X-RAY MICROANALYSIS OF LIGHT AND STRATIFIED SAMPLES, POSSIBILITIES AND LIMITATIONS

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Modeling of physical phenomena taking place in microanalysis can show limitations as well as possibilities of the method. Experimental microanalytical data, particularly k-ratios, have been compared with calculated ones obtained by mathematical simulation. The model uses the single scattering Monte Carlo algorithm, where "Bethe cross section" is introduced for the description of inelastic events. Various ionization cross sections have been tested to show their influence on the distribution functions. Accuracy of the simulation has been tested at the boride and carbide datasets, comparing calculated k-ratios with experimental data. They have revealed the uncertainty in the mass absorption coefficients and their limitation to the quantitative analysis of very light elements. Layered samples present the most crucial test for the model. Monte Carlo calculation results are compared with experimental data and commercial analytical programs. The paper has shown that the correction program based on first principles gives comparable accuracy with analytical approaches for all cases. The progress in the quantitative analysis is conditioned by the progress in the theory as well as by the progress in the accurate experimental determination of some physical parameters.

INTRODUCTION

There are two distinct trends in the quantitative Electron Probe Microanalysis (EPMA): the analytical approach that incorporates physical knowledge, empirical findings and analytical results; and physical approach represented by the Monte Carlo simulation or by a solution of the Boltzmann equation. The analytical approach was very successful in the past, enabling microanalysists to achieve quantitative information about the elemental composition of the unknown sample. The average accuracy of the quantitative determination of the present element is around 1 % for bulk samples in favourable circumstances, except for the very light elements. The correction methods have been at first represented by the so called "ZAF" models where the matrix, the absorption and the fluorescent corrections were calculated separately. They were first so named by Philibert [21] and many of them are currently available commercially. Light element analyses have challenged new approaches to correction methods issued in the so called $\varphi(\rho z)$ method. The methods use the true distribution function $\varphi(\rho z)$ for each element [6] that corresponds to the number of X-ray photons emitted from the depth between ρz and $\rho z + d\rho z$. The knowledge of the $\varphi(\rho z)$ function is the key to the quantitative analysis. It includes the complex transport of a primary electron as well as the atomic processes leading to X-ray emission. Knowledge of $\varphi(\rho z)$ function together with the

knowledge of mass absorption coefficients leads to the complete theoretical description of interactions in the sample, important for the microanalysis. The basic shapes of the functions are chosen ab initio, in the form of quadrilateral [28], parabolic [22], gaussian [19, 3], or double gaussian [16] functions. These functions have four independent variables for each element that are calculated according to simple equations, in which empirical findings, analytical results and physical knowledge are mixed together. A fifth variable is introduced in the other model [23]. It is believed, that the ab initio form of the distribution function is flexible enough to conform all combinations of samples and analytical conditions by the averaging or weighing laws [16, 22] that, however, have poor physical backing. Results of the correction programs have been tested on the set of analytical data that had often served as a starting point for the equations coming into the correction programs, arising question about their applicability in situations not covered by the tested dataset. The programs are mostly rated by the standard deviations of ratios k(calculated)/k(experimental), but presented histograms [28, 22, 16] reveal that still some analytical situations exist where the discrepancy among reality and results of the correction programs are above five percent. That means, it is very difficult to determine the precision of the chosen method for the unknown sample. Moreover, it is often hard to say which correction method is better among many analytical programs, because their accuracy

can be different for various analytical conditions or various materials.

The Monte Carlo algorithm is well known from the mathematical point of view, and it was first applied to the microanalysis by Green [10]. The large and detailed review of the use of Monte Carlo in microanalysis was presented by Karduck and Rehbach[15]. The method has been mostly used for the determination of general trends of various parameters, such as backscattering coefficient, surface ionization or electron range. Two main types of the simulations have been identified during the evolution of the Monte Carlo method: multiple scattering model and single scattering model. The two models were well compared in a workshop at NBS [12]. It was clearly shown that the multiple scattering model cannot predict the behaviour at low electron primary energy and for high atom number, where the elastic scattering probability at large angle is high. The single scattering model has been studied extensively by Karduck and Rehbach [15], in which generation of secondary electrons has been incorporated. But it was not clear so far if the theory based on first principles is able to predict the same or comparable quantitative results as analytical methods. Several authors expressed their believe [22] that the Monte Carlo method can not achieve comparable quantitative results and can serve only as a good qualitative indicator.

