TECHNICAL NOTE

Lorenzo Varetto,[1] *M.D.*

The Use of Plasma Ashing on Samples for Detection of Gunshot Residues with Scanning Electron Microscopy and Energy-Dispersive X-Ray Analysis (SEM/EDXA)

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ABSTRACT: A method is described for the removal of organic material from the adhesive tape employed for collecting particles on the hands. It utilizes the coupled action of contamination by the electron beam of the scanning electron microscope (SEM) and oxygen plasma ashing. The cells of the epidermis are destroyed and only thin filaments are left, while particles that were previously concealed become evident. The treatment does not alter the morphology or composition of gunshot residue (GSR) or of inorganic environmental particles.

KEYWORDS: criminalistics, gunshot residues, plasma ashing, microscopy

Adhesive tape is widely employed to collect samples from hand surfaces for identification of gunshot residue (GSR) by means of the scanning electron microscope (SEM) and energy-dispersive X-ray analysis (EDXA) [*1,2*]. Currently, double-sided adhesive tape is being used which has been previously affixed to the SEM stub. This procedure is fast and simple, but cells from the subject's epidermis often stick to the tape, sometimes in a nearly continuous layer. In such cases, most of the GSR particles are obviously hidden between the cells and the adhesive tape and therefore cannot be detected with SEM.

Some methods have been developed to remove the cells [*3,4*], but all of them can be employed only with other collection systems (such as the use of cotton swabs).

In this paper, a system is described for removing organic debris from the adhesive tape and for revealing the hidden GSR particles.

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[1]Technician, Istituto di Scienze Medico-Forensi, Università di Torino, Torino, Italy.

Method

The stub with the adhesive tape employed for collection of particles from the hands of a suspected shooter is carbon coated, as usual, and put into the chamber of the SEM. A first observation is performed (if any typical particle is recognized at this time, further searching may be unnecessary). Then, the electron beam is allowed to fall on and scan across a single field of observation for 5 min, without the stub being moved. The size of the spot is the same as that normally employed for backscattering and microanalysis. The magnification can be chosen by the observer; ×50 is suitable for the purpose.

Small variations in the spot size, the magnification, or the time do not change the results.

The operation may be repeated on other parts of the stub's surface. The author's personal experience in using the proposed method with casework samples that are poor in particles suggests that four or five fields (magnification ×50) are usually adequate for a positive result.

The stub is then taken out of the SEM and treated with the plasma asher for several hours (10 to 15 h). Finally, the stub is carbon coated and the final observation of the specimen can be performed.

The instrumentation used for this work includes a Cambridge Stereoscan 250 SEM with an X-ray analyzer EDXA Link and a Polaron E 2000 plasma asher.

The characteristics of the oxygen plasma asher have already been described for its application to GSR analysis with flameless atomic absorption spectroscopy [5].

Results

On the surface of the stub treated by the described method, the final observation shows rectangular areas, which are most evident at low magnification (Fig. 1). These are the

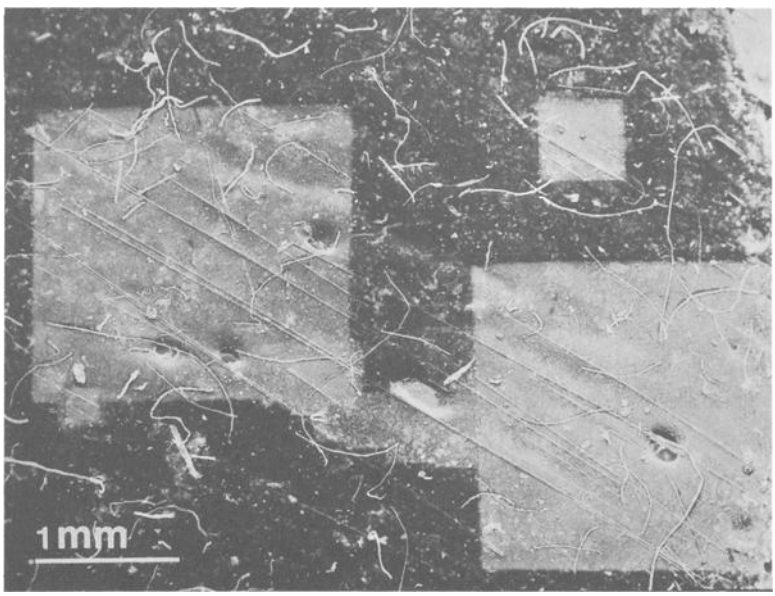

FIG. 1—*The surface of the specimen at final observation. The well-defined rectangular areas correspond to the fields of contamination revealed by the oxygen plasma asher.*

