 5 µm-thick amorphous carbon foil (ACF Metals, Tucson, AZ) was used to test susceptibility to charging.

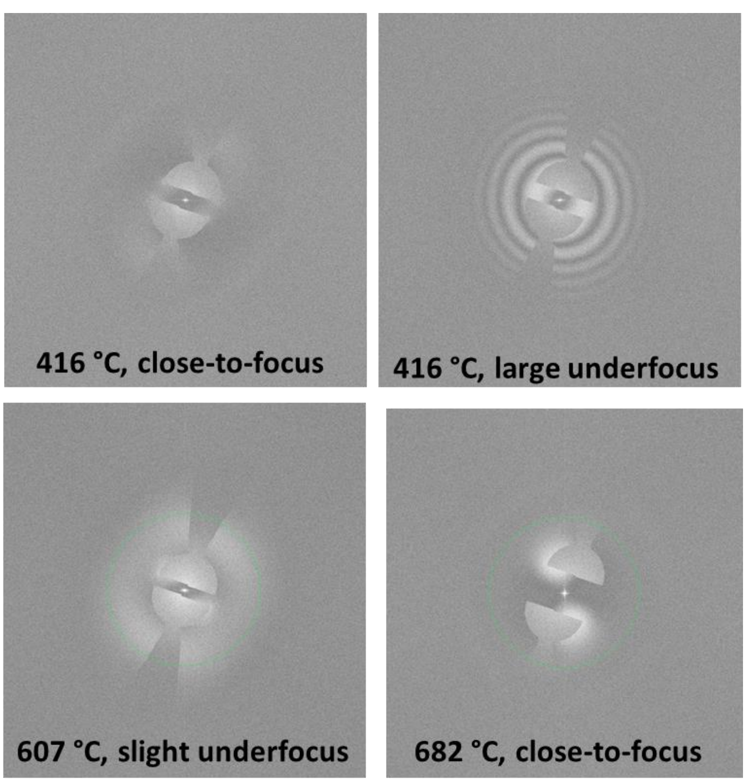

Figure 2. In this experiment, FFTs of images of an amorphous thin-carbon specimen are used to observe the spontaneous, patchy charging of a “tulip” aperture as it is heated to progressively higher temperature. In this case, a focused gallium-ion beam was used to mill the aperture into 5 µm-thick titanium foil, which was then coated with evaporated carbon as conformally as possible.

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