FORENSIC USE OF PHILIPS XL 30 ESEM

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ABSTRACT: Since late 1998, SEM has been installed in Forensic Centre, Zagreb, Croatia. Philips' Environmental Scanning Electron Microscope (ESEM) XL 30 with EDAX EDX-detector and Philips' GSR NT v. 3.2 software is dedicated primarily for investigation of gunshot residue particles (GSR). Instrument spectral resolution is about 142 eV at amplification time of 10 microseconds which qualifies the instrument to be one of the best tools for GSR analysis.

At the moment we are still performing laboratory testing involving typical number of three-component and two-component particles on the back of a shooter hand immediately after shooting. Because of a rapid loss of GSR particles with time elapsed after shooting, we have doubts about taping the hands only. It seems that taping hand only is adequate just for suicide cases. On the other hand taping hair, moustaches and beard didn't appear to be of much help because of big spread in results. Our last results showed that perhaps it would be a good idea to combine two different sampling and analytical methods (GSR and modified Gonzales test).

Another field of application of ESEM is as a comparative microscope. Acording to our specifications Philips made a special ballistic stage that can accept two cases (shells) or bullets. By use of Philips' software "Picture in Picture" we managed to use single SEM as a comparative electron microscope. Manual matching works wery well and it is reasonable in cases of matching of very small mechanical traces (typically toolmarks on wires) or in cases of traces on curved surfaces (typically strong deformed bullets).

KEY WORDS: SEM; GSR.

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Since late 1998, SEM has been installed in Forensic Centre, Zagreb, Croatia. Philips' Environmental Scanning Electron Microscope (ESEM) XL 30 with EDAX EDX-detector and Philips' GSR NT v. 3.2 software is dedicated primarily for investigation of gunshot residue particles (GSR). After initial technical problems real laboratory work started in May, 2000. Instrument spectral resolution is about 142 eV at amplification time of $10 \mu s$ which qualifies the instrument to be one of the best tools for GSR analysis. Typical time of analysis ranges between 60 and 170 min for scanned area 7×7 mm, minimum size of a particle set to 0.5 micron and minimum 2×2 pixels per particle setting.

Automated GSR analysis has been performed and controlled by Philips' GSR NT v. 3.2 software working under Win NT v. 4 operating system. Results were manually transferred to MS Access databases or Excel spreadsheets for additional statistical analysis.

Double-sided carbon tapes, 12.5 mm in diameter mounted on aluminium stub, has been used with ESEM instrument for automated GSR analysis. Use of any other type of adhesive tape, electrically conductive or non-conductive is also possible. On other types of samples which are typically non-conductive or even organic (like leafs, branches, garment, fragments of a walls, wooden doors and so on) we were performing manual search of GSR particles (combined with automated GSR analysis on tapes). There is no need for additional preparation of tape or sample surface (coating). Working in ESEM mode, it is possible to find GSR particles or small fragments of bullet (brass jacket or lead core) on those samples without coating.

GSR NT v. 3.2 software automatically compare X-ray spectrum of each found particle with X-ray spectra of GSR particles found earlier to be characteristic for particular ammunition and program is automatically putting found particle in one of predefined custom made classes. It is possible to define up to 35 different classes and in each class is possible to define up to 5 elements with their statistical weights (approximate quantities). Accuracy of motorised stage of a microscope allows us to easily revisit particular particle on a stub until the moment we take out the stub from chamber. Manual revisit and checking of three-component GSR particles found by software showed that roughly 50% of those particles should be abandoned. When the system finds one big three-component GSR particle very often it claims that different parts of the big particle are lot of small particles. In order to get relevant results this must be taken into account and that big particle must be counted as only one three-component GSR particle.

At the moment we are still performing laboratory testing involving typical number of three-component and two-component particles on the back of a shooter hand immediately after shooting. We want to find distribution of GSR particles (unique and indicative) on the back of a shooter hand. Back of the shooter hand was taped with five double-sided carbon tapes 12.5 mm in diameter. Prior to shooting barrel of a pistol was cleaned and shooter's hand carefully washed and dried. "GLOCK", M-17, calibre 9×19 mm and the ammunition "PPU-91" calibre 9×19 mm with FMJ round was used. It was found out that number of GSR particles on the back of the shooter right hand (for total of five stubs) immediately after shooting one shot inside shooting room is as follows:

- $-$ total three-component (unique) = 129.2 ± 33.4 ;
- total three-component (unique) confirmed = 57.6 ± 28.1 ;
- $-$ total two-component (indicative) = 137.2 ± 52.8 .

