

BOOK OF TUTORIALS AND ABSTRACTS



European Microbeam Analysis Society

EMAS 2009

11th

EUROPEAN WORKSHOP

on

MODERN DEVELOPMENTS

AND

APPLICATIONS

IN

MICROBEAM ANALYSIS

10 to 14 May 2009

at the

Hotel Spa Faltom

Gdynia/Rumia, Gdansk, Poland

Organized in collaboration with
Silesian University of Technology
Polish Society for Microscopy (PTMi)
Polish Academy of Sciences:
Committee of Materials Science, Institute of Physics,
Institute of Materials Science and Metallurgy



MICROANALYSIS IN THE METALS RECYCLING INDUSTRY

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Eddy Boydens obtained the degree of metallurgical engineer at the Catholic University of Leuven in 1980 and continued there on a project of powder injection moulding. He started in 1984 the scanning electron microscopy lab within the central laboratory of Metallurgie Hoboken-Overpelt in Olen. From 1993 on, he worked at the Catholic University of Louvain-la-Neuve on the microstructural characterisation of incoming and finished products and of worn refractory bricks of Umicore using the microprobe at this university. This work continued from 2004 on at the Catholic University of Leuven. Since 2007 he is Manager Analytical Laboratory in the newly organized central laboratory “Analytical Competence Centre” of Umicore in Olen where he is responsible for the group of the Physical Characterisation.

His area of expertise is the microstructural characterisation of primary materials, secondary materials, and specially generated products such as for example slags, alloys, fumes and residues of industrially applied or tested pyrometallurgical or hydrometallurgical processes. The main in-house techniques used for these investigations are stereomicroscopy, optical microscopy (under ordinary as well as under polarized light), scanning electron microscopy, microanalysis (EDS as well as WDS on a microprobe with five WDS spectrometers), and X-ray powder diffraction.

1. ABSTRACT

Microanalyses are an indispensable tool for helping the metals recycling industry to recover as efficiently as possible all metals brought together under the form of used materials or under the form of industrial waste generated during the precedent recovery of some basic elements.

Gathering all kind of materials for recycling and its following transport may be easy, the separation into valuable elements ready for re-use is more complicated. This separation asks for profound knowledge of the generated new compounds out of the incoming materials in order to establish the most suitable processes to recover the individual elements as much and as pure as possible.

Microanalyses are done to know which elements are combined together and in which concentration they are incorporated in the new compounds. Also the amount of these compounds can be calculated. X-ray diffraction analyses reveal also the present compounds but this technique is only a minor tool in helping the treatment of the microanalytical results. Optical microscope pictures in colour, or scanning electron microscope pictures in black and white, help also a lot but do not result in profound knowledge of the elemental distribution. Some examples and difficulties in microanalysis encountered are given for the non-ferrous industry because more than in the ferrous industry a much larger selection of elements from the table of Mendeleev is encountered. Therefore, the use of a microprobe working with wavelength-dispersive analyses to overcome the problems of energy-dispersive analyses, usually applied by means of scanning electron microscopes, is obligatory. Attention is also drawn to a background calculating technique based on the mean atomic number to enhance the analytical speed of the slow wavelength-dispersive analyses.

2. INTRODUCTION

Intensive recycling of metals already exists for many years. Waste from used metal-bearing materials together with waste from the production of these materials is treated in view of recovering these metals for making again new materials out of it.

Ores on the contrary have been worked for centuries to recover the necessary amounts of wanted elements. Recycling, however, has gained more importance in Europe due to the increased collection of used materials, to the higher costs of getting ores into Europe, and as a contribution for making sustainable materials. Moreover, large amounts of special metals are applied in particular applications and their recovery is mandatory in order to offset their demand as they are not sufficiently available in nature.

Several factories contribute to an efficient recovery of elements as much as possible and as long as their processes can handle the bought combination of elements and as long as the recovery generates a profit.

Many decades of treatment of raw non-ferrous metal-bearing products showed Umicore the way to a factory with quite some experience in handling quite diverse products in order to recover their constituent elements. The company focuses nowadays on applications where it knows its expertise in materials science, chemistry and metallurgy can make a real difference [1]. So, some of these elements are brought together in compounds or alloys for further processing in one or another application. Consequently, the added value of the produced materials can be increased. For some products, where no metals can be economically recovered anymore, a stock is built up till a solution is found, or an application or a cheaper recovery process becomes available, or an increase in the price of the unrecovered elements again warrants their recovery.

The separation of all the imported elements has forced the company to use microanalysis as a strategic analytical tool, as is done too when recovering elements from minerals [2], in order to help research in finding or developing appropriate separation processes and to help the production when opposed with a decreasing recovery efficiency. Because Umicore decided some years ago to close the loop of the use of metals, microanalyses gained even more importance. Recovery processes are available and constantly developed to finalize a re-use of home made pure metals or products.