areas where the electron beam has fallen for a long time during the first observation. Their dimensions depend on the original magnification.

A higher magnification shows, outside the rectangular areas, the inner surface of the cells of the epidermis, with their typical reliefs. In the rectangular fields, in contrast, there are rare and thin filaments that form a loose net in which the particles become evident (Figs. 2 and 3).

In addition, neither the morphology nor the composition of the particles changes, according to observations made before and after the treatment of the sample (Figs. 4 through 6).

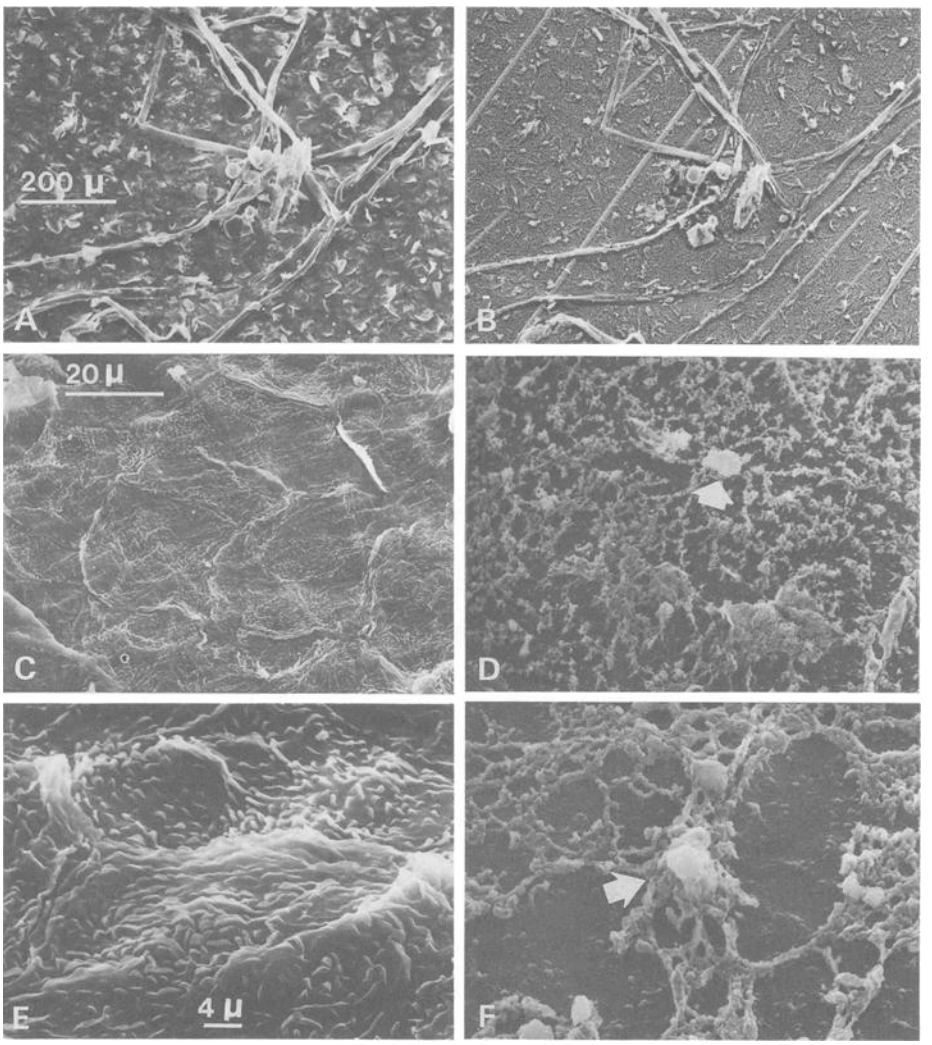

FIG. 2—*A comparison between the surface of the specimen before* (a, c, e) *and after* (b, d, f) *the treatment, at different magnifications. In* (a) *the cells of the epidermis make an evident continuous layer; their surfaces* (c, e) *show typical