Examination of firearm primers and gunpowders by scanning electron microscopy/energy dispersive x-ray analysis.

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Abstract

Observations by scanning electron microscopy/energy dispersive x-ray spectroscopy (SEM/EDS) of unfired standard primer material (lead/antimony/barium) are presented from the centerfire cartridge primers of Winchester, Speer and Remington. Also examined by SEM/EDS are the surfaces of thirty-two gunpowders (GPs) from Winchester, Federal, Hercules, DuPont and Remington. Compounds of sulfur, potassium and barium as well as primer-like particles composed of lead, antimony and barium were found on the surfaces of many of these GPs. The elements of these compounds likely contribute to gunshot residue (GSR) generated by the discharge of a firearm. We introduce the term, “hybrid GSR” which will account for both the contribution to primer GSR by the interior firearm surfaces GSR and the elements associated with GP.

Introduction

For centerfire ammunition, a cartridge consists of a projectile (usually a copper jacket covering the lead core), brass casing, gunpowder (GP), and a capsule or cup at the base of the cartridge containing the primer mixture. The compression of the of the primer capsule causes a low-intensity deflagration of the ignition mixture (the primer material) which projects incandescent particles through the casing vent hole and into the mass of the propellant GP [1]. The incandescent particles induce the combustion of GP, which produces gas at high pressure and temperature that forces the projectile from the casing into
the firearm’s barrel. The GP burning and pressure increase continues in the bore of the firearm until the projectile leaves the barrel.

The most common centerfire primer material contain lead styphnate, barium nitrate and antimony sulfide. Additional metals, such as aluminum, and other elements can be included in the primer mixture. In conventional primer mixture (lead/antimony/barium), for each ingredient of the primer mix has the following role [2]:

1. lead styphnate – initiator i.e. the explosive that, with the help of tetrazene, is activated with the percussion
2. barium nitrate – oxidant i.e. provides oxygen for both the initiation and the combustion
3. antimony sulfide – the fuel that supports the primer flame
4. aluminum micro-GP – raises the primer flame temperature
5. Tetrazene – a sensitizer which works in conjunction with lead styphnate
6. PETN – a high explosive that increases the mix brisance

Concerns over the toxic byproducts produced by three-component primer ammunition (PbSbBa) resulted in the development of new, non-toxic compositions. Lead-free primers do not contain lead, antimony and barium and their deflagration produces particles that have compositions similar to common elements in the environment or nontoxic metals. Lead-free or leadless primers have compositions (e.g., zinc/titanium, boron/potassium, etc.) depending on the manufacturer and have been commercially available since the late 1980s. Dynamit Nobel’s Sintox® was the first to introduce a nontoxic primer [3,4] which was followed by Winchester Ammunition (Super Unleaded®) and subsequently Winchester (Super Clean NT® and WinClean®). Federal, Remington and Speer all have proprietary leadless primed ammunition [5,6].

We present in the following a scanning electron microscopy/energy dispersive x-ray spectroscopy (SEM/EDS) analysis of primer material (three manufacturers) and of the surfaces of GPs from six manufacturers (32 samples).

Unfortunately, the electron beam rastering potentially energetic materials such as firearm primer particles present a danger to the SEM. Primer particle microdeflagrations have been reported during SEM examinations [7]. The damage by an energetic material deflagration is to the EDS detector window where it can be fractured by the sudden loss of vacuum as well as the force of the explosion itself. Damage to other components in the SEM chamber could also occur which would depend on the size of the explosion, but deflagrations of this size have not been reported. A deflagration in SEM chamber used in this study did not occur as a result of the exposure of the energetic material to the electron beam.

The SEM images and spectra were taken between 1990 and 1995. At the time of this study the observations were considered as a curiosity - not significant to warrant submission for publication. A recent discussion by us of the literature concerning the “memory effect” on the composition of GSR [8-17] by the authors caused a return to this early study. The review of this work on inorganic particles associated with GP led to the hypothesis that there is an additional source of elements contributing to the composition of GSR which has not been reported previously. Unfortunately, due to the danger of instrument damaged by a follow up study with a more modern equipped SEM, the publishing of this original study now, more than ever, is needed to understand the composition of GSR resulting from the
discharge of a firearm and the possible contribution of heavy metals and elements associated with GP to the resultant GSR from not only for nontoxic primered ammunition but also for standard primered (PbSbBa) ammunition.

