A New XRF Spectrometer Based on a Ring-Shaped Multi-Element Silicon Drift Detector and on X-Ray Capillary Optics

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***Abstract*—This paper describes an innovative X-ray fluorescence spectrometer designed to achieve high-energy resolution, position resolution, and detection rate in elemental mapping applications. The spectrometer is based on a ring-shaped monolithic array of silicon drift detectors (SDDs) with a hole cut in its center. A coaxial X-ray excitation beam is transported to the sample through this hole. In this way, the solid angle for the collection of the X-ray fluorescence is optimized. Moreover, the X-ray beam is collimated on the sample using capillary optics in order to obtain high photon density in a small excitation spot. Detector, optics, and generator are assembled in a compact vacuum tightened unit. The structure of the proposed spectrometer and the first experimental results of its characterization are presented.**

***Index Terms*—Elemental mapping, silicon drift detectors, X-ray optics, XRF, X-ray spectrometry.**

I. Introduction

THE identification and localization of chemical elements in a sample ("elemental mapping") is mostly based on the spectroscopic analysis of the X-ray fluorescence emitted from different portions of the sample suitably excited [1]. Scanning electron microscopes (SEMs) are usually employed to perform elemental mapping on samples down to micrometric resolution (electron probe micro-analysis – EPMA) [2]. In this case, X-ray fluorescence is excited by the high-energy electrons of the microscope scanning beam. The fluorescence is usually detected by means of an energy-dispersive detector very often a Si(Li) detector [3]. This experimental setup features excellent position resolution (of the order of one micrometer) and energy resolution (of the order of 140 eV at 5.9 keV). On the other side, the use of the electron microscope limits the sample dimension (the sample must be inserted in the vacuum chamber of the microscope) and requires the metallization of its surface. Moreover, the use of Si(Li) detectors requires liquid nitrogen cooling (with related practical and economical disadvantages) and, because of the long shaping time required by this detector (of the order of 10 μs), the detection rate is relatively low (less than 10 kcounts/s) and, correspondingly, the area scan rate is quite slow.

The new spectrometer, presented in this paper, is based on a monolithic array of 12 silicon drift detectors (SDD) cooled by a Peltier element. The SDD [4], due to its very small output capacitance, can reach energy resolutions approaching the ones of liquid nitrogen cooled detectors, with the advantage of a much shorter shaping time (shorter than 1 μs) [5]. The SDD cells are arranged in a closed ring around a laser-cut hole in the center of the chip. The X-ray excitation beam (generated by a micro-focus tube) is transported through this hole by a capillary optics system to the sample with a spot diameter down to a few tens of μm [6]. This geometry optimizes the useful solid angle for the collection of the fluorescence from the sample and the X-ray optics maximizes the photon density in the excitation spot. Therefore, high scanning rate in elemental mapping applications can be achieved. Moreover, the sample must not be metallized and can be kept in air. Due to these features, the proposed spectrometer could constitute not only a good alternative to the scanning electron microscope in several applications, but it could also widen the range of possible applications of elemental mapping. For instance, it could be employed in fast on-line analyses during the production of technological materials or in nondestructive analyses of precious works of art. A transportable instrument for on-the-field applications can also be easily implemented.

II. Spectrometer Architecture

The spectrometer is based on a ring-shaped monolithic array of SDDs [7], which surrounds a hole cut in the silicon wafer and on a X-ray capillary collimator coupled to a micro-focus X-ray generator. The SDD provides high-energy resolution and high-detection rate without requiring liquid nitrogen cooling.

The subdivision of the total active area in several independent detectors allows to increase the maximum counting rate of the system with respect to a single detector of the same total active area. The particular structure of the detector array allows to transport the X-ray excitation beam to the sample through the hole in the center of the array, thus optimizing the solid angle for the collection of the X-ray fluorescence. The capillary optics collimates the X-ray excitation beam in a spot with a di-

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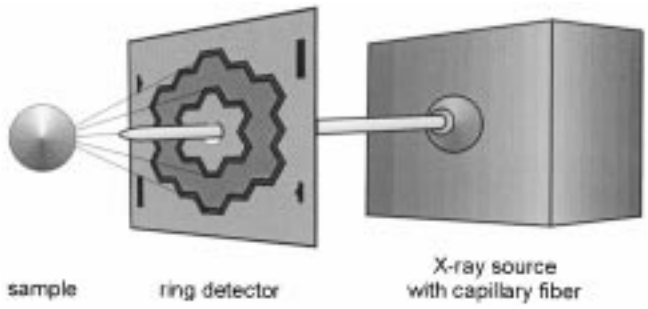

Fig. 1. Scheme of principle of the XRF spectrometer based on a ring-shaped detector.

