ANL/MSD/CP--92330 CONF-970834--26

REACTIVE GAS PLASMA SPECIMEN PROCESSING FOR USE IN MICROANALYSIS AND IMAGING IN ANALYTICAL ELECTRON MICROSCOPY*

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January 1997

Submitted to the Microscopy & Microanalysis, Cleveland, Ohio, August 10-14, 1997.

*Work supported in part by the U. S. Department of Energy, BES-Materials Sciences, under Contract W-31-109-ENG-38 and the USNRC under FIN W6610.

DISCLAIMER

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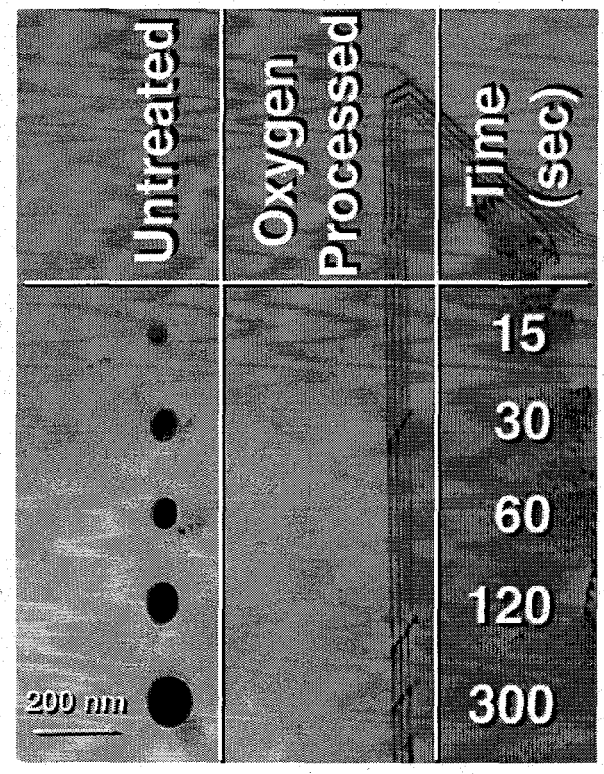
It has long been the bane of analytical electron microscopy (AEM) that the use of focused probes during microanalysis of specimens increases the local rate of hydrocarbon contamination. This is most succinctly observed by the formation of contamination deposits (fig.1) during focused probe work typical of AEM studies. While serving to indicate the location of the electron probe, the contamination obliterates the area of the specimen being analyzed and adversely affects all quantitative microanalysis methodologies. A variety of methods including: UV, electron beam flooding, heating and/or cooling can decrease the rate of contamination, however, none of these methods directly attack the source of specimen borne contamination[1]. Research has shown that reactive gas plasmas[2] may be used to clean both the specimen and stage for AEM, in this study we report on quantitative measurements of the reduction in contamination rates in an AEM as a function of operating conditions and plasma gases.

All the experimental measurements described herein, were made on a Philips CM30 AEM, operating at 300 kV and equipped with a EDAX PowerMx XEDS and a GATAN Model 666 PEELS. No extraordinary precautions were taken to minimize contamination during these experiments, the nominal column vacuum during the work was $< 1x10^{-7}$ Torr and the LN_2 cold finger was kept full at all times. Specimens were mounted in a standard RT, double tilt, Be stage and all measurements were made under constant electron optical conditions, using a nominal 20 nm LaB_6 probe having a current of 0.7 nA. Reactive gas plasma treatment of the specimen and stage to mitigate contamination effects was accomplished using a South Bay Technology (SBT) Model PC-150 system. The gases employed were nominally pure Ar and O_2, the selection and mixing of which was facilitated by the multiport gas inlet system of the PC-150. During all specimen plasma processing the gas pressure in the reaction chamber was ~200 mT, RF power a constant at 10 W, and a processing time of 5 minutes was used. The 304 SS test specimens were prepared by electropolishing using 11.6 g $Mg(ClO_4)_2$, 500 ml Methanol, 50 ml Butyl Cellosolve at -30 C at 200 V in a SBT Model 550 D vertical jet polisher. Under conventional TEM conditions these specimens contaminate slowly, however, during focused probe work the contamination rate is high. For the case of virgin specimens, after ~ 60 s the contamination deposit formed completely obscures the specimen (fig.1). To quantitatively measure the rate of contamination, electron energy loss spectroscopy was used to monitor the mass thickness of material under the probe by determining the change in the value of $\mathbf{t/\lambda = \ln (I/I_0)}$ as a function of time (fig.2,3). Figure 3 plots $\mathbf{t/\lambda}$ vs **T**, for a typical sample under unprocessed, Argon and Oxygen treated conditions. The contamination rate is $\mathbf{d(t/\lambda) / dT}$ and we obtained values of: $4x10^{-2}$, $9x10^{-4}$, $6x10^{-5}$ sec^{-1} for the three conditions reported here. We see that Argon processing reduces contamination by ~1/40th but does not eliminate it. Oxygen processing, enormously reduces the rate to ~1/600th, and repeated focused probe measurements of the same location of the specimen for 10 minute periods show no visible contamination (fig. 1,3). In addition, no surface oxide formation was detected by EELS from the Oxygen plasma. After 15+ hours (overnight) in the AEM, a small amount of hydrocarbon contamination returned which may be due to the microscope environment. This was curtailed by a second 5 minute processing with Oxygen. Work is in progress to identify the mechanism for this return and also to establish the processing parameters for other materials. We also note that once formed, contamination deposits are not removed by any plasma processing action .

