

AN INVESTIGATION OF THE METALLURGICAL MATERIAL FROM PEUN BAOLO AND THONG NA NGUAK, LAOS

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Appendix 1: analytical equipment and its working conditions

SEM-EDS

SEM-EDS was applied as a main technique in characterisation of both chemical composition of the samples and their microstructure. The SEM-EDS examination was designed to make best use of all of the advantages this method offers. In the case of the current project there were: the permanent availability of the equipment, quickness of analysis and good imaging abilities under high magnification. Obviously, SEM-EDS has also a range of limitations, among which the major are quite low accuracy and precision, high detection limit, unstable beam current and possibility of overlapping of some elements, lead and sulphur, which may have some importance for this studies, included. Although, the limits seem to be quite serious, and for sure for some purposes they would disqualify SEM-EDS as a method of choice, in the case of the present project and its goals the abilities of the technique are considered as fairly enough and its limitations were assessed as not harmful regarding the final results. The operating conditions and research strategy were adjusted to enhance the assets of SEM-EDS rather than to minimise its inherent constrains.

Sample preparation

For SEM analysis, resin-mounted polished blocks were prepared. The selected artefacts were cut perpendicularly to the surface with an abrasive tile cutter in the place of interest. Small, approximately 1-2 cm across, pieces were removed and embedded in epoxy raisin. The block was grounded flat and polished with diamond paste to 1 or 0.25 μm depending on polishing qualities of a material. Before analysing, the samples were coated with thin layer of carbon.

Equipment and operating conditions

The samples were analysed with a Phillips XL30 scanning electron microscope fitted with an energy dispersive spectrometer (Oxford Instrument INCA series) and ZAF-corrections. Calibration of the EDS using a cobalt standard was conducted regularly throughout the analysis.

The analyses were carried out over a span of two months. During this period the SEM-EDS set-up were modified by a main operator of the instrument what resulted in a different values of emission current during the first several session (in a range of 87-93 μA) and the latter ones (68-73 μA). Despite these differences, the actual current of the incident beam, so the number of electrons reaching a sample, was kept quite stable by adjustment of the spotsize, i.e. smaller (4) during the first analyses and larger (6.3-6.7) during the latter ones. The construction of the SEM is image-oriented and allows rastering the beam over a quite large area of a sample what was taken advantage of many times in the current research. The mobility of the beam, however, and the necessity to obtain a balance in a vacuum system each time the chamber is reloaded make the current slightly unstable over the time which is clearly demonstrated in totals, sometimes widely varied. The slight changes in current were tried to be recognised by observing a detector deadtime on a cobalt standard for which a target value was in a range of 35-40%. If it dropped, the spotsize was slightly increased.

An accelerating voltage of 20kV and working distance of 10 mm were fixed. Process time was set to 5 giving. Livetime of 50s was set during most analyses. However, in some cases lower value of 30s was also used.

Accuracy, precision, detection limits

The accuracy of the measurements was assessed using the certified reference material, the U.S. Geological Survey basalt standard (abbreviated BHVO-2). The standard was analysed following the same procedure which was applied in the examination of the sample

The above described equipment and its set-up allows for chemical analysis with accuracy within 5-10% relative error of the expected concentrations for the major oxides (Table 1). It means that the absolute error of each measurement usually did not exceed 0.2-0.5 wt% and in the case of silica 2.5 wt%. Precision, presented as the standard deviation (σ) of the measurements, was 0.1-0.2 wt%. It gives quite satisfactory relative σ values falling usually in a range of 1-5%. The possibility to improve the precision by extending the livetime was not used since it entails an undesirable increase in the analysis time.

P2O3 present in the standard in the amount of 0.27wt% was not detected. This sets the detection limits on a level of sensitivity generally accepted for the EDS, around 0.2 for elements with $Z > Z_{Si}$ (Ciliberto, & Spoto, 2000, p. 410; Pollard & Heron, 1996, p. 52; Potts, 1992, p. 366).