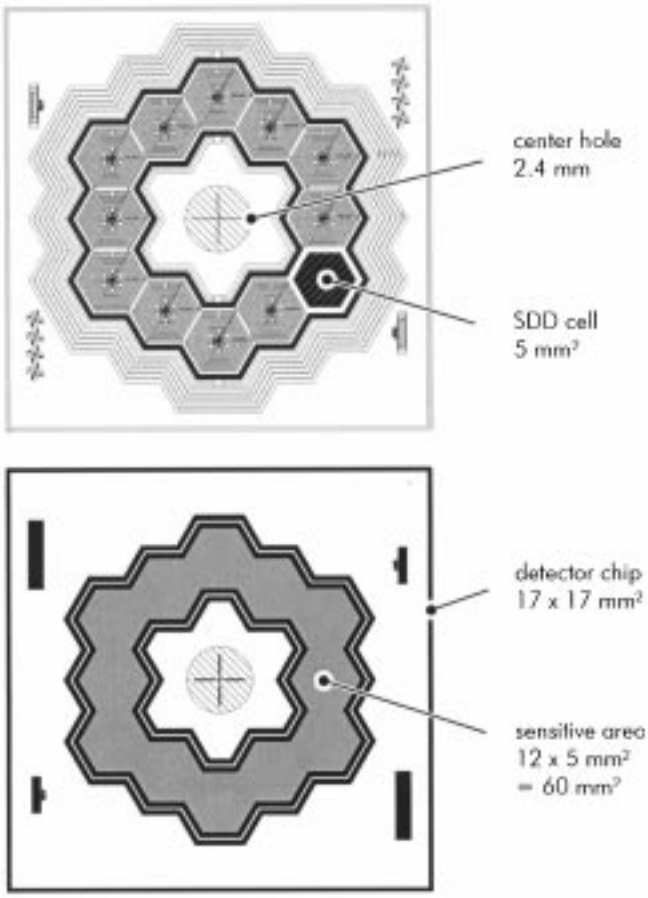

Fig. 2. Layout plot of the 12-channel SDD with the central hole. The upper plot shows the detector front side with the drift ring structures and the integrated readout electronics. The lower plot shows the detector back side with the unstructured radiation entrance window.

ameter down to a few tens of micrometers gaining efficiency with respect to simple pin-holes collimators. All these features guarantee high performances in terms of energy resolution and maximum count rate.

Fig. 1 shows the scheme of principle of the proposed spectrometer based on a ring shaped detector. The monolithic detector array adopted in the proposed spectrometer is composed of 12 independent SDDs, each one with an active area of 5 mm^2 (see Fig. 2). The starting material is a 300-μm-thick-high resistivity (2 kΩcm) silicon substrate. Each SDD of the array has the input JFET of the readout electronics directly integrated in close proximity of the collecting anode to reach optimum performance [8]. The hole in

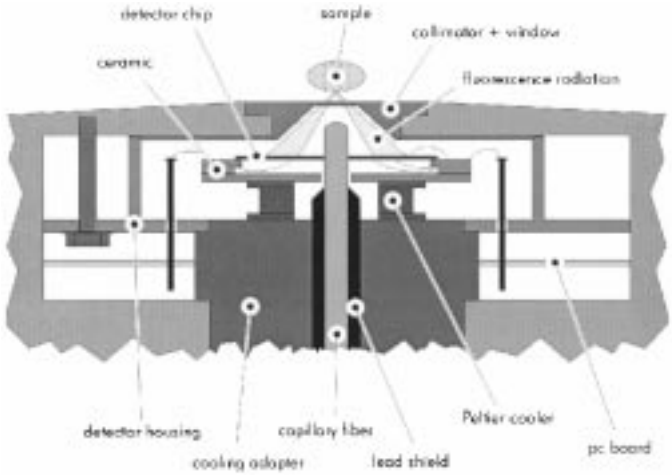

Fig. 3. Schematic cross section of the measurement head.

