A portable XRF spectrometer for non-destructive analyses in archaeometry

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Abstract

A new X-ray Fluorescence (XRF) spectrometer based on a Silicon Drift Detector (SDD) cooled by a Peltier element and on a miniaturised X-ray generator is presented here. Due to the low output capacitance of the SDD and to the integration of the front-end transistor on the detector, this system offers an energy resolution better than 160 eV FWHM at 6 keV (for a shaping time of 2 μs) at a temperature of $-15°C$. The small size of the detection–excitation module and the elimination of the liquid nitrogen cryostat make this system ideal for portable equipment for non-destructive XRF analysis. The first experimental results obtained in analyses of paintings of different ages and of metal alloys are reported.

1. Introduction

The XRF (X-ray Fluorescence) spectroscopy is a non-destructive technique of analysis widely used in archaeometry for the identification of chemical elements in pigments, metal alloys, and other materials of artistic interest [1]. Very often the works of art to be analyzed are not transportable in scientific laboratories. The development of portable instruments, based on the XRF spectroscopy, for measurements "on the field" is therefore widely justified.

The classical high-resolution cryogenic detectors, like Si(Li) and HPGe detectors (whose resolution is of the order of 140 eV FWHM at 6 keV), are not completely suitable for portable instrumentation in archaeometry. The main limitations arise from the need of the liquid nitrogen cooling (large-size of the cryostat, need of periodic liquid nitrogen refill, maintenance cost).

Recently, non-cryogenic detectors, like Peltier cooled Silicon PIN diodes, have been employed in portable systems for XRF analysis, with a big improvement in terms of size and weight of the instrumentation [2]. Their energy resolution, of the order of 250 eV FWHM at 6 keV, is nevertheless sometimes unsatisfactory (for instance, in the identification of light chemical elements).

In this work, we present an XRF spectrometer based on a new kind of energy dispersive device, the Silicon Drift Detector (SDD). The SDD, cooled by a Peltier element, can reach a resolution of the order of 160 eV FWHM at 6 keV. This feature makes this device ideal for portable high-resolution XRF spectrometers.

2. The proposed instrumentation

A portable spectrometer based on a SDD and on a miniaturised low power X-ray generator has been realized. The compact measurement head, composed of a detection module and of an excitation module, is shown in Fig. 1, during the analysis of an Egyptian linen (Antinopolis, III century A.C.) carried out at the Vatican Museums.

Fig. 1. The portable XRF spectrometer during a measurement on an Egyptian linen.

The heart of the detection module is the SDD, introduced by Gatti and Rehak in 1983 [3], with a JFET integrated on the detector chip near the collecting anode. The output capacitance of the SDD is of the order of 0.1 pF and is independent of the active area of the device. The integration of the transistor allows to fully exploit the benefits, in terms of noise, of having a very small detector capacitance [4]. The structure of the SDD used in this instrument is shown in Fig. 2. The detector area is of 3 mm^2 while its thickness is of 300 μm. The SDD is cooled by means of a single-element Peltier and is packaged in a small sealed housing with a 7.5 μm Be window [5]. The quantum efficiency of the detection system is greater than 90% in the range between 1.5 and 10 keV. The resolution is 155 eV FWHM at 6 keV, with the SDD cooled at $-15°C$ and at 2 μs shaping time. An automatic biasing circuit for the detector and a charge preamplifier complete the detection module.

The excitation module is based on a miniaturised X-ray tube TF3002 manufactured by Oxford Instruments, suitably collimated. The tube, operated with air cooling, is equipped with a Tungsten anode and is powered at a maximum current of 0.1 mA and at a maximum voltage of 30 kV.

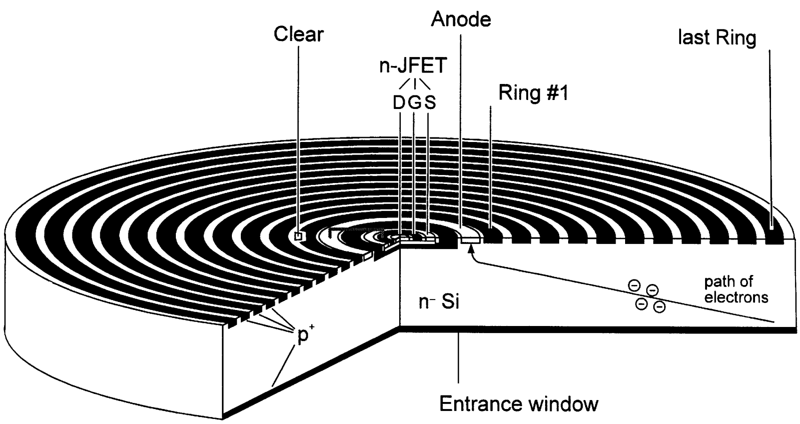

Fig. 2. Structure of the SDD with an integrated JFET.

3. Experimental results

The performances of the portable XRF spectrometer have been determined with several measurements in the laboratory on test samples and "on the field" on works of art. In particular, Fig. 3 shows the fluorescence spectrum of a metal alloy of known elemental composition. It can be noted that elements in low concentration, like Fe (0.15%) and Sn (0.2%), can be clearly identified in the spectrum. Fig. 4 shows the fluorescence spectrum of a contemporary orange color. A large number of chemical elements has been easily identified in the energy range between 2 and 26 keV. The XRF analysis carried out on the Egyptian linen shown in Fig. 1 has shown, for example, traces of gold in the yellow ochre of the earring of the young lady represented in the linen.

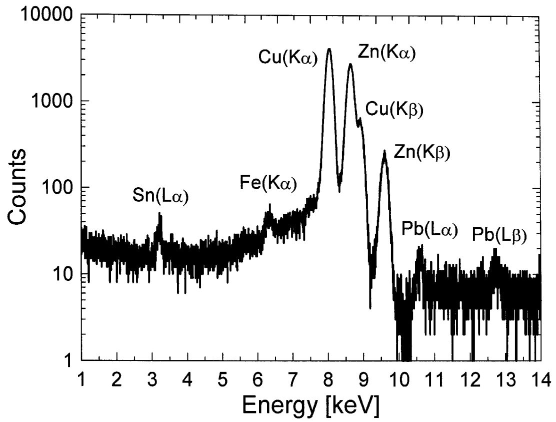

Fig. 3. Fluorescence spectrum of a metal alloy of known elemental composition (Cu–58%, Zn–40%, Pb–1%, Fe–0.15%, Sn–0.20%, …).

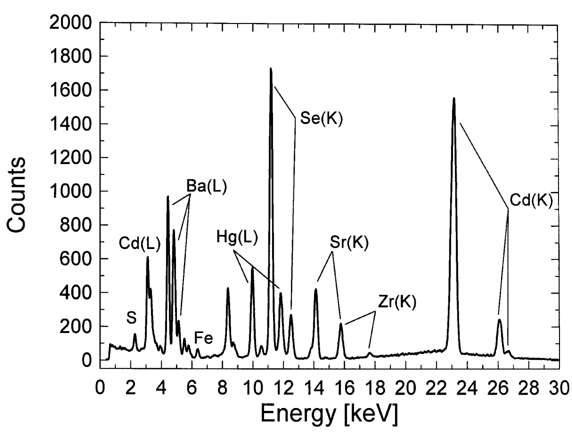

Fig. 4. Fluorescence spectrum of a contemporary orange color.

4. Conclusions

The portable XRF spectrometer based on the SDD combines high resolution with ease of operation. It seems ideal for non-destructive analyses "on the field" in archaeometry. We propose this instrument as a new basic tool for the study and the conservation of the artistic and archaeological heritage.

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