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Automated Gunshot Residue Particle Search and Characterization

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ABSTRACT: The main disadvantage to gunshot residue (GSR) particle analysis utilizing scanning electron microscope/energy dispersive X-ray (SEM/EDX) instrumentation has been the excessive operator time required for search and identification. This study uses an automated particle search and characterization program for unattended GSR search and identification. This system allows for automatic matrix search, particle sizing, chemical typing, and spectral aquisition with subsequent storage of data to disk for later operator review and verification. This work describes various aspects of the program, determines appropriate parameters adequate for both unique and characteristic GSR particle identification, and evaluates the reliability of data obtained. Samples are collected via the tape lift method from test-firings of .38, .32, .25, and .22 caliber handguns at time after firing intervals of 0 to 6 h. Unique GSR particles are consistently and correctly identified by this method on tape lift samples taken up to 4 h after firing. False positive results of unique GSR particles are not encountered on control handblank samples. This technique appears to provide the forensic science community with an operator-free method of reliable GSR particle search and an improved analyst-time-per-case ratio.

KEYWORDS: criminalistics, ballistics, gunshot residues

Law enforcement and prosecutorial agencies have long sought the "expert witness" who could reliably testify that an individual was found to have gunshot residue (GSR) present on his hands and thus must have handled a firearm or been exposed to a firearms discharge. In the past, numerous methods for detecting gunpowder and GSR on subjects' hands have been employed, including examination of paraffin casts and color tests for nitrates [*1*] and color tests for barium, antimony, and lead [*2*]. Though rapid and inexpensive, these tests lacked sensitivity and GSR specificity.

More recent instrumental methods for detection of GSR include neutron activation analysis (NAA) [*3*], flameless atomic absorption spectroscopy (FAAS) [*4*], and scanning electron microscopy/energy dispersive X-ray analysis (SEM/EDX) [*5,6*]. The first two techniques use a bulk quantitative elemental analysis approach which measures total content of barium and antimony (NAA) or barium, antimony, and lead (FAAS) that has been collected from specific regions of the hands, while the later (SEM/EDX) is potentially superior because it characterizes individual GSR particles both morphologically and elementally.

The deficiency of the bulk analysis methods lies in lack of specificity for GSR since total quantification includes environmental and occupational level contributions. GSR particle analysis by SEM/EDX, on the other hand, offers great specificity, yet may require long

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analysis time with some samples taking up to 8 h [7]. Analyses taking this much time obviously lead to concern, not only for cost and manpower effectiveness, but also for increased possibility of operator distraction leading to false negatives. Matricardi and Kilty have stated that ". . . automation of the search process is one of the essential developments needed before this technique can be routinely used" [7].

The purpose of this study is to evaluate an automated particle recognition and characterization (PRC) program and its value for routine GSR particle analysis in forensic science laboratories, establish whether unattended GSR particle searches and characterizations are feasible, and determine if data obtained from an unattended GSR analysis is reliable.

Methods and Materials

Test-Firings

Test-firings were made in an indoor vertical water recovery cylinder with the following handguns: .38 Special Smith and Wesson Model 49 revolver; .32 S & W Harrington and Richardson Model 732 revolver; .25 caliber Bauer Firearms Corporation semi-automatic pistol; and .22 caliber Rohm Model RG 10s revolver. Commercial ammunition used in the study consisted of Remmington .38 Special 158-gr lead, Remmington .32 S & W Long 98-gr lead, Remmington .25 caliber auto 50-gr metal case, and Federal .22 caliber Hi Power 40-gr lubaloy bullets.

Method of Collection

The collection procedure for GSR particles was similar to previously described methods [6,8]. Scotch Brand 666 double-sided tape was layered on 12-mm-diameter aluminum stubs. Each stub was then dabbed over the back of a hand concentrating on the web area, thumb, and forefinger until the stickiness of the tape was no longer apparent. Immediately after collection, the samples were coated with a thin conductive layer of carbon. Samples were collected immediately after firing and at intervals of 2, 4, and 6 h. Control samples were collected from individuals who had not fired a weapon.