Monte Carlo Simulation

The main conditions required for the algorithm have been preservation of logic consistency and physical background of the model without additional fitting or corrections to experiment. The description of the electron transport is based only on two fundamental cross sections that describe elastic and inelastic scatterings, respectively. Electron moving between the scatterings is straight, and its actual path Λ , for bulk sample, can be calculated according to

$$\Lambda = \Lambda_0 \ln R, \tag{1}$$

where Λ_0 is the mean free path, determined by the later introduced elastic and inelastic cross sections, and *R* is a random number. For a layered sample, the actual path is determined as described by Murata et al [18]. Another random number determines the type of the scattering at the end of each actual path Λ . If the elastic scattering occurs, next random number predicts the scattering element. The elastic scattering is described by the Mott cross-section, that is tabulated by Riley et al. [26]. For energies not available in the tables, we have carried out a linear interpolation between two adjacent energies or an extrapolation for energies below 1keV. The particular scattering angle is again determined by a random number. The detailed description of the procedure determining the scattering angle as well as the scattering geometry can be find, for example, in the excellent book by Reimer [25].

While using Mott cross-section for elastic scattering is straightforward and is generally accepted as the best choice, the situation for inelastic events is much more complicated. Many processes, such as volume and surface plasmon excitations, deep energy shell ionizations, or influence of secondary electrons contribute to it, but many of them are poorly known quantitatively. Moreover, introducing many processes drastically increases computing time. To find a simple and acceptable formula describing the inelastic scattering, we have assumed that the primary electron can lose only a distinct amount of energy ΔE . The energy loss is the same for each incident electron and independent of the scattering element. The idea was inspired by the plasmon processes that are dominant for energy loss in the interesting energy region. On the other hand, the inelastic cross section must be restricted by the requirement that it must give the same stopping power as the Bethe approximation. To conform to these constraints, the inelastic differential cross-section σ^{in} is

$$\sigma^{\rm in}(E, E') = \frac{B(E)}{\Delta E} \,\delta(E - E' - \Delta E) \quad , \tag{2}$$

where E, E', ΔE are the energies of the electron before and after scattering, and energy loss, respectively. B(E)is given by the Bethe energy-loss approximation [5], that for low energies (E < 6.338 J) is replaced by the formula by Rao-Sahib and Wittry [27]. The mean ionization potential J given by Berger and Seltzer [4] was used.

The introduction of "Bethe cross section" has enabled to replace a number of inelastic cross sections by one effective cross section, that covers all inelastic processes in the same way as it is done by Bethe approximation, and with the same inaccuracy. On the other hand, Bethe stopping power can be replaced in a simple way by the better one, as the progress will continue in the field. The variable ΔE in equation (2), the energy loss per inelastic scattering, is not determined inside the model, but it is considered here as a free variable. Nevertheless, a variation of ΔE over a wide energy range does not affect the distribution functions significantly as it is demonstrated at figure 1, for lower primary energy, and at figure 2, for higher primary energy. The distribution functions have been obtained by the simulation of 10 000 primary electrons. The functions are smoother with decreasing ΔE due to the higher number of inelastic events. Curve unevenness reflects the statistical noise that is higher for higher ΔE , because of lower ionization probability. We have the possibility to change the number of inelastic scattering per primary

electron (if it is not backscattered) by changing ΔE . If we assume small ΔE , i.e. the inelastic cross section is much larger than the elastic one, the calculated energy loss between two elastic events is close to that obtained from Bethe's law. When assuming larger ΔE (the inelastic cross section is smaller than the elastic one) a few elastic collisions occur between two inelastic events. The electron in question loses a higher portion of energy per inelastic event and its path between two inelastic events is longer. However, for the case of elastic scattering, the influence of the energy of the primary electron on its angular distribution is rather weak, and therefore not very



Figure 1. Distribution functions $\Phi(\rho z)$ for various ΔE - energy loss per an inelastic scattering. Tracer element is B(K α) in a silver matrix for 5 keV. The curves are results of modelling the transport of 10 000 electrons. \times - 10 ev, O - 100 ev, \bullet - 500 eV, \star 2000 eV



Figure 2. Distribution functions $\Phi(\rho z)$ for various ΔE - energy loss per an inelastic scattering. Tracer element is $Zn(K\alpha)$ in a copper matrix for 20 keV. The curves are results of modelling the transport of 10 000 electrons.

