

Concepts for a portable X-ray spectrometer for non-destructive analysis of works of art

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1. Introduction

X-ray fluorescence analysis is a well established non-destructive method of analysis used for quite different materials and objects. But especially the X-ray sources were a long time very similar to the first x-ray tubes already used by Wilhelm Conrad Röntgen. A portable X-ray device should have a low weight and a low power consumption, whereas the intensity emission should be large enough to allow a reasonable measurement time. Besides the effective x-ray tube a well collimated or even focussed x-ray beam is an important condition for realizing a spatially resolved analysis. The development of relative small size X-ray optical elements on glass capillary basis in the late eighties (see e.g. /1,2/) has offered new opportunities in the field of XRF analysis.

Furthermore an effective detection of X-ray fluorescence radiation is also an important component of a portable spectrometer. An energy resolution necessary to distinguish emission lines of the most elements of interest can be only guaranteed in conventional Si (Li) detectors if they are cooled by liquid nitrogen. Recently, a new noncryogenic x-ray detection system was developed, which uses a silicon drift detector cooled by a Peltier element (see e. g./3/). This detector is now developed as an industrial product and manufactured at the German company RÖNTEC (XFlash®).

On the basis of these modern devices a powerful portable XRF spectrometer can be developed.

2. General requirements to a portable XRF spectrometer

Especially for works of art which can not transported to a laboratory for investigation a portable XRF spectrometer is needed. Such a device should have a low weight and a small size. This requirement can be only fulfilled if a high brilliant X-ray source with low power consumption is realized. For the X-ray detection module a good energy resolution is required which allows a clear separation of low-Z element lines (e. g. sulphur, chlorine and calcium). As already mentioned the XFlash® detector with Peltier cooling is an appropriate solution of this problem.

For determination of the elemental composition of details on works of art an element analysis with spatial resolution should be carried out. The necessary collimated X-ray beam will be formed by a capillary element utilizing total reflection at grazing incidence on the inner wall

surfaces of the capillary channels. Some examples of such collimating elements are given in the next section.

A further important point is the possibility of visualization of the studied surface area. For this purpose a CCD camera may be used.

For obtaining reliable data with a reasonable spatial resolution a mechanically stable positioning of the excitation and detection head is necessary. This includes also an effective vibration damping.

Finally, the device should be equipped with a customer friendly operating system.

3. Capillary optical elements for beam collimation

A combination of a microfocuss x-ray tube with a capillary optical element leads to a beam focusing on the sample because the acceptance angle of such a capillary element is larger than for a pinhole collimator. Already by using a monocapillary relative intensity gains between 2 and 10 can be achieved.

Larger acceptance angles can be realized with a focusing polycapillary lens.

An example of such a focusing polycapillary lens with the following geometrical parameters was studied at different photon energies. Transmission coefficients and focus sizes are presented in Table 1.

Geometrical parameters of the polycapillary lens:

Distance between anode spot and lens entrance: 56 mm.

Length of the lens: 52 mm.

Distance between the lens exit and the focal spot: 28 mm.

Acceptance angle: 0.032 rad.

Energy (keV)	8.0	12.0	17.5	20.5
Transmission (%)	9.6	7.7	2.8	1.9
Focal spot size (FWHM) (mm)		80	50	-

Table 1: Transmission coefficients and focal spot sizes for a polycapillary lens at different photon energies

The focal spot sizes were measured by a scan with a 20 mm pinhole in the focal plane of the polycapillary lens.

Besides polycapillary lenses ellipsoidal monocapillaries may be also used for x-ray beam focusing. Ellipsoidal monocapillaries have a larger open area and therefore a better transmission than polycapillary lenses. Furthermore, the corresponding manufacturing technology is easier than for polycapillary lenses.

A comparison between the intensity gains obtained by a cylindrical monocapillary, an ellipsoid and a polycapillary lens for a source-sample distance of 55 mm, a source size of 10 mm and a photon energy of 17.4 keV shows that the relative intensity gain is for an ellipsoid about one order of magnitude higher than in the case of a cylindrical monocapillary and two orders of magnitude for a polycapillary lens.

The examples given show the high capacity of capillary optics for application in micro XRF. The corresponding optical elements may be used for an effective collimation of the primary X-ray beam as well as for an effective collection of secondary fluorescence radiation emitted by the sample. Possible versions of different spectrometer forms equipped with capillary optical elements will be briefly discussed in the next section.