reliefs. In* (b), (d), *and* (f) *only rare filamentous structures can be observed, and among them the particles can be recognized* (arrows).

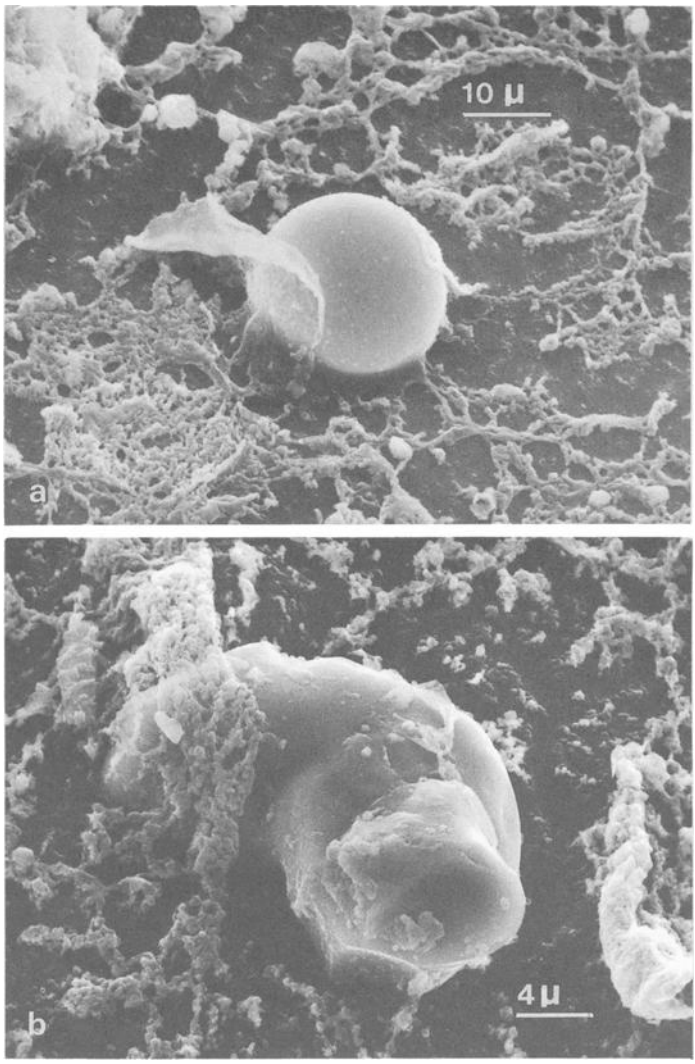

FIG. 3—*Particles that are still partially concealed by cellular debris*: (a) *GSR (Pb, Ba, Sb);* (b) *an environmental particle (Fe).*

Discussion

The method described utilizes the degradation caused by the electron beam (thermal cracking of organic bindings), combined with the action of the low-temperature oxygen plasma.

Actually, the conditioning induced by the beam alone does not produce the coarse damage evident in SEM observation because it does not involve the carbon coating, which remains unaltered; in addition, plasma ashing, which has already been suggested for the same purpose with good results [*6*], does not, by itself, produce a satisfactory destruction of the cells, according to this study.

