

TEM CHARACTERIZATION OF NANOPARTICLES ARISING FROM A GUNSHOT.

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Gunshot residues (GSR) are the main tracing source in violent crimes and can be found on human hands as well as on objects near the discharge. Therefore, it is crucial to understand how GSR is created and deposited. This kind of residues has been largely studied [1], reaching a consensus that most particles formed are regular spheroids ranging in size from 1 to 10 μm , although smaller detectable particles can be found. The most commonly chosen method by several laboratories conducting GSR analysis involves Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS) systems. However, only few works have been reported as using Transmission Electron Microscopy (TEM) within this field of research [3, 4]. Such studies claim that the GRS are found in several shapes and individual particles could be as small as 2 nm. In the present work, we propose to systematically use TEM in order to fully characterize GSR particles. A unique GSR particle is defined when Pb, Sb and Ba are combined into a single particle. Nevertheless, recent studies indicate that Pb-Sb-Ba particles arise from brake lining pads as well [2]. This is a delicate question to be treated because of the possibilities of incriminating innocent people in criminal occurrences. Samples for TEM analysis were collected on a 3 mm grid, which was previously placed at the same height of the gun shooter, at 15 cm distance away from his hand, and perpendicular to the line of fire; the grid surface being perpendicular to the ground floor. Under these conditions, one can be certain that a representative sampling of GSR particles are free to be collected into a grid and remain attached to the supporting carbon film by Van der Waals forces. A .38 *special* caliber revolver manufactured by Taurus Company was utilized with a hollow point .38 *S&W* ammunition from CBC Brazilian Industry. After collection, the samples were observed in a JEOL 2010 TEM instrument operated at 200 kV accelerating potential. Conventional diffraction contrast and phase contrast modes were applied. The first together with selected area diffraction pattern allows one to detect overall particles distribution and size down to 1 nm resolution. The second, under specific beam orientation, is capable of generating high resolution images with 2 Å resolution, which allows to define atomic planes. For the purpose of the present study, we are reporting diffraction contrast results by means of dark field (DF) and bright field (BF) images together with composition and analysis obtained through Energy Dispersive Spectroscopy (EDS) attached to the TEM instrument. Images were captured by a Gatan CCD camera (model 794). Diffraction patterns were directly recorded on negative plates. In the present TEM study, several particles in a variety of shape and size were observed, thereby confirming the existence of GSR particles smaller than 1 μm , as shown in figure 1. Among the various particles examined, we point out two, labeled A and B. Particle A, exhibited in figure 2a and 2b, BF and DF images respectively, is an example of a non-spheroidal particle, which is, in fact an agglomerate of several nanoparticles. Figure 2c is the corresponding EDS spectrum, with the identified peaks indicating the presence of Pb, Sb and Ba, characterizing a typical GSR. Figure 3a demonstrates that particle B is also an agglomerate of nanoparticles, in a different morphology, as observed in figure 3c, a DF image, which corresponds to the boxed selected area, presenting a GSR feature as well, corroborated by its EDS spectrum of figure 3d. The corresponding selected diffraction pattern is exposed in figure 3b. A high resolution image of the circled area is displayed in figure 3e, where atomic planes can be observed together with Moiré fringes. Work in progress focuses on establishing a systematic methodology to apply the above described analytical electron microscopy techniques in real forensic cases.

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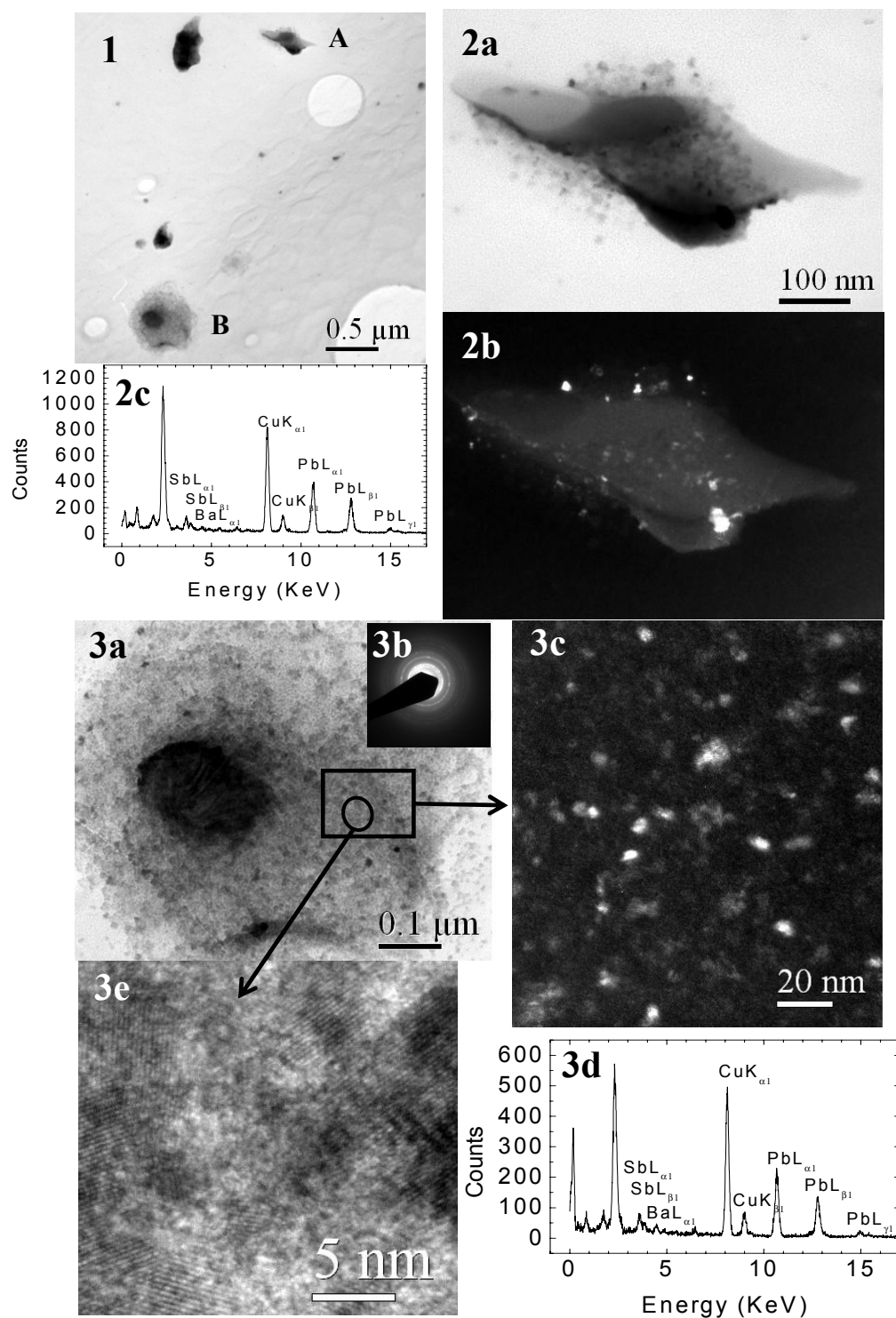


Figure 1. Low mag BF image of observed agglomerated particles, labeled A and B.

Figure 2. Agglomerate A; (a) BF image, (b) DF image, emphasizing nanoparticles area, (c) EDS spectrum.

Figure 3. Agglomerate B; (a) BF image, (b) DF image, (c) selected diffraction pattern, (d) high resolution image, (e) EDS spectrum.