Instrumentation

A Cambridge Stereoscan 200 scanning electron microscope was used in the study and was equipped with the following:

- LaB6 and tungsten emission capability,
- IXI backplate for inclined detector,
- x, y, and z axes stage motorization, and
- G W Electronics solid-state backscatter detector with high resolution mode.

Interfaced to the above equipment was a Tracor Northern TN-5500 Energy Dispersive X-Ray System with an inclined detector, Microscan digital beam control, and Programmable Automation Controller (TN-5600).

Software

The Particle Recognition and Characterization (PRC) program was developed by Tracor Northern to locate and size nonoverlapped low aspect ratio particles. During program operation, each particle analyzed is "mapped" with values for both matrix frame location and x and y particle locations within the frame. Values for the average, maximum, and minimum diameters and for area and perimeter of the particles are obtained in addition to chemical

typing of each individual particle. All of this data can be collated with subsequent output of the results to a printer or disk or to both. The program also allows for optional automatic spectral storage of all particles analyzed. One of the initial requirements of this study was to determine the appropriate parameters to be used in the PRC program for particles unique to gunshot residue to be correctly recognized, measured, chemically typed, and the resultant data stored for later evaluation and verification.

Table 1 shows the particle setup parameters used in the PRC program for a typical GSR search in this study. Use of a guard region (an operator-designated area surrounding any given frame) was a means of rejecting particles too close to the edge of a frame to be completely within the frame of interest. The width of the guard region was approximately one half of the maximum particle size; therefore, if the center of a particle lay within this region

TABLE 1—*PRC parameter setup conditions for GSR particle search.*

Label: GSR auto search setup		
Magnification	=	501
Guard region	=	25.000 μm
Minimum particle size	=	2.000 μm
Maximum particle size	=	50.000 μm
Low shape factor	=	1.000
High shape factor	=	1.400

FIG. 1—*SEM image of an irregular particle shown with the PRC program's calculated shape factor. Magnification:* × *1717.*

the particle was rejected. Minimum and maximum particle size refer to the smallest and largest particles, respectively, whose data was retained. Particles smaller than minimum or larger than maximum size were rejected for GSR analysis purposes. Shape factor is a value determined for different particles using measured perimeter and area calculations and is defined by the following formula:

$$SF = P^2/(A * 4 \text{ pi})$$

where

SF = shape factor,
P = perimeter, and
A = area [8].

The closer the shape factor is to 1.0, the closer the shape of a particle is to a sphere. Any particle whose shape factor was lower than the specified value was discarded as was any particle whose shape factor was larger than the high shape factor. Figures 1 through 3 show several particle types evaluated by the program and the resultant calculated shape factors.

As shown in Table 2, particle spectra were obtained from 0 to 20 keV with the maximum beam dwell time on any particle being 8 s. Three windows or regions of interest were established at appropriate kiloelectron volt ranges to define chemically GSR unique and characteristic particles.

In this work, the elemental composition or "chemical type setup" (Table 3) for four differ-