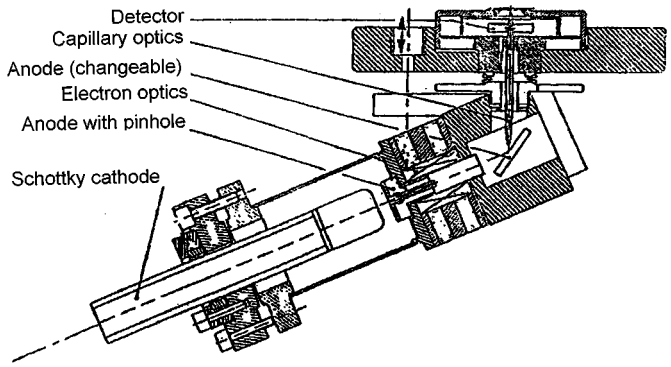

Fig. 4. Schematic cross section of the microfocus X-ray tube with interchangeable anode.

the center of the array has been laser cut. In order to avoid any undesired effect due to laser cutting the region around the central hole is isolated from the detector bulk by a specially designed guard ring structure. No increase of the detector's leakage current has been observed after the laser cut. Guard rings surrounds also the active region of the array toward the external border of the chip. The working temperature of the array is about -10°C, which is easily obtained with a Peltier cooler.

Fig. 3 shows a schematic cross section of the measurement head of the spectrometer. The optimum shaping time of the SDD is about 1 μs, much less than the optimum shaping time of conventional LN cooled detectors or of Peltier-cooled PIN diodes, which is of the order of 10 – 20 μs. Therefore, the achievable counting rate of each SDD of the array is more than ten times higher than the one of conventional detectors.

A microfocus tube with interchangeable anode material has been developed for this spectrometer. The principle design is shown in Fig. 4. X-radiation is generated on the anode by means of a focused electron beam (diameter < 30 μm). The angle between the electron beam axis and the anode is 58°. A monocapillary fiber (outside diameter 2 mm, inside diameter 50 μm) is integrated in the X-ray tube in order to collect efficiently the generated radiation and to transport it through the hole in the detector chip to the sample. The anode can be changed during

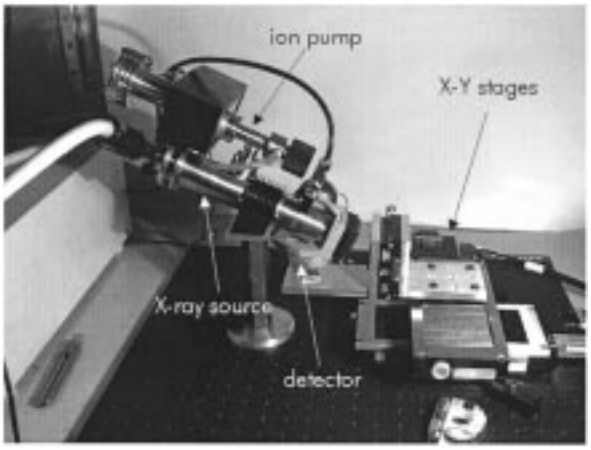

Fig. 5. Photograph of the new XRF spectrometer.

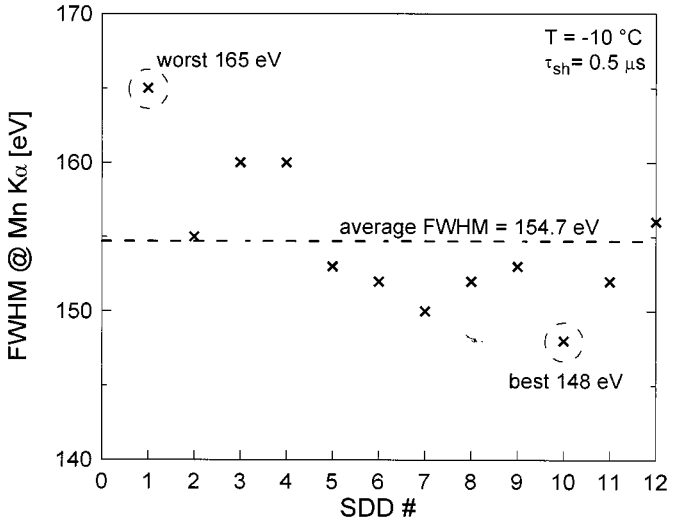

Fig. 6. Energy resolution at each detector cell measured as the FWHM at the Mn Kα line when irradiating the detector with a ^{55} Fe radioactive source. During the spectroscopic test all detector cells are biased and active, one cell at a time is read out with commercial electronics.

the operation of the generator. The operating voltage is up to 37 kV with 1.7-mA anode current.