However, in order to realize efficient microanalyses, more common techniques such as X-ray diffraction analyses, optical microscopy, as well as electron backscattered imaging are needed too.

3. X-RAY DIFFRACTION ANALYSES

When separating elements, the compounds enclosing these elements should be known. One of the most direct techniques to determine the compounds present in a given product is powder X-ray diffraction analysis. The material to be analyzed has to be available under the form of a fine-sized powder or, if not, grinded till all grains have sub-sieve dimensions ($< 45 \mu\text{m}$). A flattened area of the sample is hit by an incident X-ray beam. The characteristic pattern of the X-rays reflected by the crystalline structure of each compound is used to identify the compound constituting the crystalline material hit by the incident X-ray beam. Reference patterns for several thousands of compounds are found in a database.

When only one compound is present, X-ray diffraction analysis is a beautiful technique for the identification of the given product. An eventual change in the characteristic pattern indicates,

moreover, a change of the compound composition against the data from the database so that it even tells something about its purity. When, however, many compounds are present together, serious difficulties can be encountered when trying to identify all compounds:

- each compound can be of another composition than the one stated in the database resulting in many shifted patterns;
- crystalline structures of higher symmetry diffract more intensively, making their detection more obvious;
- overlaps of patterns can result in a never ending interpretation, especially when each pattern is shifted;
- the most suitable X-ray beam cannot always be used for the diffraction analyses. Using constantly a copper anode to create an incident beam of copper X-rays, will result in low diffraction signal in cobalt and in iron rich materials.

Microanalyses are needed to overcome these problematic cases.

From the many difficulties encountered in the interpretation of X-ray diffraction patterns four examples are given:

- Fig. 1: a residue contains CaWO_4 but also some molybdenum. The characteristic pattern of CaMoO_4 is unfortunately located at the same position as that of CaWO_4 , and the presence of Mo can, moreover, be completely dissolved into the crystal structure of the CaWO_4 compound. Microanalyses are needed in order to confirm the presence of CaMoO_4 grains next to CaWO_4 grains, or to confirm the dissolution of the Mo present in the material in the CaWO_4 grains.
- Fig. 2: a cobalt-carbon alloy containing some other alloying elements is investigated for a check of the formed intermetallic compounds. The diffraction pattern is mainly built up by the characteristic pattern of the minor graphite phase representing only 10 % of the carbon present in the alloy. The reflection of the X-rays on the cobalt metal phase and on the intermetallic compound(s?) is not much higher than the background.
- Fig. 3: only two compounds are detected in a Ca-Al-Si slag: åkermanite $\text{Ca}_2\text{MgSi}_2\text{O}_7$ and diopside $\text{MgCaSi}_2\text{O}_6$. Impurities like Na, Fe, Al, Zn, Mn being present in higher concentrations than the magnesium content are not found back and can consequently be major constituting elements of the formed silicate phases instead of magnesium but also instead of calcium or silicon.
- Fig. 4: a lead-bearing residue contains about 56 % Pb and contains surely PbSO_4 . If all lead is present under the form of PbSO_4 , a maximum of 82 % PbSO_4 is expected resulting in minimum 18 % other compounds. The diffraction pattern of this residue reveals a lot of peaks mainly due to this PbSO_4 . It is almost impossible to detect the composition of the other grains present in the residue by treating the few peaks left.

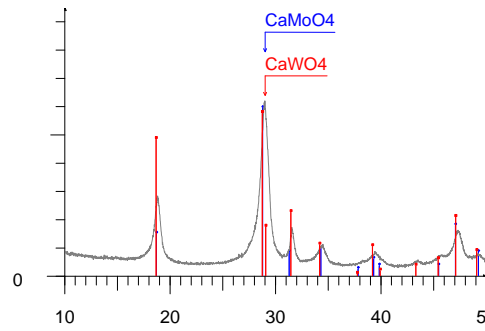


Figure 1. X-ray diffraction pattern of a calcium tungstate bearing residue.

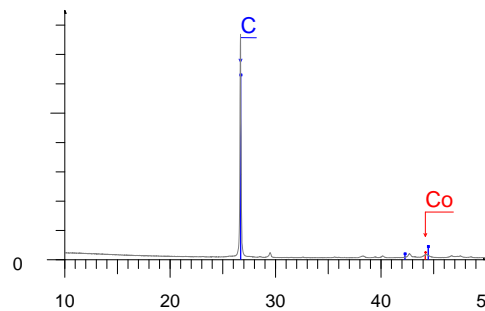


Figure 2. X-ray diffraction pattern of a cobalt-carbon alloy.