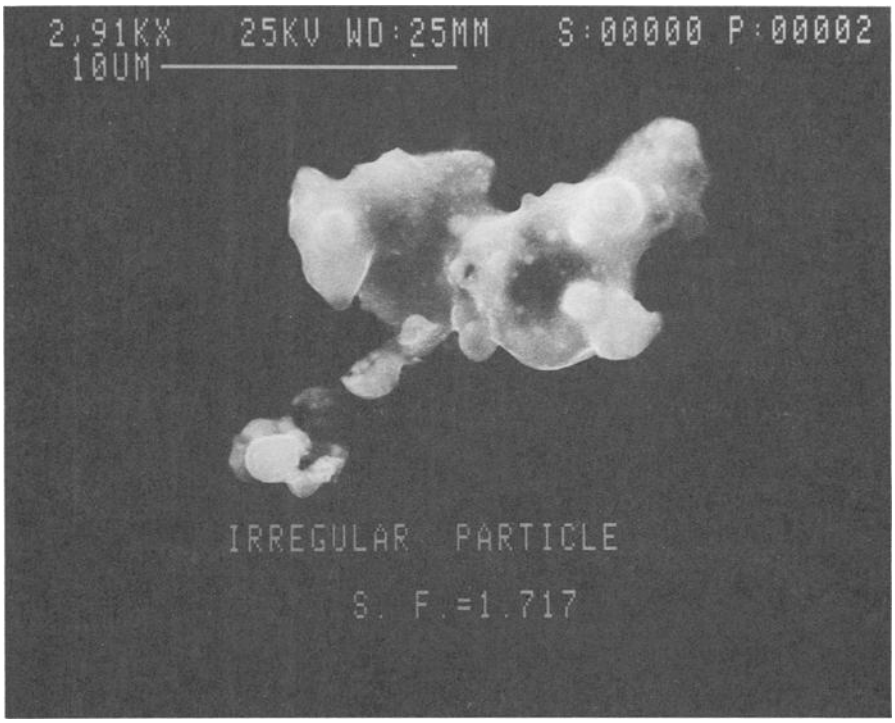

FIG. 2—*SEM image of a linear particle shown with the PRC program's calculated shape factor. Magnification :* × *838.*

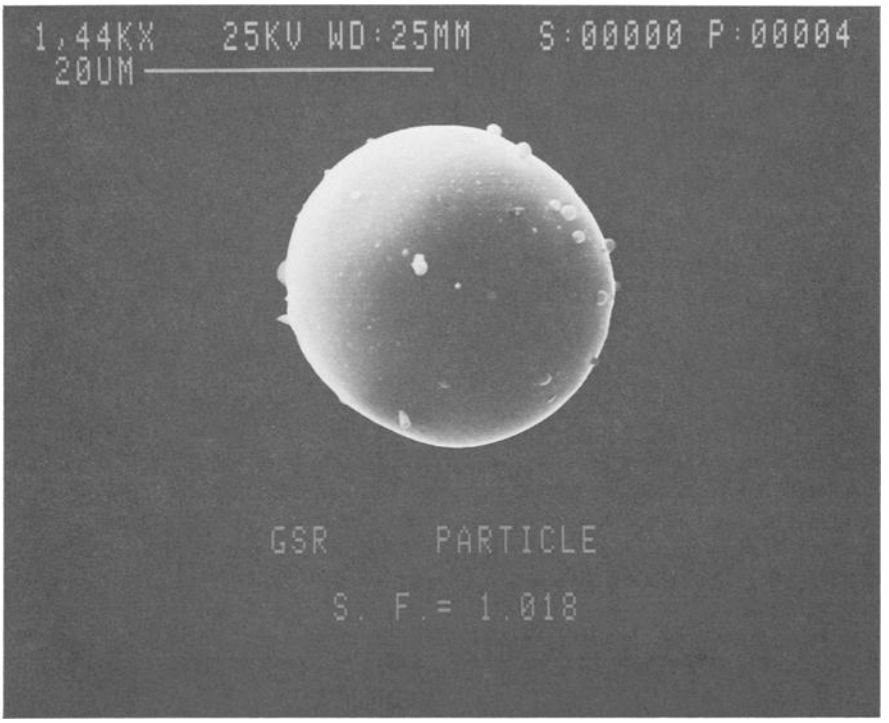

FIG. 3—*SEM image of a spherical GSR particle shown with the PRC program's calculated shape factor. Magnification:* ×*850.*

TABLE 2—*EDX setup parameters for GSR particle search.*

X-RAY PARAMETERS:			
Energy range from: 0 to 20 keV			
Acquisition time: 8 s			
Number of regions: 3			
REGION SETUP:			
Name	Emission Line	Range	Centroid
Pb	Lα	10.38:10.72	10.55
Sb	Lα	3.48:3.80	3.60
Ba	Lα	4.32:4.72	4.52

ent multi-element type particles was found to be adequate for characterizing GSR particles. The Type 62 or nonintegratable particles were particles found during the search in which insufficient X-ray counts were obtained for particle identification. The Type 63 or unknown type particles had sufficient X-ray counts but did not match any predefined particle types found in the chemical type setup table.

