TECHNICAL NOTE

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Comparison of Properties of Adhesive Tapes, Tabs, and Liquids Used for the Collection of Gunshot Residue and Other Trace Materials for SEM Analysis

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ABSTRACT: The use of adhesive tapes, tabs and liquids is a simple and cost-effective method for sampling gunshot residues and other trace or particulate materials prior to analysis in a scanning electron microscope (SEM). The adhesive material is placed directly onto a standard stub (specimen mount) used in scanning electron microscopy. The use of these prepared stubs can decrease sample preparation and collection time as well as reducing the risk of sample loss.

In this study, a number of adhesives are examined for their suitability in the areas of elemental composition, adhesive properties, and adhesive stability under vacuum and an electron beam.

KEYWORDS: forensic science, criminalistics, adhesive tapes, adhesive lifts, gunshot residue, collection, scanning electron microscopy

Adhesive tapes, tabs, and liquids are used generally as mounting media in scanning electron microscopy (1) and specifically, for the collection of gunshot residue (2,3) and other trace materials for analysis in a scanning electron microscope (SEM). The tapes, tabs, or liquids are placed onto 'stubs' (specimen mounts used in the SEM) which then act as a support for the adhesive. Ideally, double sided adhesives are used: one side adhering to the stub, the other, to the sample. The prepared stubs are then simply and systematically pressed against the area to be sampled. The advantage of collecting samples using these prepared stubs is that, in most cases, the sample is placed directly onto the specimen mount, therefore eliminating time-consuming preparations such as filtration and concentration techniques which also result in sample losses.

Fifteen assorted adhesive media were examined. They included double-sided tapes, adhesive tabs, liquid adhesives, a glue stick and 'carbon conductive cement.' The suitability of the adhesive was determined according to: (i) the elemental composition of the adhesive, (ii) the adhesive properties of the material, and (iii) the stability of the adhesive under vacuum and an electron beam (see Table 1).

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The Elemental Composition of the Adhesive

A common method used for particle analysis in general, and Gunshot Residue (GSR) analysis in particular, is the Scanning Electron Microscope (SEM) fitted with an energy dispersive X-ray analyzer (EDX) using a beryllium window. Elements contained in the composition of the adhesive media may be detected by an SEM with this configuration, resulting in a 'background.' This 'background' may interfere with automatic analysis systems, possibly resulting in false positives and/or substantially increasing analysis time. Therefore any adhesive used should have a zero or minimal background.

The Adhesive Properties of the Material

With the 'tape lift' method (2), whatever adhesive medium is used, the number of particles collected is directly related to the 'stickiness' of the adhesive. The adhesive must remain sticky even after being repeatedly applied to a variety of surfaces. For example, when sampling for GSR, large areas of skin which on occasions can be moist, must be systematically sampled with continued applications. Also, when in situations requiring the removal of samples from clothing, the fabric may contain debris or loose fibers which also cover the surface of the adhesive. The ideal adhesive media will retain adhesiveness in the presence of moisture, is not so aggressive as to forcefully pull fibers from garments during sampling and can be used for repeated applications to a site being sampled.

In comparing the adhesive properties of the various media, each adhesive was assigned a subjective rating.

The Stability of the Adhesive under Vacuum and an Electron Beam

Any mounting media used in the SEM must withstand a vacuum greater than 1.4×10^{-2} Pa (10^{-4} torr) both in the carbon coating

TABLE 1—*Elemental composition, adhesive factor, and the adhesive stability under a vacuum and an electron beam irradiation are listed for fifteen adhesives commonly used in electron microscopy.*

Brand & Manufacturer	Elemental Composition	Adhesive Factor	Stability Under Vacuum & an Electron Beam
Double Side Tape #G263 Agar Scientific	HL: Al, Si, Ca S LL: K	Strong	Surface Cracking
Adhesive Carbon Tape #G3939 Agar Scientific	None	Weak	No effect
HDDS Tape #G3914 Agar Scientific	LL: Si, Cl	Very Strong	Surface Cracking
Carbon Tabs #G3347 Agar Scientific	HL: S LL: Si, P, Na	Weak	Surface Cracking
Sticky Tabs #G304 Agar Scientific	HL: Cl LL: Si	Weak	Blistering
Leit C Plast #G3302 Agar Scientific	None	Weak	No effect
Double Sided Tape A1258 Bio-Rad	LL: Al, S	Strong	No effect
Sticky Discs A1070 Bio-Rad	LL: Si, Cl	Weak	Surface Cracking
Glue Stick #05495 Marbig	LL: Na	Very Weak	Blistering
Double Sided Tape #104 Sellotape	LL: S, Si	Very Strong	Surface Cracking
Double Sided Tape #404 Sellotape	LL: S, Si	Very Strong	Surface Cracking
Heavy Duty Mounting #110 Scotch	LL: Si	Very Strong	No effect
Double Stick #136 Scotch	HL: S LL: Si, K	Strong	Surface Cracking
Microstick #1214 Polaron Equipment	HL: Cl	Very Weak	Blistering
Microhesive #A10 Probing & Structure	HL: Cl	Very Weak	No effect

