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Elemental mapping by means of an ultra-fast XRF spectrometer based on a novel high-performance monolithic array of Silicon Drift Detectors

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Abstract

This paper describes the design of a novel X-ray fluorescence spectrometer and presents its performance in elemental mapping applications. The spectrometer is based on a new ring-shaped monolithic array of four independent high-performance Silicon Drift Detectors (SDDs). These detectors and the innovative geometry of the spectrometer setup with a polycapillary lens coupled with a low-power X-ray generator allow reaching fast elemental mapping. Moreover, dedicated data acquisition system has been designed and developed in order to fully exploit the detection rate performance of the detector. The spectrometer can be used in several applications in the field of industrial technology, geology, scientific research and works of art analyses. In particular, in the field of bio-chemical sciences the spectrometer exploits its performance in imaging analysis of elements present in very low concentrations.

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1. Introduction

X-ray fluorescence (XRF) is one of the most widely used spectroscopic techniques in elemental identification and quantification [1]. It relies on the emission of characteristic photons' energies following the excitation of the inner atomic shells by an external source. The spectroscopic analysis of the XRF radiation excited by means of an X-ray beam is inherently non-destructive, the object to be analysed can be kept in air and the excitation of the material can be achieved by means of a simple low-power Roentgen tube. Several examples of XRF spectrometers are reported in the literature [2–4].

An XRF spectrometer in its basic form allows the user to study a certain area of the sample. It could be desirable to obtain the spatial distribution of the composing elements. This technique is called elemental mapping because it allows, in principle, building a map of the distribution of each element. Elemental mapping requires focusing the primary beam in a small spot of the order of 100 μm and moving the sample with an X–Y motion stage in order to scan the area under analysis. The achievable spatial resolution depends then both on the motors' precision and on the size of the X-ray exciting beam spot. In fact, the portion of the sample which is analysed is the one excited by the X-ray radiation.

The XRF elemental mapping is used in several application fields like industrial technology, works of art investigation, geology and scientific researches. This technique usually is able to identify elements in a sample in concentration down to the ppm and thanks to this feature, it can be used also in the field of bio-chemical and medical sciences.

2. The ultra-fast XRF spectrometer

The scheme of the XRF spectrometer setup is presented in Fig. 1a and the working principle is shown in Fig. 1b. An X-ray excitation beam produced by a compact and low-power X-ray tungsten anode generator is focused by a polycapillary X-ray lens and reaches the sample going

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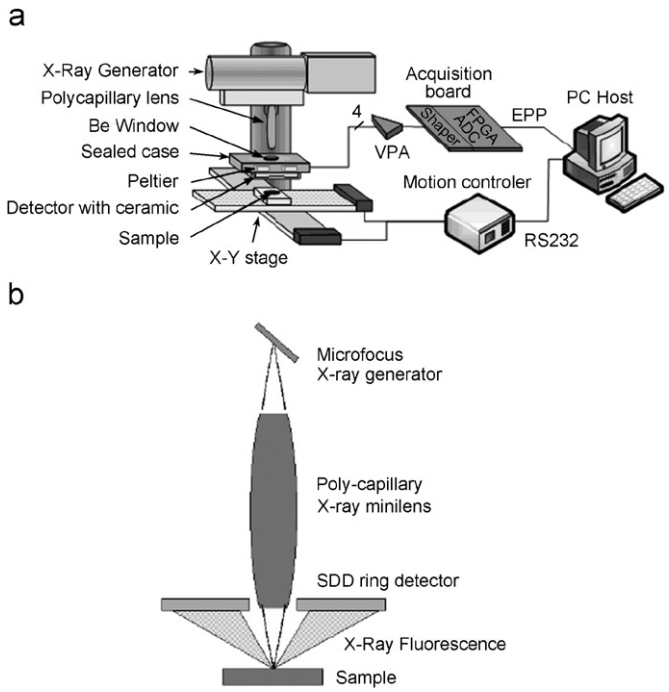

Fig. 1. (a) Scheme of the XRF spectrometer setup. (b) Working principle.

through a hole laser-cut in the centre of the ring-shaped array of detectors. This geometry optimizes the useful solid angle for the collection of the fluorescence from the sample, while the optics maximizes the photon density in the excitation spot even if a low-power X-ray generator is used. These features allow reaching very high scanning rate in elemental mapping that means fast measurements. Moreover, this kind of geometry allows positioning the sample near the detector (about 2 cm) thus limiting the absorption of low-energy X-rays by air. The sample is positioned on an X–Y stage with micrometric resolution which is responsible for the scanning.