Materials and Methods

An ETEC Autoscan scanning electron microscopy with secondary electron imaging (SEI) and backscatter electron imaging (BSEI) was used in this study. The operating voltage was 20kV. The EDS spectra were acquired by a Kevex beryllium window detector coupled to a Kevex Delta EDS analysis system and photographs were taken by a Polaroid camera system of the Kevex CRT spectral display. The Polaroid photographs of the Kevex CRT spectra were of such poor quality that they required rendering for this publication by an overlay procedure in Photoshop.

The primer samples were affixed to the SEM stub by carbon paint and were sufficiently electron conductive to not require carbon coating. Three samples of primer mixes from Winchester, Speer (CCI) and Remington were observed in the SEM and compositions determined of a number of the particles by EDS without recording spectra.

All of the GP samples showed sufficient conductivity for beam stability (i.e., no significant charging) to also not require carbon coating. It appears all the GPs examined in this study had a coating of graphite. The GP was affixed to the standard graphite impregnated sticky tape surface did not require pressing to assure good contact with the tape. Twenty-six GP samples were extracted from cartridges (Winchester, Remington and Federal), and six from bulk GP containers for cartridge reloaders (Winchester, DuPont and Hercules).

Specimen images, both SEI and BSEI, were recorded on Polaroid photograph paper which were scanned by a Epson 4180 Photo scanner and these Polaroid photographs of both SEI and BSEI were repaired (streaks and other print defects) in Photoshop. Some of the Polaroids had negatives in which digital images were taken on a light table using a Pixera 600CL with a Computar 10X macro-zoom lens.

It was discovered early in this study that different lots of Winchester 9mm Silvertip® hollow point (STHP) ammunition had GPs with different surface inorganic particles. In order to determine the range of GP types, fifteen different lots of Winchester 9 mm STHP ammunition were examined.

Results

Primer compositions. In Winchester (small pistol type) we observed granular structures of a variety of sizes consisting of tri- bio- mono elemental crystals (Fig.1A). This primer material has relatively large particles from 10 to greater than 60 microns composed of PbSbBa and Pb. Smaller particles, which are less than 5 microns, are present. One of these particles was identified as Ba (likely BaNO₃) and the other PbSb. The number of EDS analyses was, of course, too small to fully characterize this sample.

The Speer primer material had relatively large bi-mono elemental crystals of sizes ranging from 10 to greater than 60 microns which were Pb and Al (Fig.1B). Smaller particles from less than 1 to 3 microns of Ba, PbSb and BaAl were identified. A notable difference between this primer material and the Winchester primer is no particles of tri-component (PbSbBa) composition were detected. The number of EDS analyses was too small to fully characterize this sample.
The third primer material was from a Remington 9mm cartridge (no images) which was similar to the Speer primer composition (Fig. 1B) in having relatively large Al and Pb particles with smaller particles of Ba and PbSb. The number of EDS analyses was too small to fully characterize this sample.

Figure 1. A. Secondary electron image of Winchester .357 Magnum STHP cartridge primer particles; there are granular structures consisting of tri-bi-mono elemental crystals of various compositions mixed together. The carbon paint obscured some of the particles. The elemental compositions of the particles are labeled where EDS analyses were made. B. Backscatter electron image of the primer material from a Speer cartridge primer cup (small pistol type for reloaders) showing a similar irregular granular structure of all elemental compositions consisting of bi-mono crystals irregularly distributed in the mix; relatively large crystals of aluminum (Al) are present. Tri-component (PbSbBa) particles were not detected.

