

Copper Contamination of Gunshot Residue and Other Particle Spectra

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Many scanning electron microscope (SEM) laboratories use double sticky carbon tape applied directly to SEM stubs as samplers for small particles. The SEM samplers used by law enforcement to test the hands of a suspect for gunshot residue (GSR) are constructed in this manner.

A few years ago, during a presentation at a professional meeting, I presented spectra of particles that were mounted on carbon double sticky tape that was applied directly to the aluminum-alloy SEM stubs. After the presentation a colleague posited that small peaks of copper, which appeared in some of my spectra, were due to a copper support frame for the backscatter detector at the top of the SEM chamber and not the particles. Indeed, I have frequently noticed small peaks of copper in spectra of particles where these should not be present. This suggests that exposed copper somewhere in the SEM chamber is being irradiated by either electrons or X-rays or both. According to my colleague, a heavy coat of carbon paint on the offending copper would fix these pesky, spurious X-rays. Although this may be a source of contaminate copper X-rays in spectra, it is not the only possible source.

I have seen no publication concerning the issue of copper contamination of particle spectra. So, for persons new to the field and perhaps even some old timers, the following may be enlightening.

Copper is a component of the SEM stubs from most if not all

manufacturers (Figure 1A). It is from the SEM stub that problem copper X-rays originate. At 20 KV many K series X-rays from the copper in the SEM stub are not completely absorbed by the carbon double sticky tape applied directly to the SEM stub. However, the low-energy L series X-rays of copper are absorbed by the tape (Figure 1B, bottom spectrum).

The majority of the K series copper X-rays are eliminated from spectra by placing a 1.8 mm thick graphite disk between the carbon tape and the SEM stub. But, some copper K-series X-rays still get through (Figure 1B, middle spectrum). Indeed, at 30 KV acceleration voltage with the sample on a 1.8 mm thick graphite disk, the spurious copper peak is frequently present in a particle's spectrum. By sandwiching a 3.2 mm thick graphite disk (obtained from Ladd Research Industries, Burlington, VT) between the carbon double sticky and the SEM stub, the problem copper X-rays are eliminated from spectra generated from both 20 and 30 KV acceleration voltages (Figure 1B, top spectrum).

Fine lead particles from a fishing sinker were filed onto SEM stub preparations. One stub had just the double sticky tape on the surface of the stub and the other stub had a 1.8 mm thick graphite disk between the stub surface and the carbon tape. Fifty lead particles from 1 to 2 micrometers were analyzed from each stub. Copper K-series X-rays were contaminating the spectra of most of the particles that were on the tape sample that lacked the underlying graphite disk (Figure 1C, lower box plot). The intensity of contamination of the copper X-rays ranged from 0 to 14 counts per second at 20 KV acceleration voltage. Clearly, in a GSR analysis there could be difficulty in distinguishing particles that actually contain a small amount of copper from those that do not. The 1.8-mm thick graphite disk sufficiently blocks copper X-rays at an ac-



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celeration voltage of 20 KV from contaminating the lead X-ray spectra (Figure 1C, top box plot). Therefore, for 20 KV GSR analyses on the 1.8 mm thick disks, even though a direct rastering of the carbon-tape surface shows a small amount of copper contamination (Figure 1B, middle spectrum), the copper X-ray contamination is not enough to have a meaningful effect for most particle analyses conducted at this acceleration voltage.

Why is this important? In .22 and other caliber shootings, the presence of copper in GSR distinguishes a copper-coated or jacketed bullet from GSR originating from a bullet that lacks a metal coating¹. Contaminate copper X-rays in a spectrum would confuse this determination.

Conversations with colleagues involved in the analysis of gunshot residue or other particle types by SEM/EDS revealed different responses to the problem, ranging from acknowledgment to surprise. To my knowledge, none of the manufacturers of gunshot residue collection kits for law enforcement provide a remedy either by supplying their samplers with graphite disks between the carbon double sticky tape and the stub, or by using stubs that do not contain copper. ■

1. Burnett, B.R. 1998. The form and composition of .22 caliber gunshot residue from muzzle and breech deposits. *Proceeding of the American Academy of Forensic Sciences*. 4:26.

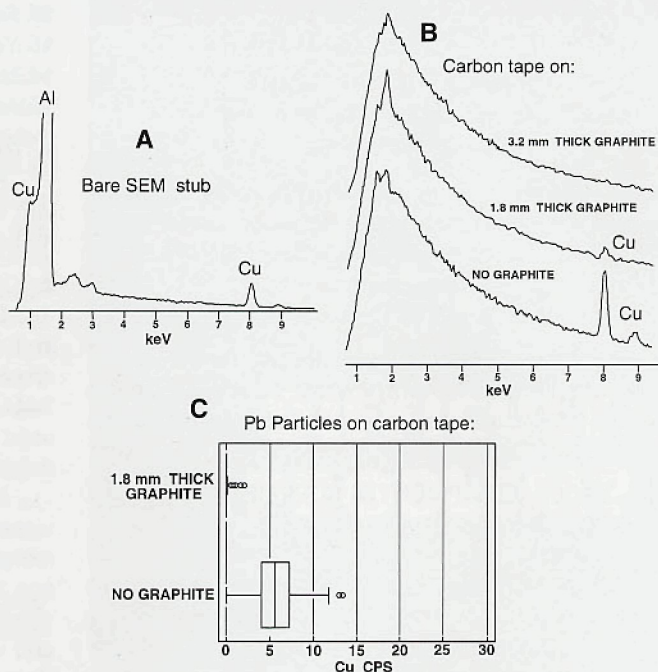


Figure 1. A: Energy dispersive X-ray spectrum of the surface of a typical aluminum/copper alloy from a SEM stub. 20 KV acceleration voltage, 1 nanoamp specimen current, vertical scale = 2000 counts. B: A series of spectra made from rastering the electron beam on the various configurations of the SEM preparations. 20 KV acceleration voltage, 1 nanoamp specimen current, vertical scale = 1000 counts. Bottom spectrum: carbon tape directly on the SEM stub surface. Middle spectrum: carbon tape on a 1.8 mm thick graphite disk on the SEM stub. Top spectrum: carbon tape on a 3.2 mm thick graphite disk on the SEM stub. C: Box plots of the copper counts from 50 X-ray samples of lead particles from a surface of carbon tape/1.8 mm thick graphite disk/SEM stub (top) and the same number from a surface of carbon tape applied directly to the SEM stub surface (bottom). 20 KV, 1 nanoamp specimen current.



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