TABLE 3—*PRC multi-element type setup conditions for characterizing GSR particles.*

CHEMICAL TYPE SETUP:		
Search for multi-element types first		
Type: 1	Name:	GSR particle
Pb: concentration > 0		
Sb: concentration > 0		Ba: concentration > 0
Type: 2	Name:	Ba-Pb type particle
Pb: concentration > 0		
Sb: not present		Ba: concentration > 0
Type: 3	Name:	Pb-Sb type particle
Pb: concentration > 0		
Sb: concentration > 0		Ba: not present
Type: 4	Name:	Ba-Sb type particle
Pb: not present		
Sb: concentration > 0		Ba: concentration > 0
Type: 62	Name:	nonintegratable
Type: 63	Name:	unknown

Experimental Procedure

The GSR stubs were mounted in a multistub specimen holder and the analyses performed on the SEM at an operating voltage of 25 keV, 25-mm working distance, ×501 magnification, and a 35° takeoff angle. Specimen current was maintained at approximately 450 pA. The microscope CRT and backscatter detector (BSD) were both operated at a slow scan rate with the BSD placed in the high resolution mode. A 100-frame contiguous matrix was set up with the PRC program and applied to random areas of the stubs. This represented approximately 3.9% of the total tape collection area of the stubs. A video recorder and camera were utilized to monitor spectral aquisition and beginning and ending analysis times.

Results and Discussion

Tables 4 through 7 show search time requirements and results of particle count and characterizations of GSR from various caliber weapons collected at designated postfiring intervals. The values shown in these tables were consistent with those found in subsequent 100-frame searches on the same stub over different matrices of the tape collection area. As would be expected, GSR particle count dropped off rapidly with increased postfiring collection time. The results seen in Tables 5 and 6 did show exceptions to this prediction in the .32 and .25 caliber test-firings. In each of these searches, more GSR particles were found on the 4-h postfiring collection stub than the 2-h one. This could have been due in part to variations in

TABLE 4—*Results of automated search of stubs collected immediately after firing.*

Caliber	Search Time, min	Total Particles	Particle Types					
			GSR	Ba-Pb	Pb-Sb	Ba-Sb	Nonintegratable	Unknown
.38	55	154	32	12	11	18	56	25
.32	52	94	33	3	3	18	23	14
.25	67	127	39	5	0	30	36	17
.22	56	185	19	30	20	7	59	50

TABLE 5—*Results of automated search of stubs collected 2 h after firing.*

Caliber	Search Time, min	Total Particles	Particle Types					
			GSR	Ba-Pb	Pb-Sb	Ba-Sb	Nonintegratable	Unknown
.38	56	63	8	3	3	7	29	13
.32	61	52	3	0	1	9	29	10
.25	97	225	6	0	12	36	124	47
.22	76	174	10	1	9	29	91	34

TABLE 6—*Results of automated search of stubs collected 4 h after firing.*

Caliber	Search Time, min	Total Particles	Particle Types					
			GSR	Ba-Pb	Pb-Sb	Ba-Sb	Nonintegratable	Unknown
.38	64	151	5	0	0	19	99	28
.32	78	188	8	1	2	31	121	25
.25	47	84	10	3	7	24	20	20
.22	47	122	6	2	10	26	44	34

TABLE 7—*Results of automated search of stubs collected 6 h after firing.*

Caliber	Search Time, min	Total Particles	Particle Types					
			GSR	Ba-Pb	Pb-Sb	Ba-Sb	Nonintegratable	Unknown
.38	67	107	0	0	1	1	97	8
.32	40	39	0	0	1	0	37	1
.25	71	147	0	0	0	18	102	27
.22	61	133	0	1	3	9	108	12

total particle deposition from different test-firings [*7*] and in part to variations in activity of the test subjects before sample collection.

In general, approximately 1 h was required to accomplish the 100-frame search; however, this value was found to vary depending upon the gain and contrast setting on the BSD and on the particle sizing values assigned in the PRC program.

When the search process in each experiment was completed, random frames within the previously searched matrix were chosen and then manually searched and analyzed for comparison with the PRC program's results. In every frame examined in which the program defined a unique GSR particle, subsequent manual examination verified a correct chemical typing. The manual searches were performed using secondary electron and slow scan backscattered electron imaging. The efficacy of using BSD over SEI in GSR particle search can be seen in Figs. 4 and 5. This demonstrates several particles as seen in the secondary image and in the mode utilized by the GSR autosearch program, the backscattered mode.