Surfaces of GP. Thirty-two GP samples were examined by SEM/EDS (Table 1). All the GPs showed inorganic coatings and inclusions of some or most in their graphite coatings, except three (Table 1: 8, 15 and 21) which had no inorganic coatings or particles detected.

a. Gunpowder covered by sulfur/potassium/barium (S K Ba). The GPs from eight 9 mm Winchester cartridges of different lot numbers (Table 1: 1, 2, 6, 7, 9, 12, 13 and 14) show high backscattering electron material that coats some of the GP flakes (Figs. 2B, 2C and 3A). Higher magnification of these heavy electron backscattering GP surfaces (Fig. 2D) show this material does not uniformly cover the GP flake’s surface. Engery dispersive spectral analysis of this material shows it is composed of sulfur/potassium/barium (S K Ba, Fig. 2E). One other Winchester caliber cartridge (Table 1: 16) and a Remington-Peters (R-P) of cartridge had GPs coated with S K Ba (Table 1: 26). Two of the Hercules bulk powers, had also GPs coated with S K Ba (Table 1: 28 and 30).

b. Gunpowder with primer-like (PbSbBa) particles. Twelve of the 32 GPs examined had primer-like (PbSbBa) particles associated (Table 1: 1, 2, 3, 6, 7, 16, 18, 22, 26, 30, 31 and 32; Figs. 3, 4, 5 and 6). These particles were less than 1 to 10 microns and embedded in the graphite coating on the GPs. They were usually evenly distributed over the surfaces of the GPs (e.g., Figs. 3 and 4). One of the GP samples had numerous primer-like particles on its surface which was associated with a surface coating of sulfur/potassium (Table 1: 31; Fig. 5). Some of these flakes appeared to completely covered with this material, others partially and some none at all (Fig. 5A).
Table 1. Listing of the GPs examined in this study; GP samples 1 through 26 were extracted from cartridges, sample 27 through 32 from bulk containers. The first fifteen entries are the results of examination of the GPs of fifteen different lots of Winchester 9mm STUP.

**Key:**

<table>
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<tr>
<th>ORIGIN OF GP</th>
<th>GP FORM</th>
<th>Major ASSOC ELEMENTS</th>
<th>OTHER NUMEROUS PARTICLES ASSOCIATED</th>
<th>OTHER PARTICLES?</th>
<th>COMMENTS</th>
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<td>S K Ba</td>
<td>PbSbBa 1 - 10 μm</td>
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<td>(S) K</td>
<td>PbSbBa 1 - 10 μm</td>
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*gp not coated, but particles on all surfaces
Figure 2. A. Secondary electron image of GP from a Winchester 9 mm STHP cartridge (Table 1: 1). B. Backscatter electron image of the same sample area as A. C. A sample from a different Winchester 9 mm STHP cartridge (Table 1: 14). D. Higher magnification the surface of the heavy electron backscattering GP flake in C. E. Spectrum from the heavily electron backscattering area shows this material is a composed of sulfur-potassium-barium.

c. Gunpowder covered by sulfur/potassium (S K). Of the thirty-two GPs examined, six had flakes coated with sulfur/potassium (Table 1: 3, 4, 5, 24, 29 and 31). Coating of GP with this material ranged from partial for some (e.g., Winchester 9mm GP (Table 1: 3; Fig. 6) to most of the flakes totally covered (e.g., Table 1: 31; Fig. 5).

d. Gunpowder covered by sulfur(trace)/potassium ((S) K). Two of the GP samples had a trace of sulfur with potassium (Table 1: 17 and 27; Fig. 7). Both of these were flatten ball GPs.
Figure 3. A. Winchester GP from a 9mm Win STHP cartridge (Table 1: 6); primer-like particles (PbSbBa) are associated with the graphite coating of the GP. B. Secondary electron image of one of these particles embedded in the graphite coating of a GP flake. C. Backscatter electron image of the particle shown in B. D. Spectrum of the composition of these particles.

Figure 4. Flake GP from a Winchester .38 Special cartridge (Table 1: 18). A. Secondary electron image of the GP. B. Higher magnification SEI showing a particularly rough surface compared to the other flake GPs. C. Backscatter image of the area shown in B; the small heavily backscattering particles (white), range from 3 to 10 microns, are primer-like particles with the composition PbSbBa.
Figure 5. DuPont 700X bulk GP (Table 1: 31). A. Backscatter electron image of this GP showing most of the flakes are covered with a coating of potassium–sulfur; some of the flakes have only partial coatings of the potassium-sulfur (arrows), others almost none at all (top of image). B. Backscatter electron image at a higher magnification of the surface of a flake. C. Spectrum of the coating (gray material in B) of potassium-sulfur. D. Spectrum of one of the many primer-like particles (white in B) associated with the potassium-sulfur coating.