× - 10 ev, O - 100 ev, \bullet - 500 eV, \blacktriangle 2000 eV

extreme increase of ΔE changes the distribution function insignificantly. Of course, the lower the primary electron energy the smaller value of ΔE should be chosen. Figures 1 and 2 show, that even for $\Delta E = 500$ eV and primary energy $E_0 = 5$ keV, the distribution function is nearly the same as for $\Delta E = 10$ eV. Figures also stand as the confirmation of correctness of introducing the "Bethe cross section". Introducing the energy loss in the form of cross section also introduces the energy straggling in a natural way, as it is in reality.

The elastic and inelastic cross-sections fully determine the electron transport in the sample. To calculate the X-ray production, we have to complete the description of elemental processes by introducing the ionization cross section. We have tested and compared a few: Classical $((\ln u)/u)$, where u is an overvoltage; often used by correction programs for its simplicity), Mott-Massey [17], Worthington-Tomlin [29], Pouchou-Pichoir [22], Hutchins [13], and Gryzinski [11] cross-sections. Among them, the Gryzinski cross section gives the best agreement among calculated distribution functions $\Phi(\rho z)$ and the experimental ones [7]. The $\Phi(\rho z)$ functions for various ionization cross sections are given in figure 3 for light matrix and in figure 4 for heavy matrix, respectively. Experimental results were taken from Parobek and Brown [20]. The figures confirm as the best choice the Gryzinski cross section and the worse accuracy of older cross sections. It is especially interesting to compare the classical cross section, often used in pioneering times of microanalysis due to its simple integrability. Its large deviation from experiments explains its failure for light element analysis, where the correct distribution function is undoubtedly extremely important.

We have developed a correction program for quantitative analysis on the basis of the above mentioned elemental processes. The photon production Q_{α} is calculated by means of Monte Carlo procedure for each analyzed element α and for each iteration step. The Q_{α} is normalized to unit concentration after the end of each iteration procedure. The values for standards are calculated in advance and serve as a standard library later. The k_{α} -ratio for element α is thus given by simple equation

$$k_{\alpha} = \frac{c_{\alpha} Q_{\alpha}(\mathbf{c})}{c_{\alpha}^{s1} Q_{\alpha}^{s1}(\mathbf{c}^{s1})} \quad , \tag{3}$$

where the superscript "st" denotes variables related to the standard. The iteration procedure comes at once from equation 3

$$c_{\alpha}^{(n)} = k_{\alpha} c_{\alpha}^{st} \frac{Q_{\alpha}^{st}(\mathbf{c}^{st})}{Q_{\alpha}(\mathbf{c}^{(n-1)})} \quad , \tag{4}$$

where n indicates the iteration number.





Figure 3. Distribution functions for various ionization cross sections. System $Al(K\alpha)$ in Si; 10keV; experimental data taken from Parobek and Brown [20].

"Bethe cross-section" has another great The advantage for its use in the correction model. It is independent of the particular scattering element, so it is not only effective for the inelastic scattering (covering all of them), but it is also "effective" for the sample. Thanks to this property, we can calculate the probability of scattering on each element for every inelastic event. Next we calculate ionization probability for each element and result is multiplied by $exp(\mu \rho z)$, where μ is mass absorption coefficient and z is the depth of the inelastic event. This way we calculate the probability of photon detection for each analyzed line and for each inelastic event. That means, the assessment of trace element takes the same amount of computer time as any other element, what is especially advantageous for a correction program.

RESULTS AND DISCUSSION

In this section we compare the results of the correction program with experiment for bulk and stratified samples separately. It is worth to stress that all

Figure 4. Distribution functions for various ionization cross sections. System $Si(K\alpha)$ in Au; 10keV; experimental data taken from Parobek and Brown [20].

calculated results are obtained without any experimental adjustment and are based only on the elemental processes discussed above.

Bulk samples

The program was first tested on bulk samples prepared by NIST [8]. The results obtained by Monte Carlo algorithm were fully comparable with the analytical approaches used by various authors. In the next step, we have tested our model for more sensitive situations. As an excellent test has served Bastin and Heiligers borides' [1] and carbides' [2] datasets, which have been also used by many other authors to demonstrate the validity and accuracy of their approaches. The error histograms [9] reveal for the Monte Carlo model, that the ratios k(calculated)/k(experimental) have for accompanied elements the same scattering around unity as for any other type of samples. The standard deviation of the ratio k_{cal}/k_{exp} for accompanied elements is 2.14 %, and the average is 1.00 in borides; standard deviation of the ratio k_{cal}/k_{exp} is 1.82 %, and average is 0.99 in carbides. This is an indication that the electron transport was described in a satisfactory way.