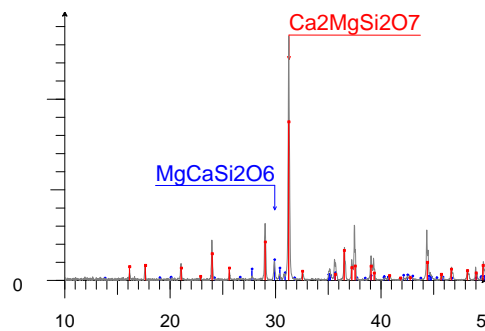


Figure 3. X-ray diffraction pattern of a Ca-Al-Si slag.

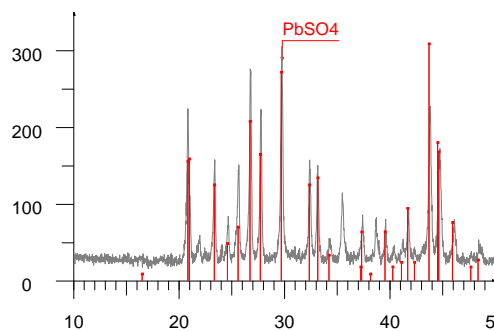


Figure 4. X-ray diffraction pattern of a lead sulphate bearing residue.

4. OPTICAL MICROSCOPY AND SCANNING ELECTRON MICROSCOPY

Polished cross-sections made of the diverse materials reveal more clearly the presence of the different compounds that constitute the investigated matter, their size and their bonding. Samples as such or after breaking or cutting the material have, however, to be embedded in resin, grinded and finally polished. Once the polished surface is put perpendicularly to an incident light beam, a preliminary identification of the present compounds can be done. The difference in the reflectivity of the incident light and the reflected colours are used. Moreover, the different behaviour under polarized light can be observed. Even the amount and nature of grooves due to the preceding grinding and polishing preparation already give some indications about the kind of material. Quite some knowledge is summarized for sulphidic and oxidic minerals [3] but mankind has made so many synthetic compounds that own experience has to be built up using indispensable microanalyses.

The produced cross-sections can be observed in a scanning electron microscope too. Because the samples are polished, most likely no secondary electron images are made but backscattered electron images. The differences in colour from the optical microscope are substituted by differences in intensities of these backscattered electrons. Heavier elements reflect more electrons resulting in bright zones where they are present. Lighter elements reflect less and cause the appearance of darker zones. Problems arise, however, when lighter and heavier elements are combined into complex compositions of both types, enhanced by the content of some intermediate elements too as illustrated by the next two examples:

- Figs. 5a and b: a sulphidic ore shows in optical microscopy at least seven different phases whereas in backscattered electron image only five phases are clearly detected.
- Figs. 6a and b: a sulphidic product made in an industrial process, called matte, is showing quite other colours than the sulphidic ore from Fig. 5a due to the other types of compounds present. But also here, at least seven different phases including some oxides are seen while in the backscattered electron image only five phases are clearly detected.

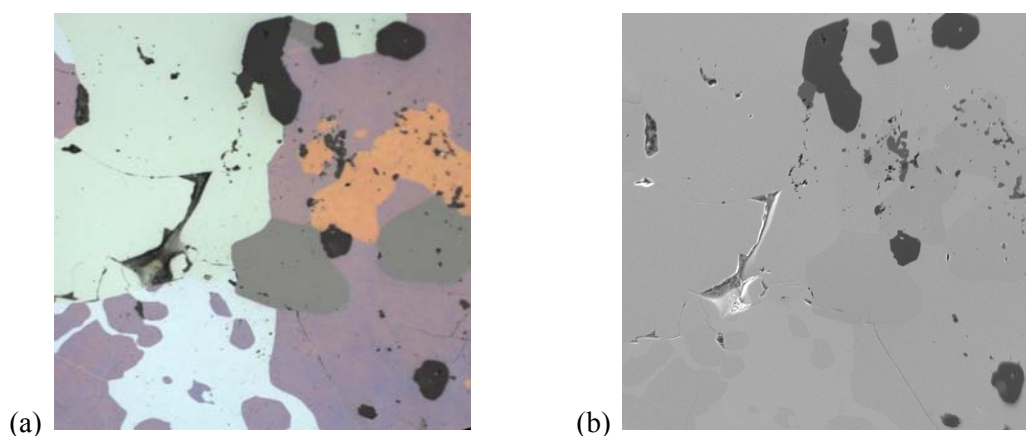


Figure 5. a) Optical micrograph of the constituting phases in a sulphidic ore. b) Back scattered electron image of the same zone as in (a).