The detector array, the X-ray tube, and the optical fiber are assembled in a compact enclosure, vacuum tightened, with a Beryllium window, as shown in Fig. 5. An ion pump guarantees the vacuum level required to operate the X-ray generator ($\cong 5 \times 10^{-7}$ mbar), which allows safe operating conditions also for the Peltier-cooled detector.

Twelve independent readout channels constitute the read-out electronics that has been specifically developed for this spectrometer [9]. Each read-out channel consists of a shaping amplifier, a baseline restorer, a peak stretcher, a 12-bit A/D converter, a pileup rejector, and control logic. Each channel is interfaced through a common signal bus to the parallel port of a personal computer for the acquisition and for the handling of the spectra. The acquisition rate of the readout system is up to now limited to 300 kcounts/s by the parallel port protocol used for the comunication to the host PC.

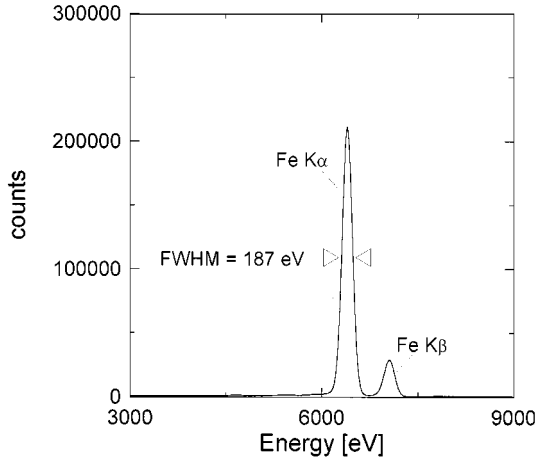

Fig. 7. Spectrum obtained by summing the 12 spectra independently and simultaneously collected by irradiating a sample of pure iron (99.99%) with the X-ray generator of the spectrometer equipped with Cu anode. Data acquisition and processing is performed with the 12-channel electronics. The detector operating temperature is about -10°C and the shaping time is 1 μs.

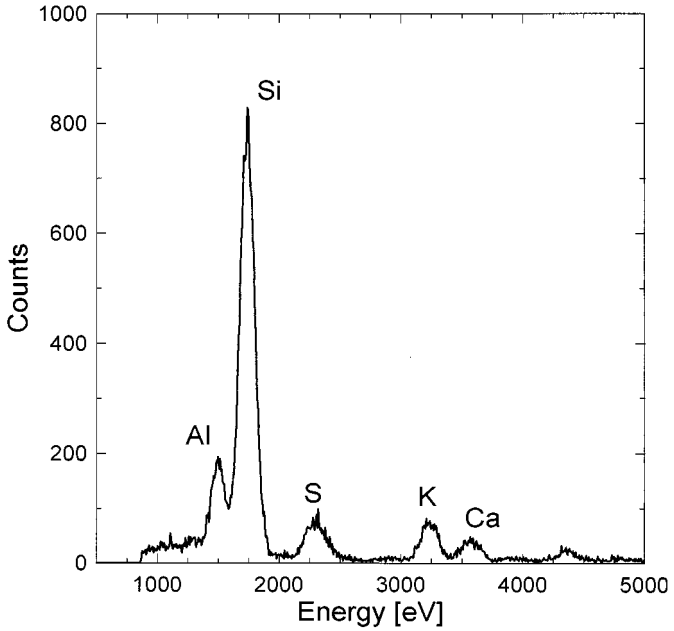

Fig. 8. Spectrum obtained by irradiating a tile, the separation between the Al and Si Kα lines is clearly visible.