Figure 5. Standard deviations for boron and carbon increase with increasing energy of primary electrons. Standard deviations for accompanying elements in carbons are decreasing with increasing energy. Standard deviation for accompanied elements in borides is highest for lowest energy, then decreasing with energy up to 15 keV and then slowly increasing.

O - accompanied elements in carbides, \bullet - carbon, Δ - accompanied elements in borides, \blacktriangle - boron



Figure 6. Standard deviations of carbon in carbides and boron in borides increase with increasing absorption coefficients.

● - carbon, ▲ - boron

The physics used for the description of the electron transport is, in general, more adequate for the higher electron energy. The dependence of standard deviations on the primary energy is shown in the figure 5. The standard deviation for accompanied elements decreases with increasing energy, confirming the above mentioned general physical trends, while the deviations of carbon and boron increase. This is a clear indication of the inaccuracies in mass absorption coefficients.

The standard deviation is 5.90 % and average is 1.04 for boron in borides; and 4.00 % and 0.99 for carbon in carbides, respectively. The main reason for this discrepancy is, as we believe, in the uncertainty of absorption coefficients. This is confirmed by the results of carbon boride analyses where the standard deviation and average are for boron 1.17 % and 1.01, respectively. The figure 6 documents the strong dependence of standard deviation on the mass absorption coefficient. The large improvement of the results could be also achieved by adjusting the mass absorption coefficients (we have used ones proposed by Bastin and Heiligers), so that the calculated values were as close as possible to the experimental ones. However, we do not think it is the best way how to determine these coefficients, although systematic deviations of calculated results from experimental ones were obtained for some elements. As an example, standard deviation 5.5 % and average 0.92 have been obtained for tungsten carbide, and Bastin and Heiligers' mass absorption coefficients. Replacing mass absorption coefficients according Merlet [16], we have got standard deviation 1.3 % and average 1.01. The examples demonstrate, how extremely careful we have to be to declare absorption coefficients determined by means of a fitting the microanalytical data as the right ones. Very different coefficients are the best choice for different programs, but to decide which one is physically correct is impossible to do in this way.

Layered samples

Probably the most crucial test of the accuracy of the Monte Carlo correction model is its use for the layered

Calculation of k-ratios for carbon and boron differs from the calculation of accompanied elements only by the ionization probability (the overvoltage is much higher), and in the extreme high absorption coefficient. The higher absorption coefficient the more important is the accurate determination of the surface ionization, and the less important is the integral of the $\varphi(pz)$ function. On the contrary, the integral of the $\varphi(pz)$ function plays role only for the very low absorption coefficient. That means, the accuracy of a k-ratio of a very light element depends considerably on the good description of the initial course of the $\varphi(pz)$ function and on the precise value of the mass absorption coefficient.

system, where the inaccurate form of the $\varphi(\rho z)$ is not compensated so much by reference standards as it is for bulk samples.



Figure 7. K-ratio of the Au(M α) line on Si substrate; 20 keV. × - experiment, ∇ - MC, O - STRATA



Figure 8. K-ratio of the Al(K α) line on Au substrate; 20 keV. \times - experiment, ∇ - MC, O - STRATA

We have prepared thin layers of gold on platinum, gold on silicon, aluminium on silicon and aluminium on gold of various thickness by magnetron sputtering. The details of the preparation are described elsewhere [9]. The layers and substrates were chosen so that to cover various combinations of the light/heavy layer on the light/heavy substrate. Intensities coming from the layer were measured for 10 keV, 20 keV and 30 keV. Experimental k-ratio were determined by measuring pure bulk standards of gold and aluminium. The k-ratio is in the case of layered sample defined as the ratio of the intensity of a thin layer to the intensity of the bulk sample.