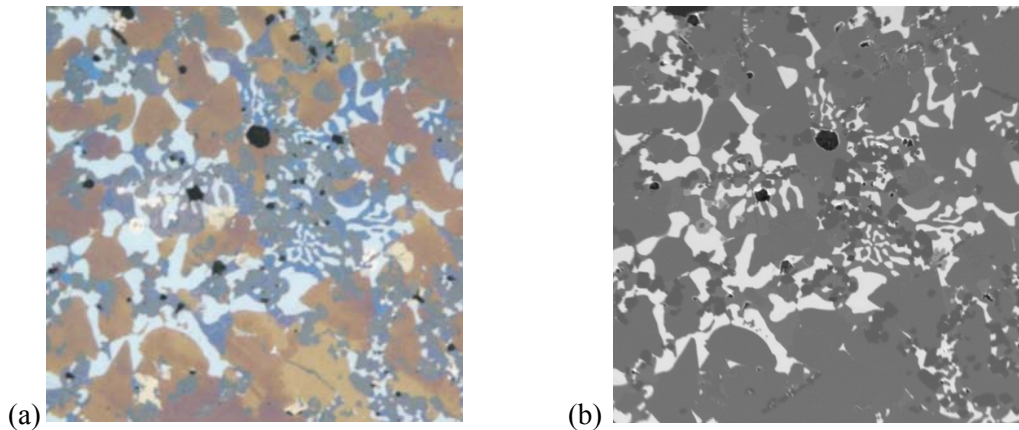


Figure 6. a) Optical micrograph of the constituting phases in a matte. b) Back scattered electron image of the same zone as in (a).

A combination of optical micrographs and backscattered electron images is in many cases a must in order to realize efficient microanalyses in the scanning electron microscope or in the microprobe.

5. MICROANALYSES EDS-WDS

Electrons interact with the atoms of the spot hit in the cross-section in the scanning electron microscope. They generate characteristic X-rays that can be detected in order to establish the composition of the investigated spot. Consequently, the several compounds present can be distinguished not only by their different reflectivity as seen under the optical microscope or as seen by the intensity of the backscattered electrons, but also by their different composition.

Two systems are available: an energy-dispersive system (EDS) or a wavelength-dispersive system (WDS). Both have advantages and disadvantages that are mentioned in literature [4, 5]. When dealing with microanalyses in the non-ferrous metals recycling industry, however, the use of a microprobe is mandatory instead of a scanning electron microscope with EDS or even with one WDS spectrometer mounted for various reasons:

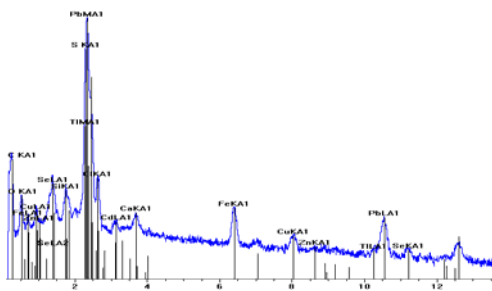
- Many elements in one single phase result in difficulties to calculate the background in EDS spectra and result in overlaps of several elements leading to incorrect results. WDS provides better peak resolution and a better peak-to-background ratio leading to more precise analyses, though a lot of interferences can appear.
- Many elements lead to long, time consuming, WDS analyses if only one spectrometer is attached. A microprobe is equipped with more than three spectrometers, leaving space for an EDS that is still obligatory as a preliminary check of the elements present in each phase.
- EDS results are most of the time normalized and obtained standardless. In WDS, on the other hand, the use of standards is forced and hence missing elements, another oxidation

state of an element than stoichiometrically attributed, or phase porosity can be rather quickly detected.

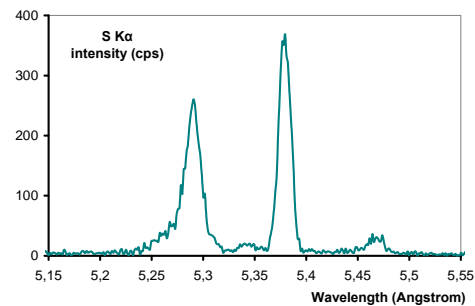
- A follow up of the sample current in a reference sample is essentially foreseen in a microprobe in order to work under reference conditions when dealing with standards.
- The acquisition time for a more or less useful EDS spectrum detecting the minor elements in a phase takes as many minutes as one single spot analysis with WDS when using five spectrometers.
- The search for a particular element is possible as an X-ray intensity meter coupled to an audible signal is foreseen in a microprobe.

Two examples of better resolution are given:

- Figs. 7a and b: the coexistence of Tl with Pb and S is difficult to prove with EDS whilst this is clear in a WDS scan as the peaks at both sides of the S K α peak at 5.37 Å show: the large Pb M α peak on the left at 5.29 Å, and the small Tl M α at 5.46 Å.
- Figs. 8a and b: iron is difficult to detect in an intermetallic Al-Mn-Fe phase by EDS due to the intense Mn K β peak but is clearly present as shown by a WDS scan at 1.94 Å, the analytical Fe K α position, next to the Mn K β peak at 1.91 Å.