References:

1.) J. J. Hren, *Introduction to Analytical Electron Microscopy*, Plenum Press, (1979), Chptr. 18
2.) *Simultaneous Specimen and Stage Cleaning Device for Analytical Electron Microscopy* US Patent # 5,510,624 - Argonne National Laboratory and the University of Chicago
3.) Supported by US. DoE under BES-MS W-31-109-Eng-38 and the USNRC under FIN W6610.



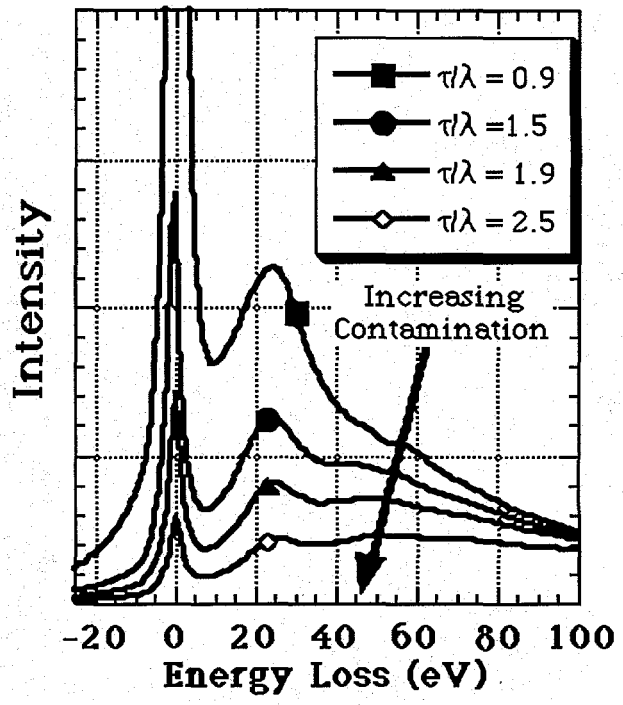


Figure 1: Micrograph, showing contamination formed after 15, 30, 60, 120 and 300 sec, by a 20 nm 0.7 nA 300 kV probe before processing and their absence after O_2 plasma processing.
Figure 2: EEL Spectra at selected times during focused probe mode analysis (untreated specimen) illustrating build up of contamination and it's effect on the EELS low loss data.

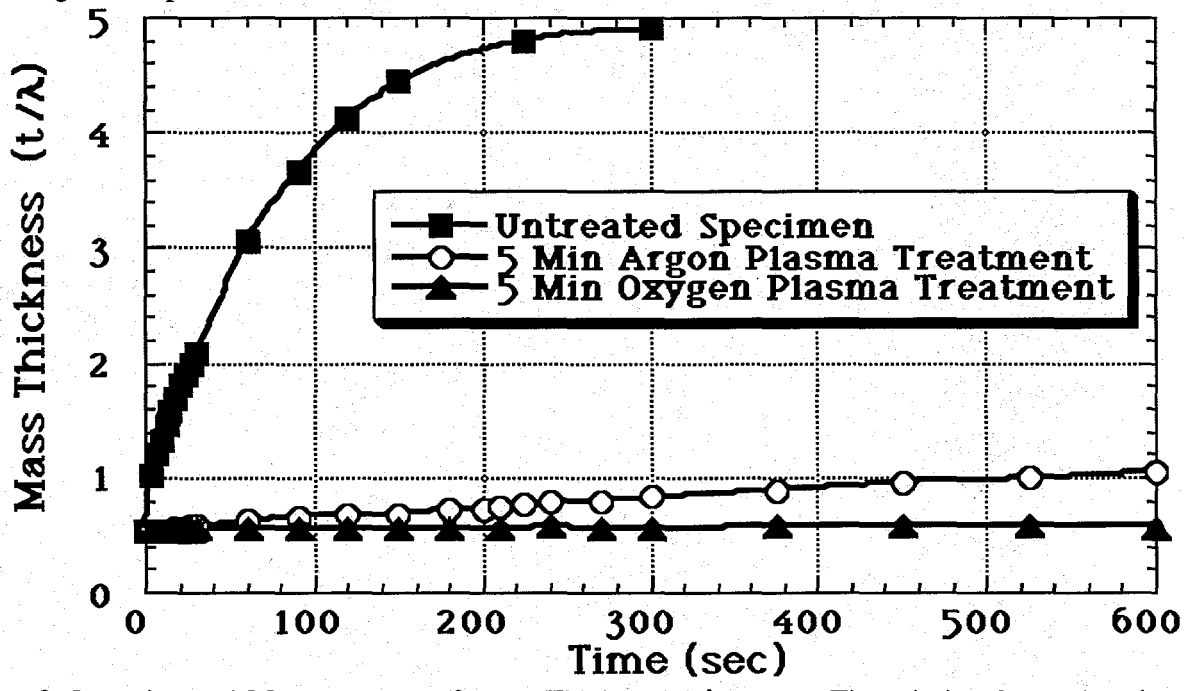


Figure 3: Experimental Measurements of Mass Thickness (t/λ) versus Time during focused probe mode analysis for untreated (■), Ar (○) and O_2 (▲) processed specimens, the contamination rate is given by $\mathbf{d(t/\lambda) / dT}$ and is $4x10^{-2}$, $9x10^{-4}$, $6x10^{-5}$ sec^{-1} respectively. This data illustrates that Ar plasma processing reduces the contamination rate by ~ 40x, while O_2 processing reduces the rate by ~ 600x.