In contrast, in the fields conditioned by the beam, the plasma ashing reduces the cells

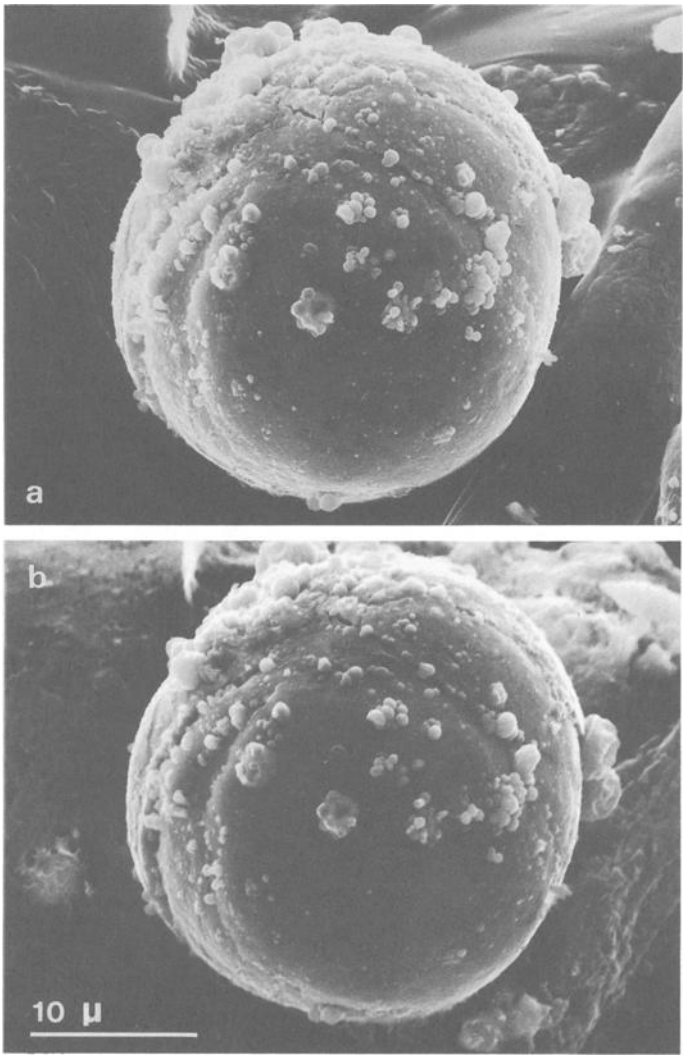

FIG. 4—*The same GSR before* (a) *and after* (b) *the treatment. The morphology is identical.*