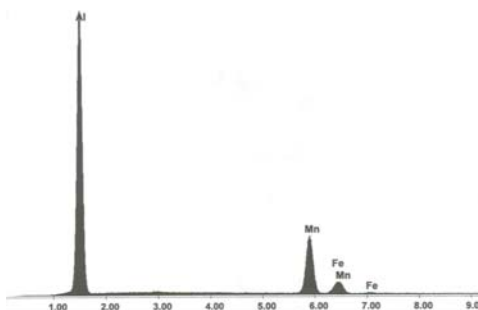


(a)

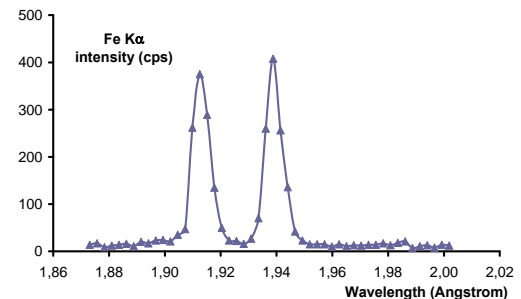


(b)

Figure 7. a) EDS spectrum of a grain concentrating Pb and S. b) WDS spectrum of the same grain as in (a) at the S K α position.



(a)



(b)

Figure 8. a) EDS spectrum of an intermetallic Al-Mn-Fe phase. b) WDS spectrum of the same grain as in (a) at the Fe K α position.

6. AREA ANALYSES BY EDS

It is clear that microanalyses are only correct when dealing with spot analyses because in order to establish the composition all correction calculations from an acquired spectrum are based on a homogeneous composition for each hit spot.

As EDS analyses are, however, so simple and give quick results, the enthusiasm to apply it for analyzing a complete area of the polished sample is high. However, this treatment results only in a determined composition close to the real total composition of the analyzed area if the microstructure is very fine as homogeneity is approximated. Moreover, scanning an area is in fact looking for an area/volume distribution and is only equal to a weight distribution if the densities of all analyzed phases are the same. Especially in the non-ferrous industry where oxide phases with densities from 2 to 5 g/cm³, sulphides with densities from 5 to 7 g/cm³, and metallic phases with densities from 7 up to 18 g/cm³, are often found together in one area of the polished sample, area analyses are out of the question.

Fig. 9a shows the case of two phases in a Zn-Si alloy: the Zn-phase and the Si-phase. Both are quite pure and hence the acquired counts of the X-rays detected in the shown area (Fig. 9c) are the sum of the X-ray counts detected in the Zn-phase and the X-ray counts detected in the Si-phase. When calculating the composition out of the spectrum acquired during scanning of the total area, the programme unfortunately takes into account the normal correction factors for this mixture of detected Zn and detected Si, though both elements are in reality everywhere well separated. A resulting composition of 48.6 wt% Zn and 51.4 wt% Si is found. However, according to a treatment of the image (Fig. 9b), the investigated area reveals: 42.4 % green corresponding to the Zn-phase, 51.9 % blue corresponding to the Si-phase, and 5.7 % black corresponding to pores and borders. After normalisation, following area or volume percentages are obtained: 45.0 vol% Zn and 55.0 vol% Si. Taking into account the density of 7.1 g/cm³ for Zn and 2.3 g/cm³ for Si, a weight percentage composition can be calculated: 71.6 wt% Zn and 28.4 wt% Si. This result is close to the real composition and is quite different from the trial area analysis by means of an EDS scanning (48.6 wt% Zn and 51.4 wt% Si).

7. BACKGROUND CALCULATION FOR WDS

Background calculations are well developed for EDS analyses and are incorporated into the software for calculating the composition from the acquired spectra. The entire spectrum is treated in one single operation, whatever the different peaks present. When many peaks appear as in samples in the metals recycling industry, these calculations encounter difficulties to fit a background to the acquired spectrum. This is also one of the reasons that WDS has to be used when the composition of such phases has to be determined.