Another feature of the PRC program is its ability to evaluate size and shape of GSR particles simultaneously while determining the chemical composition. Table 8 shows the average

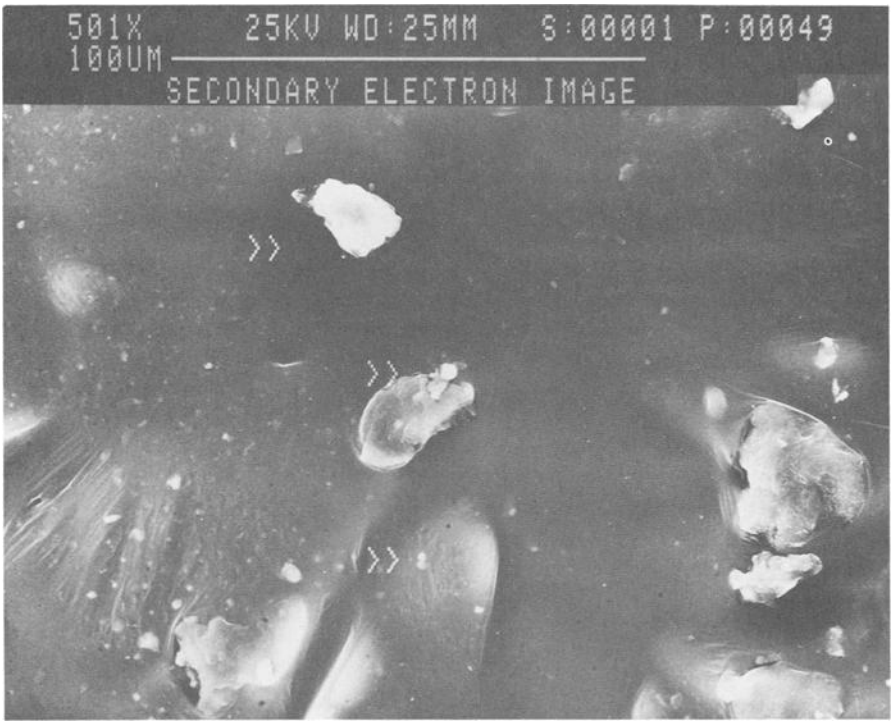

FIG. 4—*Secondary electron image of one frame of a search matrix showing difficulty of observing GSR particles in this imaging mode. Magnification:* ×*276.*

TABLE 8—*Shape and size characterizations of GSR particles found on search of stubs collected immediately after firing.*

Caliber	Total GSR Particles	Average			
		Shape Factor	Standard Deviation	Diameter, μm	Standard Deviation
.38	32	1.167	0.096	2.95	0.90
.32	33	1.261	0.073	3.26	1.07
.25	39	1.173	0.084	2.82	0.96
.22	19	1.184	0.104	3.38	1.17

calculated shape factor and average particle diameters obtained from samples collected immediately after firing the test handguns. These values indicate that the average diameter of a GSR particle located by random searching of stubs collected immediately after firing was approximately 3 μm. These particles were also spherical in shape and fit well within the limits incorporated in the PRC parameter setup table.

Postexperimental review of video tapes made during the search process revealed EDX spectra of characterized GSR particles typical to that shown in Fig. 6.

The unknown particles found during the search generally consisted of lead, barium plus sulphur, and calcium-containing particles, while the nonintegratable particles contained mainly iron, copper, potassium, chlorine, silicon, bismuth, and tin.