Small number of outdoors shooting was also performed but no substantial difference to indoors shooting was found.

Fig. 1. Typical three-component GSR particle.

Normally, we are using 12.5 mm in diameter double-sided carbon tapes mounted on aluminium stub, but because we have ESEM instrument automated GSR analysis is also possible on any tape, electrically conductive or non-conductive. There is no need to prepare (coat) tape. Besides automated GSR analysis on tapes we are performing manual search of GSR particles on different types of samples that are typically organic or non-conductive like leaf, branches, garment, and fragments of a wall… In all those cases, working in ESEM mode, it is possible to find GSR particles or small fragments of bullet (brass jacket or lead core) on those samples without coating.

Because of a rapid loss of GSR particles with time elapsed after shooting, we have doubts about taping the hands only. It seems that taping hand only is adequate just for suicide cases. On the other hand taping hair, moustaches and beard didn't appear to be of much help because of big spread in results. We want to find out the best lifting technique and we want to co-operate with the forensic laboratories dealing the same problems. Our last results showed that perhaps it would be a good idea to combine two different sampling and analytical methods (GSR and modified Gonzales test). Currently, method that is still in normal use in Croatia is modified Gonzales test. Instead of paraffin we are making gloves of silicon paste that dentist use for imprints. Silicon gloves are treated by difenilamin in order to prove nitrates (constituent of gunpowder). We know this method is proved to be non-specific and has lot of false results, but we also know that lifting efficiency of this method is high (much better than tape lift method) and we know it is possible to find partially burnt gunpowder particles on hands even days after shooting. On the other hand, GSR analysis is highly specific method and even though lifting efficiency of tape lift method is not high, this method is a must. So, at the moment we are experimenting with the combination of those methods. First, we tape interesting parts of a hand with tapes and after that we make the silicon glove of the whole hand. Tapes are analysed with SEM-EDX and silicon gloves are analysed by use of coloured reaction of nitrates and difenilamin. It is clear that (because low lifting efficiency of the tape) we can detect partially burnt gunpowder particles even in the areas on the hand, which already had been taped. On the one hand this lack of lifting efficiency of tapes is disadvantage, but on the other hand it can be advantage because it allows us to combine two different methods and perhaps give us a method to work in a real casework. We are also experimenting with lifting technique that use big adhesive tape in a shape of a hand. Most interesting part of that hand shaped tape we cut and without coating perform automated GSR particle analysis, while rest of the tape we treat with difenilamin. With this method it is possible to find out linear distribution of GSR particles along forefinger for example. With this method we are still at the beginning, but first results are promising.

Another field of application of ESEM is as a comparative microscope. Being fully aware of problems with optical comparative microscope (strong reflections of illumination light on cases and bullets, standardization of angle of light, poor depth of field) we proposed to solve those problems with SEM. We bought SEM mainly to analyse GSR particles on hands, but once we have SEM it seemed a good idea to try to broaden its application. Acording to our specifications Philips made a special ballistic stage for ESEM that can accept two cases (shells) or bullets. It is possible to translate them in X, Y and Z direction as well as rotate and tilt. By use of Philips' software "Picture in Picture" we managed to use single SEM as a comparative electron microscope.

Picture of an interesting part on a case from scene of crime is "frozen" (put to hard disk of a computer) and that "frozen" picture is compared with live picture of appropriate part on a test fired case. Matching of individual characteristics is done with same techniques as with classical optical comparative microscope. This manual matching works wery well and it is reasonable in cases of matching of very small mechanical traces (typically toolmarks on wires) or in cases of traces on curved surfaces (typically strong deformed bullets). Of course, our pictures are not real pictures but distributions of intensity of signal (pseudo BSE) with respect of X and Y coordinates on the sample, but if we mix an image with tilted image of the same trace we could (by use of a existing software) get real 3D (X, Y, Z) of a trace and we could import that file in a database and later perform search inside that database. In this

Fig. 2. SEM as a comparative microscope.

field we are still at the beginning, but the first results are good and we are planning to explore possible application of the SEM as a comparative microscope as well as a tool for input pictures of mechanical traces of mechanisms of arms on the bullets and cases in our custom made database.