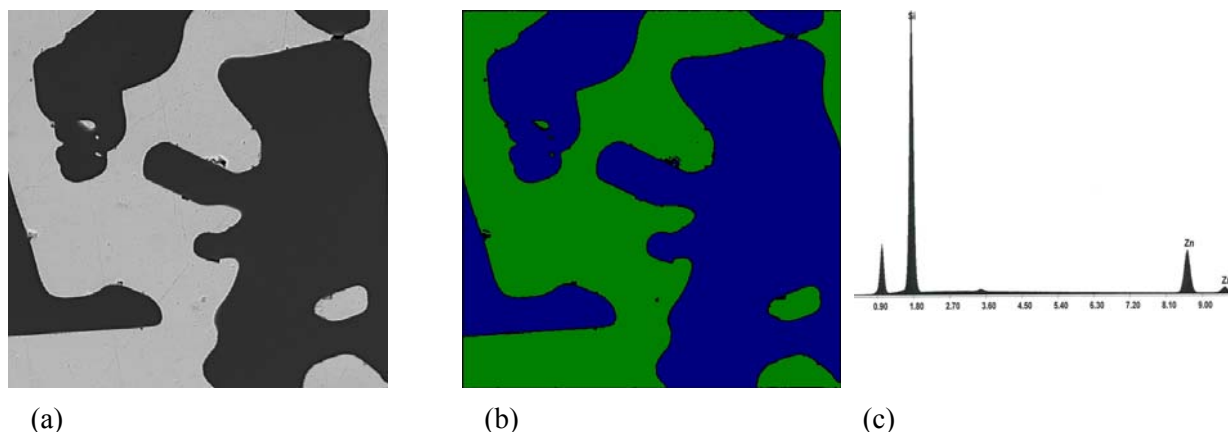


Figure 9. a) Back scattered electron image of a Zn-Si alloy. b) Treated image of the same zone as in (a). c) EDS spectrum of the area as shown on (a).

For WDS analyses, however, the background of continuum X-rays has to be calculated separately for each analytical peak out of measurement at the left and right side of these analytical peak positions. This leads to long acquisition times because the time of analyses triples. In practice, half of the time chosen for counting at the analytical peak position is used for the left and right side measurement respectively, so that the analysis time only doubles. Nevertheless, peaks from other elements can still contribute to a background increase at this left or right position making a check of the left and right position by wavelength scanning around each analytical peak for each standard and each unknown phase vital before starting the analytical programme. In conclusion, a gigantic operator task and a huge displacement effort of the spectrometers are asked for.

Luckily, a method based on the fact that the continuum X-ray background is a function of the mean atomic number has been further developed by Donovan and Tingle [6] for calculating the unknown background differently without these many supplementary WDS measurements. The Mean Atomic Number - MAN - for a product composed of different elements, i , with content, $\text{wt}\%i$, and atomic number, Z_i , is defined as:

$$\text{MAN} = \Sigma (\text{wt}\%i \times Z_i) / 100$$

For the compound SnO_2 (78.8 % Sn and 21.2 % O) with $Z = 50$ for Sn and $Z = 8$ for O, this MAN definition results in: $\text{MAN}_{\text{SnO}_2} = (78.8 \times 50 + 21.2 \times 8) / 100 = 41.9$. For the element Sn (100.0 % Sn) with $Z = 50$ for Sn: $\text{MAN}_{\text{Sn}} = (100.0 \times 50) / 100 = 50.0$.

The software programme *Probe for Windows* [7] applying this correction and developed by Donovan is since more than 15 years used with much satisfaction. The method fails however where concentrations of less than 0.1 % have to be more accurately analyzed. Long time

acquisition can help here for some part but the left and right side measurements are still the best way to determine the background when investigating very low concentrations.

X-rays are acquired on the analytical peaks of the characteristic X-ray wavelength for each analyzed element on different pure elements or simple compounds of well known composition. Compounds containing many elements are avoided, as each element present can cause many peaks, and consequently have to be eliminated for the knowledge of the continuum X-ray background. Their composition should, moreover, be well known so that their MAN can be calculated precisely. These samples are further called “MAN standards”. The intensities of the X-rays are put in a graph for each analyzed element (for example Fe $K\alpha$ in Fig. 10a and Si $K\alpha$ in Fig. 10b) and by linear or other correlation the background can be calculated for any other phase with a known MAN. It is obvious that the X-ray intensities for a particular element measured on the pure element itself are not put in this figure neither are the X-rays measured on all compounds that contain this specific element. Hence in Fig. 10a the signal for Fe $K\alpha$ measured on the MAN standard “pure Fe with MAN = 26” is not added but the signal for Si $K\alpha$ measured on this same pure Fe MAN standard is used in Fig. 10b for the background calculation at the Si $K\alpha$ analytical peak. Here, pure Si (MAN = 14) was not used at all.

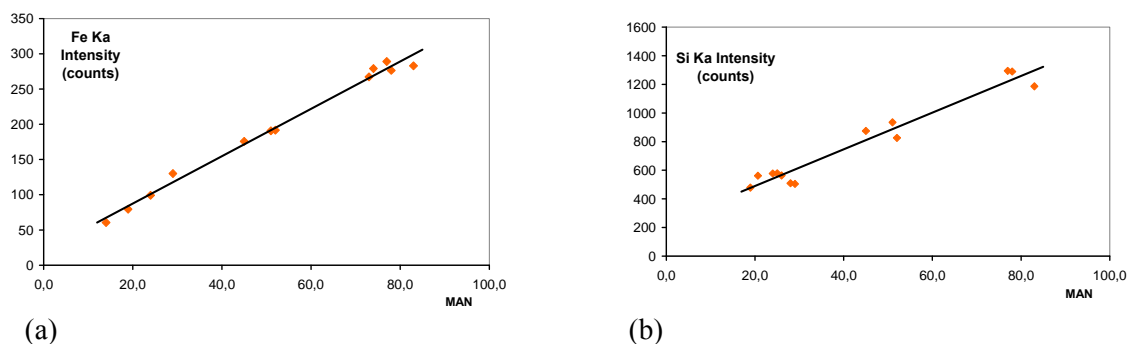


Figure 10. a) X-ray counts for the analytical peak at Fe $K\alpha$ on a LIF crystal for no Fe containing diverse MAN standards. b) X-ray counts for the analytical peak at Si $K\alpha$ on a TAP crystal for no Si containing diverse MAN standards.