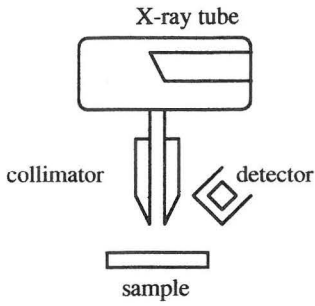
4. Different micro-XRF spectrometer concepts and preliminary experimental results

Usually a XRF spectrometer consists of a X-ray tube and a detector with a corresponding power supply unit, a control unit and a PC with data processing software. If a spatial resolved analysis should be realized, a conventional pinhole collimator is situated in front of the X-ray tube (see version I in Fig. 1).

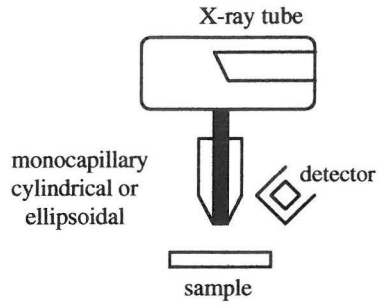
In the versions II and IV a conventional collimator is substituted by a monocapillary and a microlens, respectively. For improving the effectiveness of secondary fluorescence excitation instead of focusing optical element a monochromatizing X-ray concentrator (MONO-XC) /4/ can be used. This element consists of straight monocapillaries combined with mosaic graphit crystals (HOPG). At the entrance side collect the capillaries the X-radiation from the source and transport to the monochromator crystals. A second set of monocapillaries collect the monochromatized radiation and focus it on the sample. A more detailed description of this element is given in /4/. Applications of such an optical element are shown in V and VI.

As already mentioned a spatially resolved elemental analysis is also possible if in front of the detector a polycapillary conic structure is situated (see III and VI in Fig. 1). This structure collect the secondary fluorescence yield from a small surface area of the sample so that it is not necessary to collimate the primary beam. Further information about this polycapillary conic collimator (POLY-CCC) and its application in micro XRF one can find in /5/.

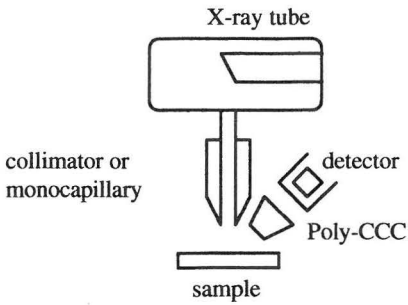
Table 2 describes advantages (+) and disadvantages (-) of the different versions shown schematically on Fig. 1. The large diversity of combinations presented offers specified solutions for different analytical problems.



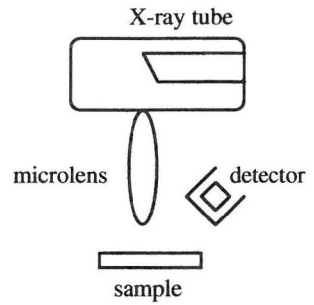
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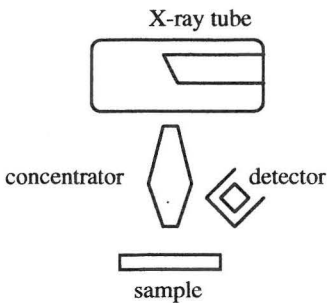
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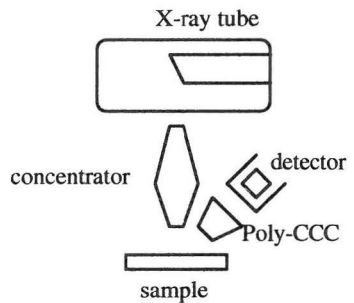
III



IV



V



VI

Fig. 1: Schemes of different X-ray spectrometer types

Table 2: Advantages and disadvantages of different X-ray spectrometer types

I. Source-Collimator-Sample-Detector

- + simple design
- + anode spot is not critical
- low sensitivity
- low intensity at local analysis

II. Source-Monocapillary-Sample-Detector

- + spatial resolution 10 mm
- + higher intensity compared with a collimator
- minimum anode spot size about 10 - 200 mm
- precise alignment necessary

III. Source-Collimator-Sample-POLY-CCC-Detector

- + simple design
- + anode spot is not critical
- + spatial resolution about 20 mm
- minimum distance source - sample

IV. Source-Microlens-Sample-Detector

- + spatial resolution 10 - 200 mm
- + high intensity
- minimum anode spot size about 10 - 200 mm
- precise alignment necessary

V. Source-Concentrator-Sample-Detector

- + spatial resolution about 100 mm
- + high intensity
- + high sensitivity (monochromatization)
- minimum anode spot size about 100 - 300 mm
- precise alignment necessary

VI. Source-Concentrator-Sample-POLY-CCC-Detector

- + spatial resolution about 20 mm
- + high intensity
- + high sensitivity (monochromatization)
- minimum anode spot size about 100 - 300 mm
- precise alignment necessary

For testing the general concept a laboratory version of type I was realized and a standard glass probe (trace elements with a concentration of about 500 ppm) was analysed. A small sized low-powered tube (30 W) with a tungsten anode and a 0.8 mm collimator were used as X-ray source. The fluorescence radiation spectrum was measured with a XFlash detector. Due to a glass window used in the X-ray tube, the low-atomic-number elements were not effectively excited, as seen from the spectrum shown in Fig. 2. Several improvements, such as the substitution of the glass window by a beryllium one, the use of capillary optics for beam collimation, are subject of future work.