	Na2O	MgO	Al2O3	SiO2	K2O	CaO	TiO2	FeO	total
average	2.0	7.0	12.8	52.4	0.5	11.2	2.9	11.2	117.1
σ	0.05	0.13	0.12	0.06	0.04	0.12	0.04	0.08	2.08
relative σ	2.3	1.8	1.0	0.1	7.3	1.1	1.5	0.7	1.8
given	2.2	7.2	13.5	49.9	0.5	11.4	2.7	11.6	
absolute error	0.22	0.23	0.66	-2.46	0.01	0.19	-0.16	0.40	
relative error	9.82	3.15	4.89	4.93	2.69	1.67	5.86	3.48	
average	2.1	7.0	12.9	52.3	0.5	11.2	2.9	11.1	95.3
σ	0.04	0.10	0.16	0.14	0.04	0.15	0.05	0.04	3.57
relative σ	1.7	1.4	1.2	0.3	8.2	1.3	1.6	0.4	3.7
given	2.2	7.2	13.5	49.9	0.5	11.4	2.7	11.6	
absolute error	0.10	0.21	0.62	-2.39	-0.01	0.19	-0.13	0.50	
relative error	4.65	2.86	4.62	4.79	1.92	1.67	4.64	4.28	
average	2.0	6.9	12.8	52.2	0.5	11.2	2.8	11.5	100.5
σ	0.08	0.10	0.10	0.14	0.01	0.09	0.10	0.12	0.55
relative σ	4.0	1.4	0.8	0.3	2.8	0.8	3.6	1.0	0.5
given	2.2	7.2	13.5	49.9	0.5	11.4	2.7	11.6	
absolute error	0.17	0.33	0.71	-2.28	0.02	0.21	-0.11	0.14	
relative error	7.77	4.50	5.28	4.57	3.85	1.80	4.12	1.16	

Table 1. SEM-EDS results (wt%) for the basalt standard (BHVO-2), normalised; nd – not detected; σ – standard deviation; given – certified content in the standard; relative σ : $\frac{\sigma}{\text{average}} \times 100\%$; absolute error: *given minus average*; relative error: $\frac{\text{absolute error}}{\text{given}} \times 100\%$.

Considering all the parameters, the result presented in the dissertation may be considered as quantitative. All major and minor elements were measured with quite good accuracy allowing for comparison between the samples. However, differences of less than 0.5-1% and in the case of silica below 3% must be handled carefully.

Types of analyses

Basically, two kinds of chemical analysis were carried out with the SEM-EDS: analysis of single phases and bulk analysis. In both cases some difficulties were encountered and the most suitable solutions were tried to be established. All of them are described below.

Bulk analysis

Bulk analysis was conducted on each type of material by scanning with a beam a selected area of a sample. This method entails several problems. First of all, the SEM-EDS electronics are set up for analysis of a single homogeneous phase. Therefore, when several different phases, such as for instance oxides and metallic prills, are analysed at the same time some slide in accuracy must be taken into consideration. Another concerns were a quite small area which can be scanned each time, limited by the size of area possible to be encompassed in an image and the problems with the geometry of the beam-sample-detector system, and an associated question about the representativeness of the results. On the other hand, however, the opportunity to see what is actually analysed allows assessing each analysis separately and explaining any deviation which possibly occurs. In order to balance the positives and negatives, a strategy for bulk analyses was adopted where the possibly largest, but no bigger than c. 1mm^2 , area is analysed which includes all main phases recognised in a sample and excludes, if possible, large voids and clearly corroded regions. As a result areas of varied size from $200\text{ }\mu\text{m}$ to 1 mm across were analysed what, however, is not considered as a drawback. Each sample was analysed at least 3 times and the bulk composition used in the studies is a mean of all scans.