III. Experimental Results

The detector was irradiated with a ^{55} Fe radioactive source to measure the achievable energy resolution. The average energy resolution of the 12 detector cells measured at the Mn-Kα line (5.9 keV) is 154.7 eV, as shown in Fig. 6. The specified values of the measured full-width at half-maximum (FWHM) demonstrate the high degree of homogeneity in the response of the individual detector cells. The obtained energy resolution is within the range known for the single unit SDDs, proving that the properties of one detector cell do not "suffer" from the integration in a multicell SDD.

Fig. 7 shows the spectrum obtained by summing the 12 spectra independently and simultaneously collected by irradiating a sample of pure iron (99.99%) with the X-ray generator

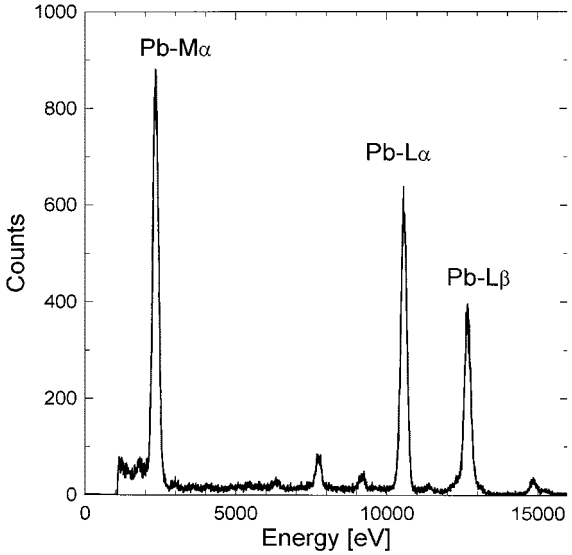

Fig. 9. Spectrum of obtained by irradiating a Pb sample. The enhancement of the lower energy Pb-Mα line with respect to the higher energy Pb-Lα and Pb-Lβ lines is clearly visible.

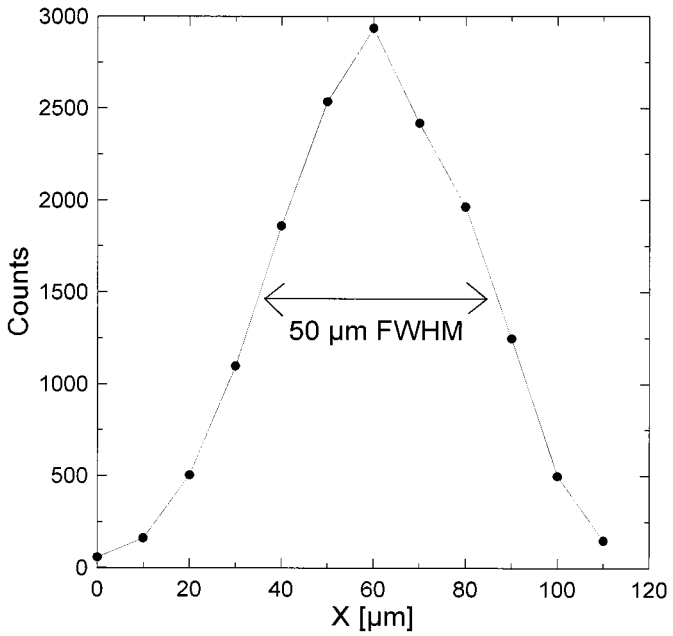

Fig. 10. Integral of the counts under the Au L lines as a function of the wire position when a gold wire of 50 μm diameter has been scanned by the excitation beam in a direction perpendicular to the wire axis.

of the spectrometer equipped with Cu anode. All the detectors were biased at the same set of voltages, chosen as a compromise of the best biasing condition for each detector (no individual adjustment of each SDD was performed). The average count rate at each detector channel was about 4500 counts/s.

