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Metallurgical Sample Surface Cleaning Using UV/Ozone

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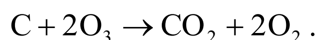
Hydrocarbon surface contamination is one of the major problems in low voltage electron microscopy. Due to the interaction volume phenomena, a sample that appears to be clean when viewing at high kV (i.e: 20kV) will often suffer from a contamination problem once it is viewed at an acceleration voltage of 5kV and below. The general use of UV/Ozone to remove residual hydrocarbon from sample surfaces has long been presented and published elsewhere by Vig et al. and Mcintyre et al [1,2]. In fact, UV radiation was also used to remove organic contaminants from the surface of Si during wafer fabrication [3]. The objectives of this paper is to first provide a literature review on this cleaning mechanism then followed by the improvements that can be made when this cleaning technique is introduced into the field of electron microscopy.

The use short wavelength radiation to decompose hydrocarbons has been known since the early 1970s. Extreme UV (λ <254nm) is the common source of radiation that is used during this cleaning process. This cleaning mechanism is the result of photochemical oxidation process in which short wavelength radiation is being bombarded to the surface and the contaminants (i.e: chained hydrocarbons) are activated and dissociated by breaking down these polymer chains. In parallel, atomic oxygen (O) is generated through the process of photon reaction of oxygen gas (O_2) and atomic oxygen will further react with oxygen gas ultimately generating ozone (O_3). The ozone produced here will then react with the excited molecules forming volatile gaseous compounds and ultimately gets removed from the sample surface. A schematic illustrating this cleaning mechanism is shown in figure 1. As an example, excited molecules such as carbon (typical element found in hydrocarbon) is reacted and removed according to the below chemical reaction:



$$C + 2O_3 \rightarrow CO_2 + 2O_2 .$$

To validate this cleaning technique, a test pilot device was made which consist of a low pressure mercury lamp mounted inside of a chamber unit that is made out of aluminum. A low pressure mercury lamp is selected here because of its ability in generating dual wavelengths at respectively 185 and 254nm. For this experiment, high resolution gold nanoparticles on tin particles were made by sputtering using a high vacuum evaporator. To induce hydrocarbon contamination, samples were left in vacuum (~10Pa) chamber that was pumped down using an oil rotary pump. At its best vacuum level (~10Pa), rotary pump oil will backstream causing hydrocarbon pump oil vapor to condense and deposit on top of the sample surface. All SEM micrographs showed here were taken using the Hitachi SU8000 CFE-SEM at a landing voltage of 300V, at a probe current of 6 pA. Figure 2a is the SEM micrographs showing the Au-Sn resolution sample after being contaminated by the rotary pump oil. Here, it can be seen that the gold nanoparticles were covered by a layer of hydrocarbon resulting a blurry obscured image. It's also interesting to point out that during imaging, additional hydrocarbons were accumulated over the SEM scanning area. This electron beam induced carbon contamination is also described elsewhere and it's the result of deposition of hydrocarbon from the sample surrounding ultimately causing a dark rectangle to appear when going from high to low magnification of SE imaging [4,5]. Figure 2b is the SEM micrograph

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illustrating e-beam induced contamination during one minute of scanning at 200kx magnification (probe current 6 pA at 1e^{-6} torr vacuum). Figure 2c shows the same sample that is cleaned after being exposed to 25 minutes of UV/ozone. Here, the resolution of the image is much improved and now the morphology of tin particles (bigger feature) and the gold nanoparticles (on top of tin) can be easily resolved. The treated sample also has minimal e-beam induced carbon contamination resulting in a much better environment for SEM operator such as performing all necessary beam and stigma alignments.

Using this pilot cleaning device, we found UV/Ozone is very effective in removing contaminants on variety of surfaces (i.e: Silicon, Copper, Nickel, Gold, Iron, Mo, CNT). UV/ozone is a very gentle cleaning technique when compared to plasma because the photon energy of UV is very low (<180 kcal/mol) such that it can only cause excitation/breaking of polymer chains. Lastly, it should be noted that the greatest cleanliness can only be achieved when gross contamination was first removed by ultrasonically clean in ethanol, followed by pure water rinse then thoroughly drying the sample prior to UV/Ozone cleaning treatment.

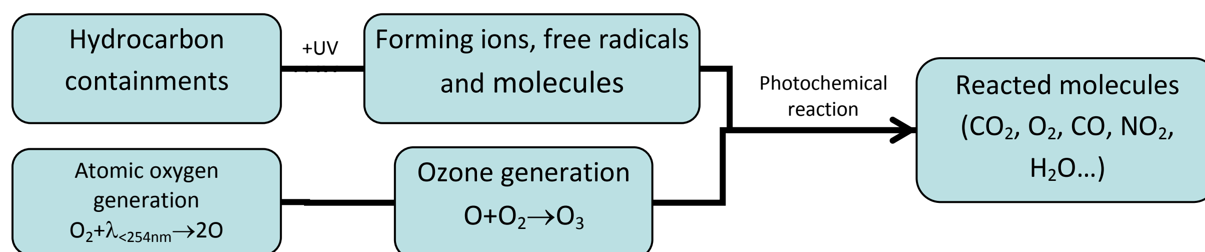

FIG 1. Schematic representation of UV/Ozone cleaning

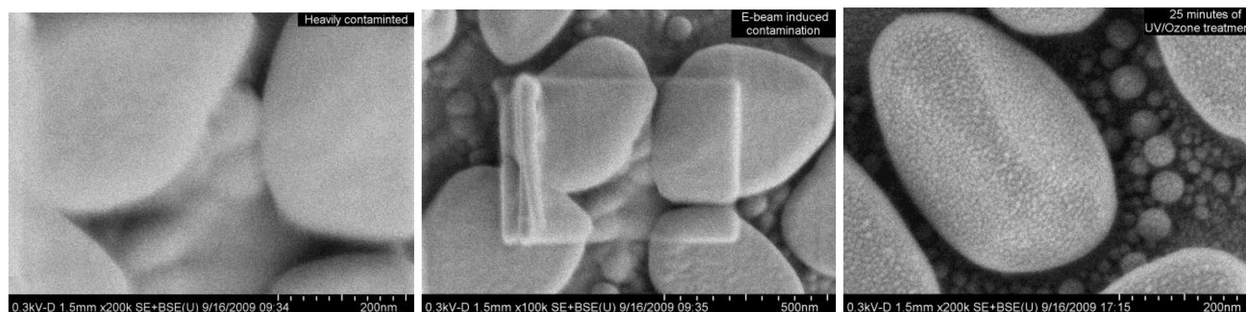

FIG 2. SEM micrographs showing: a) sample after contamination, b) e-beam induced sample contamination, and c) the same sample cleaned using the In-house pilot UV/Ozone cleaning device

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