The detector developed for this spectrometer is a monolithic array of Silicon Drift Detectors (SDDs) characterized by high-energy resolution at near-room temperature and high detection rate capability thanks to the low optimum shaping time (near 1 μs), consequence of the low output capacitance [5]. Fig. 2 shows the photograph of the 4-element detector mounted on the ceramic board where a part of the front-end electronics is connected. In particular, the detector is composed of four independent SDDs arranged around a hole cut in the centre of the chip. The heart-like shape of each SDD allows a nearly perfect surrounding of the central hole with a limited number of independent detectors. The active area of each of the four SDDs is about 15 mm^2, giving a total active area of the whole detector of about 60 mm^2, and the thickness of this detector is about 450 μm. The anode and an input JFET are integrated in the corners of each of the four SDDs. This configuration allows the design of SDDs with a very small collecting anode, characterized by an output capacitance of the order of 120 fF much lower than

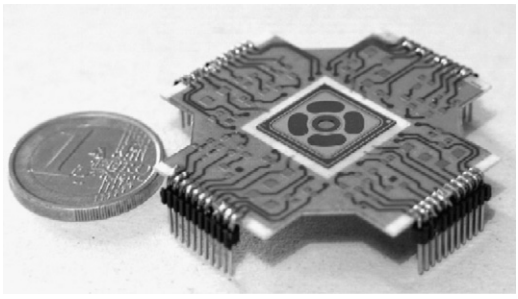

Fig. 2. Four-element detector mounted on the ceramic board where a part of the front-end electronics is mounted.

that of the conventional SDDs with central anode (greater than 200 fF). As a consequence, the spectroscopic resolution improves [6].

Fig. 1a shows also the four-channel data acquisition (DAQ) system that has been designed and realized for this spectrometer [7]. It provides low-noise and high-speed performance which makes it ideal for the high-resolution and high rate measurements in elemental mapping XRF.

The analog section of the DAQ is composed of four fifth order pseudo-Gaussian shaping amplifiers with user selectable shaping time (150 or 450 ns) followed by large-bandwidth peak-stretchers and base line restorers. Additionally, four fast amplifiers (30 ns shaping time constant) are used to provide the trigger signals for the pile-up rejection unit (PUR). Digital section contains a single 14-bit ADC (common for all the four channels) and a Virtex II field-programmable gate array (FPGA), responsible for the hardware control, on-chip histogram memorization and data transfer to the PC.

The whole system is controlled by custom made C++ software (running on Windows OS), which takes care of data transfer as well as of the graphic visualization and data analysis. Moreover, the system can perform automatic X–Y scan of the selected area of the sample with on-line data visualization. This feature is very useful in elemental mapping applications.

3. System qualification

The results of the deep characterization of the novel 4-element detector are presented in Refs. [6,8]. Here are reported the relevant results that show the main spectrometer performance. Fig. 3 shows the best energy spectrum collected by one of the four elements without any collimation when the detector was irradiated with a ^{55}Fe radioactive source at 1 μs shaping time. The detector was cooled by a Peltier system at about −15 °C, the average count rate was about 5 kHz. The measured average resolution of the four detectors is about 140 eV FWHM at the Mn Kα line at 1 μs shaping time. Up to 300 kcps the resolution stays below 220 eV FWHM with 375 ns shaping time. A peak-to-background ratio (defined as the ratio between the peak value of the Mn Kα line and the average value of the shoulder evaluated between 800 and 1200 eV)