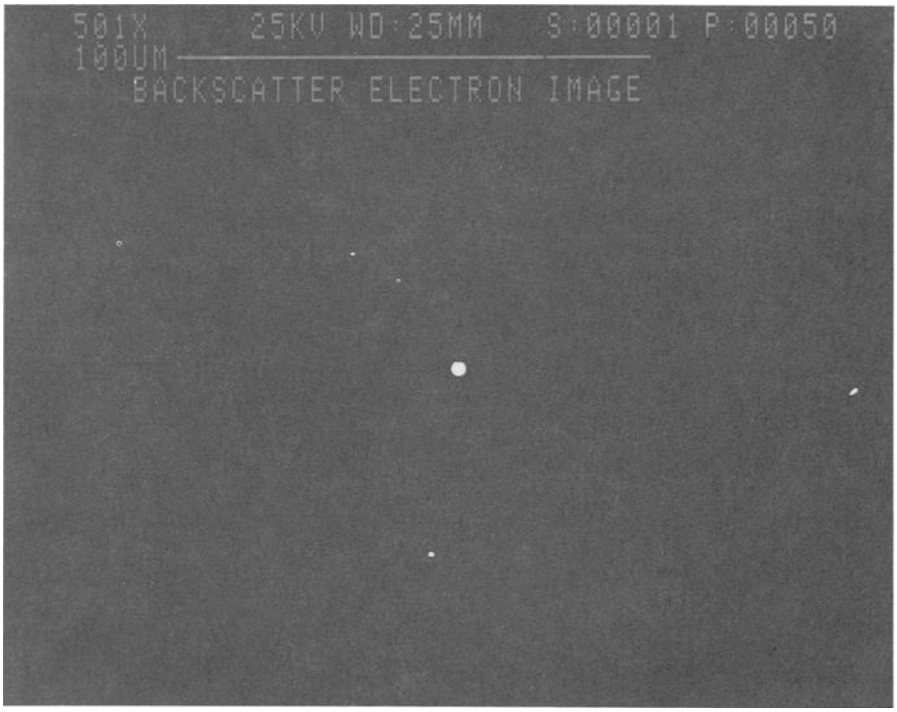

FIG. 5—*Backscatter electron image of the same frame as shown in Fig. 4 readily showing GSR particles. Magnification:* ×296.

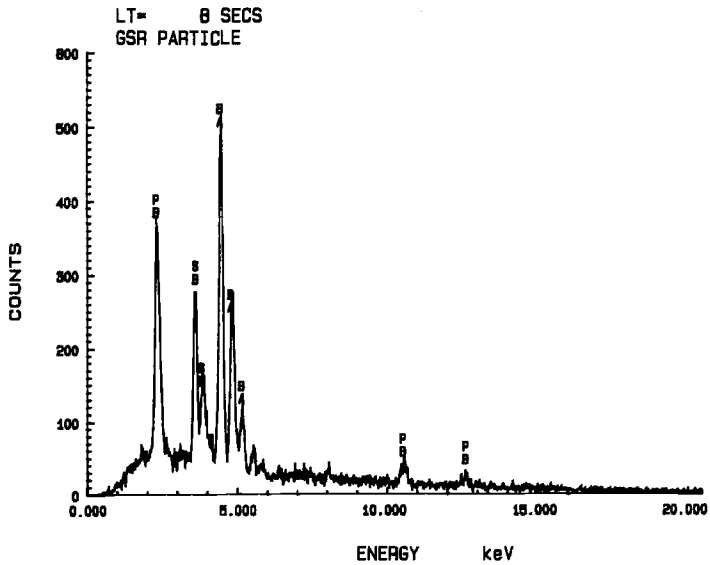

FIG. 6—*Typical EDX spectrum of a particle characterized by the PRC program as a GSR type.*

Control stubs were made from the hands of five subjects who had not fired a weapon. Using identical parameters to those performed on the test stubs, no GSR particles were identified on any of the five stubs.

Conclusions

Particle analysis in recent years has emerged as the most accurate and successful method of identifing and detecting GSR; however, this advantage has been overshadowed by the often inordinate amount of time required for search and identification. Findings in this study support the effectiveness of using the PRC program for GSR identification. The system seems to provide reliable data not only in regard to chemical typing but also in terms of morphological shape characterizations. Forensic scientists are relieved of the tedious and lengthy process of manual GSR particle search, allowing them to proceed with other tasks. The data generated automatically by the program is available for in-depth evaluation and reporting of results at the convenience of the scientist. These factors provide both improved qualitative and quantitative productivity in the forensic science lab.

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