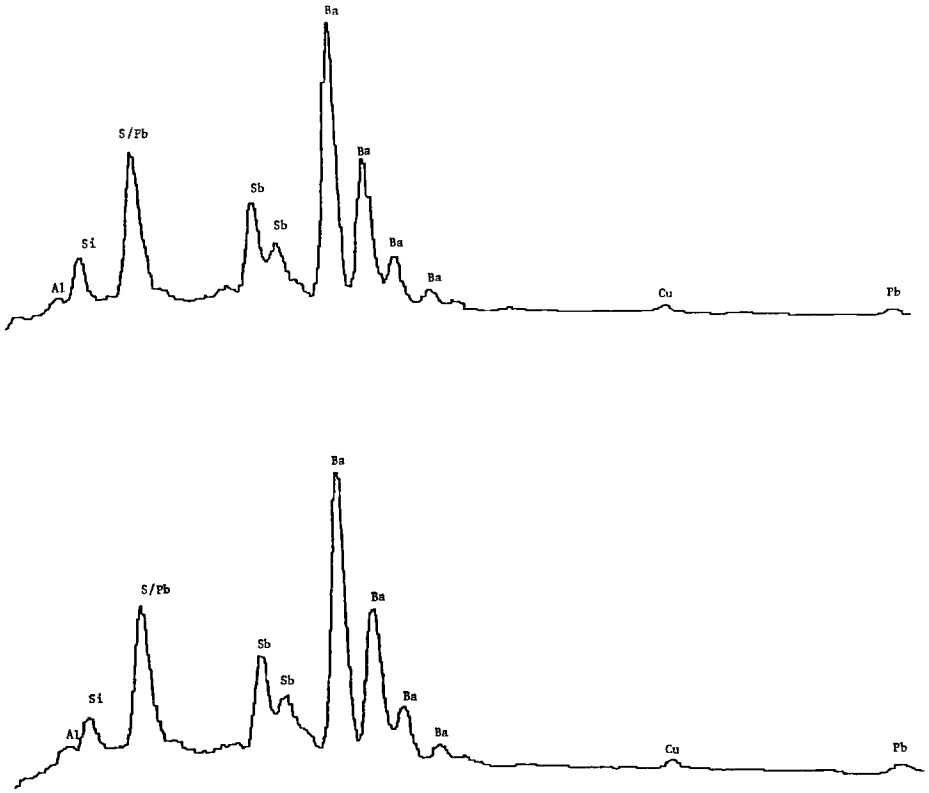

FIG. 5—*The composition of the particle in Fig. 4 before* (top) *and after* (bottom) *the treatment.*

to thin structures, which allow easy detection of inorganic particles that were previously concealed.

It is interesting to note that neither the morphology nor the composition of these particles (both environmental and GSR) changes after the described treatment, and therefore there is no added risk of mistakes in classification.

The particles of partially burned powder are obviously destroyed, but they could not have been confirmed by SEM searching anyway. These particles could have been examined and collected by using a stereomicroscope prior to SEM examination.

Conclusions

In conclusion, it seems that the suggested method could be usefully employed for searching for GSR on adhesive tape when the sample is poor in particles, because even those particles previously concealed by organic structures thus become visible.

Besides, after the described treatment, the searching is performed over limited quadrangular areas; this allows one to orient the stub in the same way during successive observations and to recognize, even after a long time, the interesting particles already identified, if a map of the sample has been drawn.

Finally, this method is also useful with automated GSR detection and analysis [*7,8*].

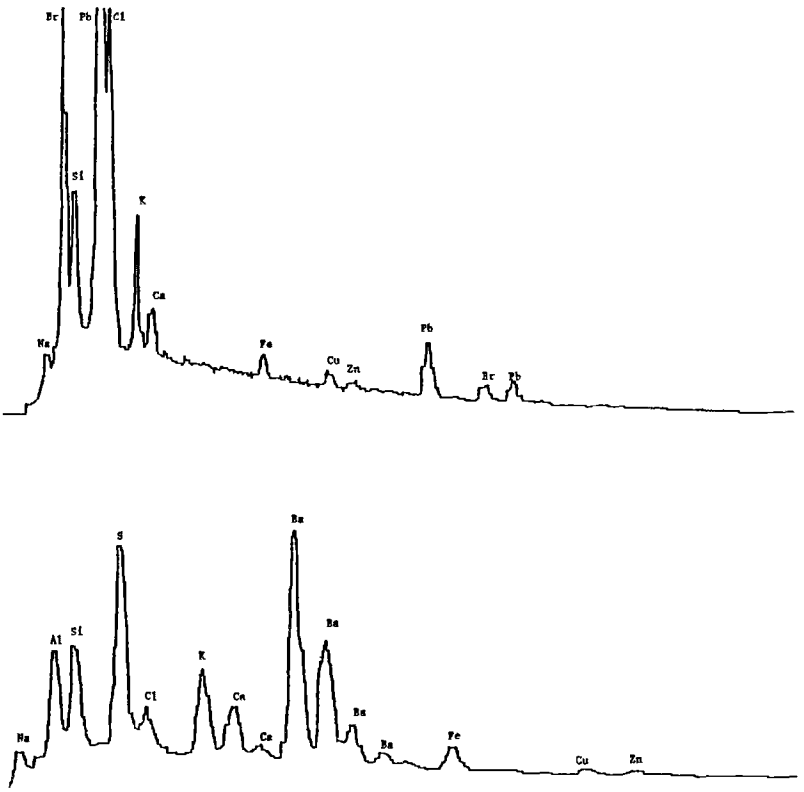

FIG. 6—*Composition of two environmental particles after the treatment. The sulphur and the bromine, which allow a precise classification, are preserved.*

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Address requests for reprints or additional information to
Lorenzo Varetto, M.D.
Istituto di Scienze Medico-Forensi
Corso Galileo Galilei 22
10126 Torino
Italy