Some difficulties are encountered by the lack of low-MAN standards (MAN < 20) for the background calculation for Si as most of them are oxides containing Si. Also, some medium-MAN (30 to 40) and larger-MAN standards (55 to 72) are not easily available. All lanthanides, together with Cs and Ba, can fill the interval from 55 to 70 but are not stable as they strongly oxidize. Heavy elements, on the contrary, or their compounds reveal a lot of spectral lines resulting in a signal higher than the expected background for many analytical peaks so that their application as MAN standard is limited.

8. INTERFERENCE CORRECTIONS FOR WDS

When establishing the background curves for an analytical peak, MAN standards containing another element that causes also characteristic X-rays at that investigated peak will exhibit a higher signal than interpolated from the background curve as determined by all the other standards which do not show this particular disadvantage. Fig. 11a shows the same background curve for Fe K α as in Fig. 10a but with the signals added for three standards containing interfering elements on Fe (Mn, Co and Ni). The “raised background values” on the analytical peak position are not only directly visible but their value on top of the background curve is a measure for the degree of interference [8]. Consequently these MAN standards can be used too but as “Interference standards”.

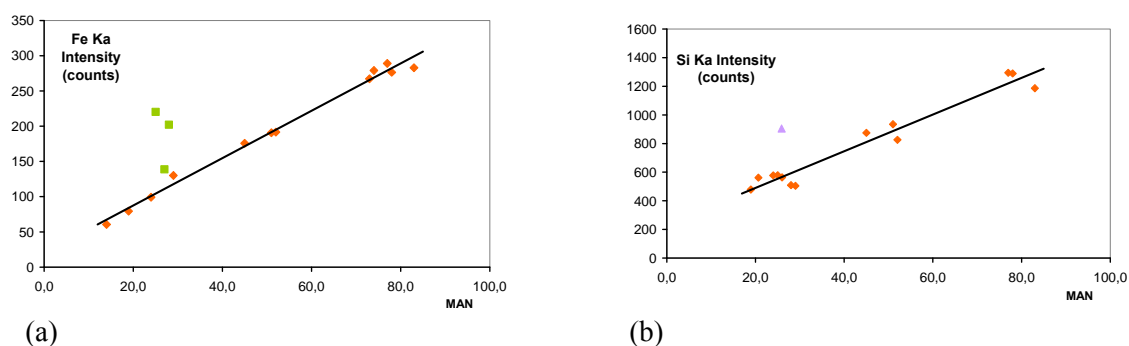


Figure 11. a) Same as Fig. 10a but adding the standards Mn, Co & Ni. b) Same as Fig. 10b but adding a steel standard with 0.15% Si.

9. ELEMENT STANDARDS FOR WDS

Similarly, if the element for which the background curve is established is present, an increase above this curve will be visualized, as shown in Fig. 11b for 0.15 % Si in a steel standard. For quantification of the concentration of an element in a phase, element standards have to be used. They can be in fact the same as the MAN (or interference) standards but now there is really looked for the intensity of the analyzed element at the analytical peak position. The background to subtract from this intensity is found in the background curves as the composition and hence the MAN is well known.

10. MICROANALYTICAL EXAMPLE WITH WDS

The microprobe used is the model SEMQ from ARL and has five WDS spectrometers and a silicon drift EDS detector avoiding the use of liquid nitrogen.

After establishing an analytical programme for the elements present and calibrating all the needed analytical peak positions, the MAN standards, interference standards, and element standards are measured. The background curves are completed, the standards are assigned and the interference corrections are set up and checked as well as the standard compositions. The unknowns can be directly analyzed and all this just by measuring on the analytical peaks. Many spot analyses have sometimes to be realized to obtain average compositions because the different phases made during short processing times are not always homogeneous in composition. Their composition can be determined by trial and error starting with the not for background corrected intensities so that a MAN can be calculated leading to a first approximation for the background subtraction. In a second round, a more precise composition and corresponding MAN are obtained, resulting in a more realistic background subtraction, etc. This process goes on till convergence [6].

According to this procedure the four different phases spinel, pyroxene, zinc silicate, and a silicate matrix, in a zinc-bearing slag are analyzed (Table 1) and their eventual formulae are calculated. The amount of each phase can be determined based on the known chemical composition (Table 2) as well as the amount of zinc attributed to each phase (Table 3).

Table 1. The composition of each phase.