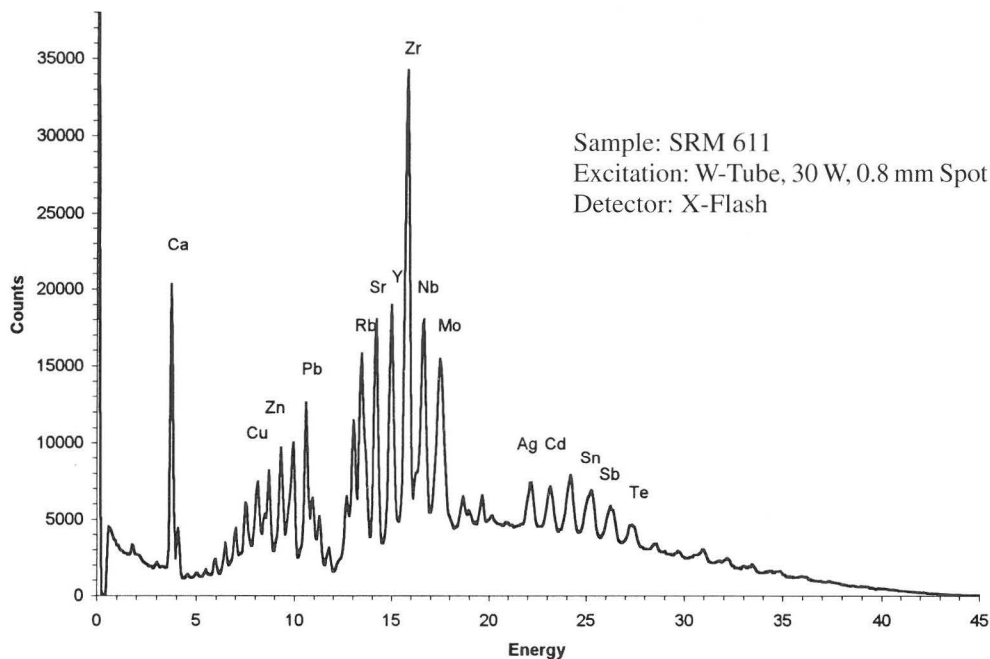


Fig. 2: X-ray fluorescence spectrum of NIST 611 glass standard with trace elements (About 500 ppm)

5. Concluding remarks

It has been shown that a combination of a high brilliant low-powered X-ray tube with a capillary optical system and a non-cryogenic X-ray detector X-Flash allows to realize a portable x-ray spectrometer. First results obtained with a preliminary laboratory version of such a device have shown that the elements of interest in works of art may be analysed.

Acknowledgment

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References

- [1] V. A. ARKADIEV, A. I. KOLOMIITSEV, M. A. KUMAKHOV, I. YU. PONOMAREV, I. A. KHODEEV, YU. P. CHERTOV, I. M. SHAKHPARONOV, „Wide-banded x-ray optics with large aperture“, *Usp.Fiz.Nauk*, Vol.157, pp.529-537, 1989 (in [*Sov.Phys.Usp.*, Vol.32, pp.271-276, 1989])
- [2] M. A. KUMAKHOV, F. F. KOMAROV, „Multiple reflections from surface x-ray optics2, *Physics reports*, Vol.191, No.5, pp.289-350, 1990.
- [3] C. FIORINI AND A. LONGONI „Application of a new noncryogenic x-ray detector in portable instruments for archaeometric analyses“, *Rev. of Sci. Instr.*, Vol. 69, No. 3, 1523 - 1528, 1998.
- [4] A. A. BZHAUMIKHOV, „Monochromatizing x-ray concentrator (MONO-XC)“, *Proceedings of SPIE.*, Vol. 3444, 512 - 516, 1998.
- [5] A. A. BZHAUMIKHOV, N. LANGHOFF, J. SCHMALZ, R. WEDELL, V. I. BELOGLAZOV, N. F. LEBEDEV, „Polycapillary conic collimator for micro-XRF“, *Proceedings of SPIE*, Vol. 3444, 430 - 435, 1998.