The performance of the spectrometer at low energy is shown in Figs. 8 and 9. The clear separation between the Al and Si Kα lines is obtained thanks to the good energy resolution of the spectrometer in the low energy range. In Fig. 9 we can observe that the Pb-Mα line is relatively intense with respect to the Pb-L lines. This is due both to the short distance between the sample and the detector achievable with this spectrometer that allows

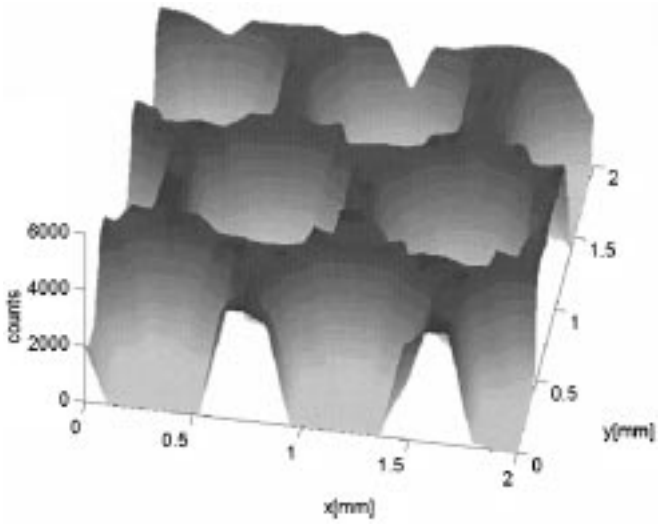

Fig. 11. Mapping of the nickel distribution in the sample described in the text.

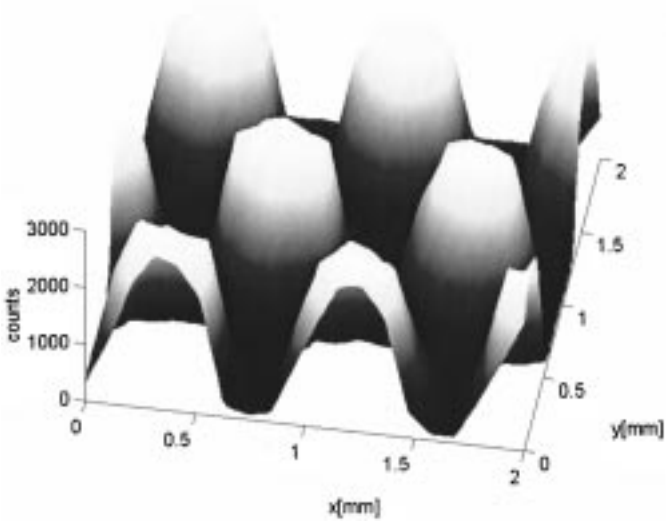

Fig. 12. Mapping of the iron distribution in the sample described in the text.

to reduce air absorption and to the focusing effect of the X-ray capillary, which is pretty high at low energy.

Fig. 10 demonstrates the spatial resolution achievable with the detection system presently equipped with a capillary fiber with an inner diameter of 50 μm. A gold wire of 50 μm diameter has been scanned by the excitation beam in a direction perpendicular to the wire axis. The wire was at a distance of 1.5 – 2 mm from the Be window of the spectrometer. The figure shows the integral of the counts under the Au L lines as a function of the wire position. The width of the curve, approximately triangular in shape, indicates that the profile of the X-ray beam is approximately rectangular with a width of about 50 μm.

Figs. 11 and 12 are an example of elemental mapping obtained with this spectrometer. A 2×2 mm area of a nickel wire gauze (the one of the blade of an electric shaver) put over an iron foil (99.99%) has been scanned with the spectrometer and the fluorescence has been sampled in 20×20 points. The measurement at each point lasted 5 s.

IV. Conclusion and Perspectives

We presented the results obtained with the first prototype of a spectrometer based on a compact assembly of a monolithic array of 12 SDDs, an X-ray capillary collimator, and on a microfocus interchangeable anode X-ray generator. The average energy resolution of the detector array is about 155 eV at the Mn Kα line. The detector showed high degree of uniformity in the behavior of the single cells making possible to use a single set of biasing voltage for all the detectors without individual trimming. The measured maximum count rate is about 4500 counts/s (limited by the capillary fiber) and the position resolution is about 50 μm (limited by the capillary fiber). The substitution of the fiber with a polycapillary lens will improve the position resolution to about 20 μm. The improvement foreseen on the intensity of excitation is of the order of 100. Moreover, a mixed analog/digital VLSI chip for the read-out electronics will be designed in view of the development of a compact transportable spectrometer based on the presented concepts. A large number of applications of this instrument are foreseen, from material analysis in the microelectronic industry or in archaeometry [10] to biological studies and to forensic investigations.

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