Phase	Total	SiO ₂	PbO	K ₂ O	CaO	MnO	ZnO	FeO	Al ₂ O ₃	Cr ₂ O ₃	MgO	Na ₂ O
1	94.3	0.1	0.2	0.0	0.2	2.6	17.9	67.2	1.9	2.1	2.1	0.0
2	100.2	46.1	0.2	0.2	18.9	4.1	6.7	15.7	2.5	0.0	5.4	0.5
3	101.0	30.3	0.2	0.1	1.3	3.5	49.2	9.5	0.2	0.0	6.8	0.0
4	99.3	42.0	3.7	1.6	14.6	4.6	12.0	11.6	2.5	0.0	1.8	4.8

1 = spinel (Zn_{0.50}Fe_{0.29}Mg_{0.12}Mn_{0.08}Ca_{0.01})_{1.0}(Fe_{0.92}Al_{0.04}Cr_{0.03})_{2.0}O₄
 2 = pyroxene; 3 = zinc silicate; 4 = silicate matrix

Table 2. The weight % distribution “%Phase” of the four phases.

Phase	Phase%	SiO ₂	PbO	K ₂ O	CaO	MnO	ZnO	FeO	Al ₂ O ₃	Cr ₂ O ₃	MgO	Na ₂ O
1	38.2	0.0	0.1	0.0	0.1	1.0	6.8	25.7	0.7	0.8	0.8	0.0
2	16.8	7.7	0.0	0.0	3.2	0.7	1.1	2.6	0.4	0.0	0.9	0.1
3	22.9	6.9	0.0	0.0	0.3	0.8	11.2	2.2	0.0	0.0	1.5	0.0
4	19.8	8.3	0.7	0.3	2.9	0.9	2.4	2.3	0.5	0.0	0.4	1.0
	97.7	23.0	0.9	0.4	6.4	3.4	21.6	32.8	1.7	0.8	3.6	1.0
Chemical composition		23.0	1.2	NA	6.9	3.4	21.6	32.8	2.0	0.3	2.8	NA

1 = spinel (Zn_{0.50}Fe_{0.29}Mg_{0.12}Mn_{0.08}Ca_{0.01})_{1.0}(Fe_{0.92}Al_{0.04}Cr_{0.03})_{2.0}O₄
 2 = pyroxene; 3 = zinc silicate; 4 = silicate matrix.

Table 3. The relative distribution of the several elements over the four phases.

Phase	Phase%	SiO ₂	PbO	K ₂ O	CaO	MnO	ZnO	FeO	Al ₂ O ₃	Cr ₂ O ₃	MgO	Na ₂ O
1	38.2	0.1	9.9	1.1	1.2	29.3	<u>31.7</u>	78.3	43.5	99.0	22.0	0.0
2	16.8	33.6	4.0	8.4	49.4	20.2	5.2	8.1	24.8	0.4	25.1	7.5
3	22.9	30.1	3.9	3.8	4.7	23.5	52.1	6.6	2.7	0.3	43.0	0.2
4	19.8	36.1	82.2	86.7	44.8	27.0	11.0	7.0	29.0	0.2	9.9	92.3
		100	100	100	100	100	100	100	100	100	100	100

1 = spinel (Zn_{0.50}Fe_{0.29}Mg_{0.12}Mn_{0.08}Ca_{0.01})_{1.0}(Fe_{0.92}Al_{0.04}Cr_{0.03})_{2.0}O₄
 2 = pyroxene; 3 = zinc silicate; 4 = silicate matrix

These results from a slag before zinc removal can then be compared with the results afterwards so that a better evaluation can be made of the possible recovery of zinc from such a material. These microanalyses lead for example to the conclusion that one third (31.7 % ZnO) of the oxidized zinc is assigned to spinel grains making a total recovery of zinc out of this slag very difficult. A loss of about 33 % zinc can therefore be expected.

11. CONCLUSIONS

It is obvious that many products with lots of elements occur in non-ferrous recycling.

X-ray diffraction analyses alone often fail to identify the present compounds.

Microanalyses are essential to characterize the present phases, to really know their composition, and their quantity. Spot analyses have to be realized on many spots to obtain average compositions as the different phases made by mankind in some hours are not always homogeneous compared to similar phases formed in nature over many centuries.

Wavelength-dispersive spectrometers are indispensable as many elements cannot be treated by an energy-dispersive spectrometer and as quantification with standards is often necessary. A background calculation technique based on the mean atomic number drastically enhances the analytical speed of these wavelength-dispersive analyses.

Area analyses with an energy-dispersive spectrometer are not performed but this spectrometer is used for a qualitative check of the present elements in a phase.

Optical micrographs and backscattered electron images contribute, moreover, to observe all phases present, their size and their bonding.

Knowledge of these analytical data allows further optimisation or development of recovery processes.

12. REFERENCES

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