Elemental composition was placed into two levels:
HL: High levels, greater than twice the background.
LL: Low levels, less than twice the background.
Adhesive were categoried as: very strong, strong, weak and very weak.
The affects of the vacuum and electron beam is described as surface cracking, blistering or no effect.

process and within the SEM (unless an environmental chamber is used). The adhesive must be relatively chemically stable if surface cracking, particularly after coating, is to be prevented. If cracking does occur 'charging' of the surface results. The 'out-gassing' of organic materials in the adhesive can also result in deposits on inner surfaces of the chamber, including detector windows, thus reducing detector sensitivity. In addition, the adhesive must also withstand the heat generated by the electron beam at 25 kV.

Materials and Methods

Eight double-sided adhesive tapes, three adhesive tabs, two adhesive liquids, a glue stick, and 'carbon conductive cement' were analyzed for elemental 'background' composition. The adhesive materials were placed onto a standard $^1/_2$ in. diameter, aluminum SEM stub with an $^1/_8$ in. pin. The stub was a standard SEM specimen mount.

For each adhesive, a bulk analysis of an area approximately 3 by 2 mm was analyzed in a 'CamScan Maxim 2000' Scanning Electron Microscope (SEM) fitted with an 'Editor' Energy Dispersive X-Ray Analyzer (EDX). The EDX detector was fitted with an EDAX 'Sapphire' beryllium window.

SEM/EDX analysis is a standard technique used for GSR analysis. It provides an accurate measure of elemental composition of concentrations greater than one percent by weight, for elements with an atomic weight between that of fluorine and uranium. A working distance of 35 mm, an accelerating voltage of 25 kV, a count rate of 300 s and an X-Ray detector take-off angle of 40°, were used in all analysis.

To compare the adhesive properties of the various adhesives, samples were placed onto the stubs, covering the top surface. This allowed samples with the same surface area to be compared. In turn, each stub was systematically compared to another by placing a piece of 2-mm thick cardboard between two opposing stubs, pressed tightly together. They were then pulled apart. This was repeated until each adhesive was rated in one of four categories, namely very strong, strong, weak or very weak.

To determine the stability of the adhesive under vacuum and an electron beam, the adhesive coated stubs were carbon coated and viewed under a 25 kV electron beam concentrated on an area of 20×30 μm for 5 min. Observations were made of the amount of distortion, charging and damage produced. Digital images were recorded of the damage for comparison purposes. The damage was described as "surface cracking" (see Fig. 1a), "blistering" (see Fig. 1b) or "no effect" (meaning no visual effects).

Discussion

The evaluation of the adhesives was conducted to find the most suitable adhesive for the collection of GSR and other trace materials, for example glass, fibers, pollen etc. prior to SEM/EDX analysis. Table 1 is a summary of the results.

The elemental composition of the adhesive is important if it is not to interfere with the analysis of the samples. This is an important factor in the characterization of ammunition as the presence of elements such as calcium, silicon, aluminum, sulfur etc. can also occur in the adhesives.

A major problem with automatic GSR searching programs is that a high sulfur background can lead to false positive results and increase the search time dramatically. This is due to interference