Analysis of single phases

SEM-EDS analysis of single phases with the use of spot mode or small scans was applied as a main method of their chemical and mineralogical identification. With the accelerating voltage of 20 keV , the actual size of the area analysed falls in a range of $2\text{-}6\text{ }\mu\text{m}$ in width and depth depending on the average density of sample (Pott 1992, 336-337). Therefore, it was assumed that the phase for which an analysis without contribution of the background may be securely conducted should be at least $10\text{-}20\text{ }\mu\text{m}$ across. Since most of the phases occurring in the samples are much larger, measurements of their composition may be considered as fully qualitative.

In several cases, however, an attempt was made to recognise the composition of the phases smaller than $10\text{-}20\text{ }\mu\text{m}$. In this case only average composition may have been assumed by comparing the chemistry of the phase and its surroundings. This issue was recognised as quite inconvenient in the case of copper prills since the size of the majority of them does not extent $20\text{ }\mu\text{m}$. Furthermore, they occur in the iron-rich background so an artificial enrichment in this element was likely. Since the amount of iron in copper prills may be significant in an interpretation, the size of the prill analysed was always recorded considered.

Data processing

As may be easily seen in the tables through the dissertation, the totals of both bulk and single spot analyses are widely varied. There are several reason for that. First of all, the electron beam was diagnosed as quite unstable, what was especially true in the case of the measurements done during the first several sessions. It means that the beam of wide-ranging current streaked the samples generating a varied amount of X-rays. Regarding the low totals for the crucible fabric, they usually result from its high porosity. In the case of both, varied

current and high porosity, normalisation to 100% has been considered as a good solution what was confirmed when results obtained for the standard and artefacts with high and low totals after normalisation were compared (Table 1)

The only situation when normalisation was not applied was when the presence of missing elements were suspected. That was the case when malachite samples were analysed giving consistent totals of c. 87% and during the examination of carbonate phases probably associated with weathering.

Since the EDS system is generally considered as incapable to measure oxygen accurately, the amount of this element was calculated by stoichiometry. In most cases the INCA analysis software was used to calculate oxides by stoichiometry. The software was set up to calculate iron as occurring exclusively in the form of FeO what is obviously a simplification and may cause some minor fluctuation in totals. There were, however several situations where stoichiometry was not used, at least not automatically, or other oxides were calculated. Regarding copper oxide minerals, the results were firstly assessed in a raw format, where copper and oxygen were measured by element. The ratio of these two elements was compared and possibility of the existence of some missing (C, H) elements was considered. Similar procedure was followed in the case of the phases with ion configuration not matching the general rule accepted for stoichiometry calculation.

OPTICAL MICROSCOPY

Reflected light microscopy

Before carbon coating the polished blocks were examined under polarised reflected light using a metallurgical microscope Olympus BX60. The examination was carried out in order to initially characterise the microstructure and types of phases in the slag samples as well as to orient the further studies with the SEM-EDS. The images made under the reflected light microscope and included in the dissertation are called in the captions *Reflected light micrographs*.

Unfortunately, none of the samples was etched. The etching may have been very useful in the case on the copper ingot to reveal the features of its microstructure and allow the studies on the techniques used in its manufacture (grain size). It is strongly recommended in the future. The etching of the crucible fabrics may also be used for better understanding of the composition of fine-grained fraction under the SEM-EDS.

Transmitted light microscopy

The ceramic petrography was applied to examine several crucible and pottery samples. Cross-sections of the samples were fixed to a glass slide and abraded to a standard thickness of 0.03 mm. Thin sections were examined under a Leica polarizing microscope at magnification up to 200x. The images taken under the petrographic microscope are referred to as *Optical micrographs* in the captions.

The technique was applied to check if any of the features of the crucible fabrics was not overlooked in the examination with the SEM-EDS. In the case of the domestic pottery samples it was aimed to recognise their general character and similarity to the clay used in the manufacture of crucibles as well as to assess if further studies with SEM-EDS may be useful.

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