Figure 6. A. Another GP sample from a Winchester 9mm STHP cartridge (Table 1: 3); in this sample there were no small primer particles distributed over the surfaces of the GP flakes, but a small number of flakes were almost totally covered with primer particles. Other GP flakes had partial coatings of sulfur-potassium (S K). B. Spectrum of the primer-covered flake shown in A. C. Spectrum of sulfur-potassium material which covered some other flakes in this sample.
Figure 7. A. low magnification SEI of Winchester flattened ball GP extracted from a .357 STHP cartridge (Table 1: 17). B. Higher magnification SEI of the GP shown in A. C. Same GP as B, but a BSEI. D. Spectrum of the material coating the GP shown in C; virtually all the GP have coatings of potassium particles with a trace of sulfur. E. Winchester 296 bulk GP sample (Table 1: 27) for cartridge reloaders for “.357 Magnum, .44 Magnum and M1 carbine;” only a small number (< 20%) of the GP has potassium with a trace of sulfur associated coating (high BSE GP). No primer-like particles were observed associated with either of these GPs.

Figure 8. Remington–Peters 380 Auto +P GP (Table 1: 19). A. Backscatter electron image of the flake GP; the bright spots are aggregations of lead particles. B. Secondary electron image of one of the bright spots showing numerous angular particles. C. Backscatter electron image at the same magnification as the SEI B; the bright spots correspond to the angular particles and are lead, presumably lead styphnate crystals.
Figure 9. Flake GP from Winchester 9mm STHP (Table 1: 11). A. Secondary electron image. B. Backscatter electron image of same field as in A showing a potassium coated flake; the small heavy BSE particles are CuZn. C. Spectrum of the heavily BSE coated GP.

e. **Gunpowder covered by potassium (K).** Two GP samples had potassium associated with some of the flakes without any other direct associated elements (Table 1: 11 and 23; Fig. 9).

f. **Gunpowder covered by silicon/sulfur/calcium (SiS Ca).** Only a single GP sample from a Winchester 9mm Silvertip® hollow point (Table 1: 10) was observed which had a silicon/sulfur/calcium (SiS Ca) coating on all the GP in this sample. This GP was flattened ball (e.g., Fig. 7E). There was one other GP of this form (Table 1: 13) observed in the fifteen samples of the Winchester 9mm STHP GPs. But this GP sample did not have silicon/sulfur/calcium association.

**Discussion**

A number of different chemical additives to GP are flash suppressants and experiments have demonstrated that compounds of potassium (K$_2$SiF$_6$ or potassium silicone fluoride – K$_3$P0$_4$ or potassium phosphate – KC$_4$H$_5$O$_6$ or potassium hydrogen tartrate – K$_2$SO$_4$ or potassium sulfate – KNO$_3$ or potassium nitrate) assure a reduction of the light emission during a firearm discharge. These compounds, composed of potassium and sulfur, likely act as reducing agents and, as noted by Heimerl et al. [18], will suppress flare. Potassium associated with sulfur was observed on GPs (Table 1; Figs. 5, 6 and 7). It is reasonable to assume the barium association with sulfur and potassium (S K Ba, Table 1; Fig. 2) on a GP coating is to not only to reduce of the muzzle flash, but the barium (likely barium nitrate) serves to accentuate GP oxidization within the barrel [2].

It is also possible some manufactures add on GP both Ba(NO$_3$)$_2$ and primer mixture GP to improve ignitability. But barium alone without sulfur and potassium association was not seen in this study.

The GP of one of the Winchester STHP cartridges (Table 1: 10) had a coating of silicon-sulfur-calcium which was unlike any other GP examined. Morin notes, “Calcium is usually detected in GSR produced
by priming mixtures containing calcium silicide added to antimony sulfide as extra fuel. See, for example the RWS ‘Sinoxyd’ primer or the U.S. Patent 1,851,398” [19]. Antimony was not detected. The actual compound or compounds composed of these three elements is currently unknown.