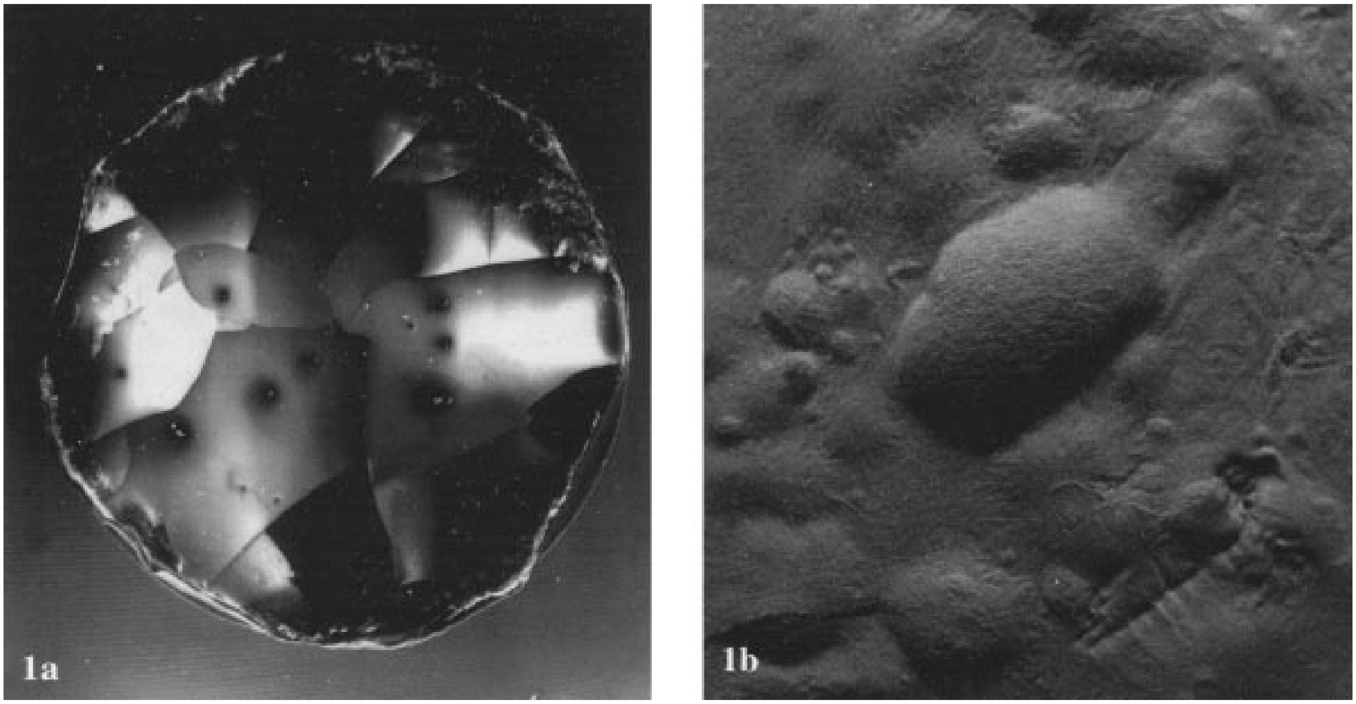

FIG. 1—*The two predominant forms of surface damage are 'out-gassing' (1a 'surface cracking') or electron beam irradiation (1b 'blistering').*

between the sulfur K-alpha and the lead M-alpha peaks overlapping. For example, when a barium particle is detected, and where the area analyzed overlaps the particle and the adhesive, peaks registering in the lead M-alpha region-of-interest from the sulfur K-alpha, and the barium L-alpha, may falsely indicate a lead/barium particle.

The adhesive properties of the materials were also evaluated. The liquid adhesive fared worst, ranging from very weak stickiness when applied, to almost no adhesion when dry. The use of these adhesives would rely on their application onto the stub immediately prior to application. This is not suitable for GSR collection, especially at crime scenes.

Another problem with the liquid adhesives is that only a relatively thin layer of adhesive coats the stub. The amount of surface area in contact with the particles is less than with the tapes, lessening the retention of the particles (see Fig. 2). With thicker adhesive layers some of the smaller particles can be partly submerged in the adhesive. This however does not prevent detection with a 25 kV electron beam. The thin liquid coating also allows the electron beam to penetrate into the aluminum stub.

An advantage of using the carbon conductive adhesives is that carbon coating is not required. However, the surfaces of some particles are not conductive and if large particles of non-conductive material e.g., hair, skin, fibers, are present, charging will still occur and coating will still be necessary.

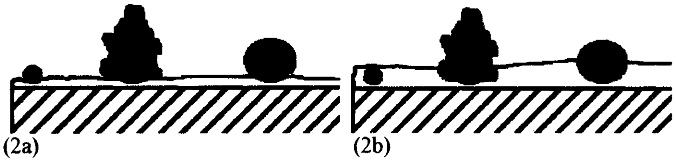

FIG. 2—*Figure (2a) represents a thin layer of adhesive. The surface contact with particles is minimal, (2b) a thicker adhesive with greater surface contact. Partially embedded particles can still be detected with the SEM.*

The long-term storage properties of the adhesives is also important, particularly if the samples require re-analysis at a later stage. The adhesives should remain useable for up to six months and preferably longer (experience with Sellotape 404 over the past ten years, has shown that samples stored away from sunlight for twelve months or longer show no appreciable difference in the quality of the adhesive). If the adhesive cures before sampling it should be discarded. This is easily detected through viscosity and discoloration effects. If the sample has been stored incorrectly, the cured adhesive can be dissolved in an appropriate solvent, e.g., chloroform, and the sample recovered by filtering through a nucliapore filter.

Conclusion

The composition of adhesives used to adhere small particles, powders and thin films to SEM stubs can interfere with the analytical results of the sample. This necessitates a prior knowledge of the composition and characteristics of the adhesive to determine that which is most appropriate. In my experience the best overall is Sellotape 404 double sided adhesive tape which we routinely use for gunshot residue sampling.

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