The Monte Carlo algorithm described above has been used to calculate k-ratios for layered samples. Calculated values were compared with the experimental ones. Moreover, we have used the commercial analytical program STRATA to calculate k-ratios, too. All results have been summarized into figures, where k-ratios versus thickness of thin films are shown. The case of the heavy element (Au) on the light substrate (Si) is presented at the figure 7. The Monte Carlo as well as STRATA give higher values of k-ratios comparing with experimental points; difference between Monte Carlo and STRATA is small. Our approach is comparable with the analytical one for very thin layer, results coming from the analytical approach are more accurate for thicker layer. The situation for Al/Au (a light element on the heavy substrate) at the figure 8 is very similar but the difference between Monte Carlo and STRATA is more pronounced. The accuracy of Monte Carlo is better with increasing electron energy and for some cases overcomes the STRATA results [9].

The maximum deviation between Monte Carlo and experiment is about 25%, independent of the type of film and substrate. The deviation comes above all from the modeling of the elementary processes as is indicated by its energetic dependence.

CONCLUSION

The Monte Carlo model is based on two elemental processes describing the electron transport, and the ionization cross section determining the photon production. The angular parts of inelastic events are neglected for the sake of simplicity. This fact limits the model at the lower side of primary energy. The introduction of effective "Bethe cross section" has enabled to use the model for a routine quantitative analysis. As an example, calculation takes for one sample only two minutes by using common PC. Although the model looks very simple, it is able to produce quantitative results fully comparable with the analytical approaches, as for bulk samples as well as for layered ones.

The simulation has proved that the analytical approaches do not give better results than physically based ones, so they reflect the state of the art in theory. Without a better theory in the description of elemental process, especially in description of inelastic ionization cross section and mass absorption coefficients every new correction method will bring only another new program to decide how to evaluate the experimental data. The question arises, if the effort invested in the creation of many analytical programs should not be spent for the better description of elemental processes and measurement of some uncertain experimental coefficients. The weakest point of the theory appears to be the ionization cross section; experiment has its great debt in the precise determination of mass absorption coefficients for very light elements.

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RTG MIKROANALÝZA LEHKÝCH PRVKŮ A VRSTEVNATÝCH VZORKŮ, JEJÍ MOŽNOSTI A HRANICE

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Matematické modelování fyzikálních jevů probíhajících při mikroanalýze vzorků může prokázat nedostatky, ale i přednosti a další možnosti této metody. Experimentální data, získaná na mikroanalyzátoru, zejména tzv. k-hodnoty, byly porovnány s vypočtenými k-hodnotami, získanými matematickým modelováním pronikání elektronů pod povrch vzorku a fyzikálních procesů s tím souvisejících.

V našem modelu je použit Monte Carlo algoritmus pro jednoduchý rozptyl elektronu, kde se popisuje nepružná srážka elektronu pomocí Betheho účinného průřezu. Byly použity různé hodnoty pro ionizační účinný průřez a zhodnocen jejich vliv na tvar distribuční křivky pro vznik charakteristického rtg. záření v různých hloubkách pod povrchem vzorku. Přesnost a správnost modelování byla ověřena na vzorcích karbidů a boridů, jejichž databáze jsme měli k dispozici ke srovnání vypočtených k-hodnot s experimentálně určenými k-hodnotami. Ukázalo se, že nepřesnost v určení hmotnostních zeslabovacích koeficientů pro charakteristické rtg. záření je hlavní příčinou omezující správnost kvantitativní analýzy, zejména pro velmi lehké prvky. Kvantitativní mikroanalýza vrstevnatých vzorků představuje zároveň kritický test pro použitý model. Výsledky našich výpočtů metodou Monte Carlo jsou porovnány se známými experimentálními hodnotami a jinými výpočty, pomocí komerčně dostupných programů

Tento článek ukazuje, že korekční program založený na metodě Monte Carlo, která simuluje všechny fyzikální děje, doprovázející pronikání primárních elektronů do pevné látky, dává srovnatelné analytické výsledky ve všech případech analýz. Lepší kvantitativní výsledky korekčních metod, kterých je dnes celá řada, jsou podmíněny lepším teoretickým popisem jednotlivých fyzikálních dějů, které elektron podstupuje a také lepším experimentálním určením fyzikálních konstant a koeficientů, které se ve výpočtech objevují. Nelze tedy očekávat výrazné zlepšení kvantitativní rtg. mikroanalýzy a každý nový korekční program bude pouze jedním z mnoha, které dávají přibližně stejné výsledky. Výrazný pokrok je jednoznačně spjat s pokrokem experimentální a teoretické fyziky v této oblasti.