An added note, both calcium and phosphorus can be found in gunshot residue (GSR) as sub process impurities during manufacture of 22 Long Rifle rimfire primer in situ (directly in internal surface of rimfire cartridge case): in fact calcium hypophosphite Ca(H$_2$PO$_2$)$_2$ is used for lead styphnate and for lead nitrate-hypophosphite preparation, the latter as a function of igniter in case of inadequate flame sensitivity of the primary explosive [20].

There are primer particles (PbSbBa) associated with the apparent graphite coating of some of these DPs (e.g., Figs. 3, 4C and 5) and one GP sample had some of its flakes totally covered with primer particles (Fig. 6A). Gunpowder flakes in other samples were covered in whole or in part with S, K and Ba (Figs. 2 and 3).

“All analyses of GSR particles from the ‘Clean’ ammunitions regardless of type indicate a significant presence of the Ba, Sb and Pb particles as well as barium only, antimony only and lead only particles.

The origin of these particles may be traced to the standard ammunition fired from the same firearm previously. Apparently, regardless of how well the firearm was cleaned, it retains enough old particles and releases some of them each time a round is fired.” [14]

Our results indicate inorganic compounds associated with GP likely affect the composition of the GSR resulting from the current discharge of the firearm. So, with the discharge of a leadless primed cartridge, if its GP has associated primer particles with the composition of PbSbBa as well as K and Ba, it will produce characteristic and consistent GSR particles (e.g., Pb, PbSb, PbBa and SbBa) regardless of the firearm’s lacking a history of firing standard primer ammunition. Indeed, a major source of contaminate Pb, Sb and Ba to the GSR produced by leadless ammunition could be from the inorganic compounds associated with GP.

**Conclusions**

The primer material composition (PbSbBa) within the cartridge cup differs between manufactures in that all three elements with their associate anions can be combined in individual tri-component particles (Fig. 1A) or separate in PbSb, Pb, and Ba only, without tri-component PbSbBa particles (Fig. 1B). The primer sample with tri-component (PbSbBa) particles (Fig. 1A) also had separate particles of PbSb, Pb and Ba.

The composition of muzzle or breech GSR upon discharge of a firearm has three sources:

1) the primer material from within the primer cup of the cartridge at discharge,
2) the GSR coating the bore and other parts of the firearm from previous discharges,
3) the inorganic compounds associated with the GP.

The different GPs from the same manufacturer with the same caliber and ammunition type (Table 1: 1–15), but different lot numbers suggests, at least for Winchester, that they buy GPs from outside manufacturers. Even the form of GP varied between lots. Use of the same ammunition type, but a
different lot number in testing a firearm could produce a dissimilar GSR composition from a sample collected from a suspect or surface where the “same” ammunition was used.

In effect, GSR collected from a suspect shooter’s hands or other surfaces will not be reflective of a GSR sample from within a casing which will be only from the primer material of that cartridge as well as its GP elements. Not only does the elemental composition of GSR sample show a “memory effect” [8-17], that is, the contribution of elements from previous discharges of a firearm, but there also is a possible contribution of primer particles (i.e., PbSbBa) from the GP and other inorganic additives associated with the GP. This implies that even for the leadless primer ammunitions [6], a contribution of GP tri-component (PbSbBa) particles and other inorganic elements (Ba, K etc.) could come from the GP. Therefore, we propose the term “hybrid GSR” for both muzzle and breech GSR populations and abandonment of the term “memory effect.”

The knowledge of the composition of the priming mixtures used by the various brands and the coatings of GPs of the cartridges could allow estimating the composition of the GSRs released by the firing of a specific cartridge.

Could there also be inorganic inclusions within the GPs?

With this work we warn of risk in analysing priming mixtures and GPs. As noted other researchers have had ignition of primer material within the chamber of the SEM causing expensive destruction of their EDS detector windows [7]. It is unknown why our examinations of primers and GPs did not end in disaster. Perhaps it has to do with our SEM operating at an electron beam acceleration voltage of 20 kV, while the others which experienced deflagrations were at a higher kV.

References


