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NOTES

Thin windows for flow proportional counters in x-ray fluorescence spectrometers

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Notes on experimental technique and apparatus

from the anode of the photomultiplier is fed through an inverting amplifier to a Schmitt trigger which is sensitive to a preset current level corresponding to points A and B in figure 1. The output of the Schmitt trigger controls a number of flip-flops and gates, which in turn control the direction of the stepping motor, the counter and the pulse rate. The system is started by resetting flip-flops FF1 to FF4 by means of a push-button. The motor shifts the slit towards the left of the spectral line. When the photomultiplier current drops below the value at point A the counter is reset by the action of the Schmitt trigger, flip-flop FF_1 and gate G_1 , causing R of the counter to be positive. A time delay of 5 μ s (mono) ensures that the reverse command and the JK command do not coincide. The stepping motor reverses its direction, moving the slit into the line and when point A is sensed the counter starts counting upwards until point B is reached which has the same light level or current intensity as point A. The content of the counter is stored by keeping its JK inputs positive while the reset command is blocked by FF_1 and G_1 . After the time delay (mono) the counter and the motor reverse direction. The slit is again shifted into the line from the right. The counter starts counting down while the motor moves at only half the clock frequency which is derived from flip-flop FF5. When the counter reaches zero the control of the clock pulses will be interrupted by the flip-flops FF_3 and FF_4 and the gates G_3 and G_4 .

4 Components

Integrated circuits have been used in the design of the electronic circuit: 9 JK flip-flops MC 790P; 4×2 input NOR gates MC 724P; (also used as inverters); 2×4 input NOR gates MC 725P; 1 operational amplifier Burr Brown 3009-15C. Other components are: a 12 bit binary reversible counter (using integrated circuits, the construction of this counter is fairly simple, and the cost low); a Philips stepping motor (AU 5105-80), resolution $7\frac{1}{2}^{\circ}$, together with driving unit (2P 72786); a micrometer, $625 \ \mu$ m per revolution – together with an additional gearbox with a 5:1 ratio, this gives a horizontal slit movement of $2 \cdot 6 \ \mu$ m per step.

5 Discussion

In most cases of misalignment the slit will still be inside the line profile. Should the slit be outside it must be brought back to an arbitrary position inside the line either manually or by means of an independent control of the motor before the system can be used. The step resolution of 2-6 μ m in the arrangement described has proved to be sufficient for a 3.4 m Ebert direct reading spectrometer. The resolution and the accuracy may be increased by using a stepping motor with more steps per revolution, or by using a micrometer with a higher transmission ratio. Backlash in the transmission does not affect the accuracy because of the compensating effect when the direction of movement is changed twice at the two turning points.

The time required for the whole alignment procedure depends on the optical system used, on the widths of the entrance and exit slits, on the transmission ratio, on the frequency of the clock pulses and on the preselection of the amplitudes of the points A and B. With the arrangement described, the time for one alignment cycle is of the order of a few seconds. This time may be reduced by changing any one of the parameters mentioned.

References

van der Piepen H Schroeder W W and Jacobs P P J 1966 J. Sci. Instrum. 43 597-8

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Thin windows for flow proportional counters in x-ray fluorescence spectrometers

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Abstract Makrofol KG, the polycarbonate of 4.4'-dihydroxydiphenyl-2.2'-propane, which can be obtained as 2 μ m thick film, is suitable for flow proportional counter windows. When correctly fitted it is durable and, of commercially available films, has the largest transmission for the K-radiation of the light elements, Z = 11-16.

Flow proportional counters with thin windows are required when analysing for light elements (Z=11-16) by x-ray fluorescence spectrometry. The window material must transmit as much of the incident radiation as possible and also be durable to withstand the strains caused by the pressure difference (about 1 bar) between the gas-filled counter and the evacuated spectrometer chamber. Various materials have been used, including Mylar films 6 μ m and $3\frac{1}{2}$ μ m thick, and 2 μ m thick polycarbonate films, all commercially available, and polypropylene, approximately 1 μ m thick. The last has to be specially prepared in the laboratory by stretching thicker sheets of polypropylene (Caruso and Kim 1968). The material referred to as polycarbonate is the polycarbonate of 4.4'-dihydroxydiphenyl-2.2'-propane, (C16H14O3)n; the variety Makrofol KG is available as film $2 \mu m \pm 10\%$ thick from Farbenfabriken Bayer AG, Leverkusen, Germany.

Of the commercially available materials, $2 \mu m$ thick polycarbonate would be expected to have the smallest absorption. Some users have found that polycarbonate films have to be replaced frequently because they soon rupture on use, but it has been found that films carefully fitted last for more than six months, in the course of which time they are subjected to many hundreds of evacuation cycles. The material used, Makrofol KG, a crystallized and longitudinally stretched cast film, is anisotropic. It is essential to fit the film so that the direction of easy splitting, which is parallel to the length by the film, is transverse to the collimator blades that support it when the spectrometer is evacuated. Also, the ends of the collimator blades must be smooth.

Because the long life of $2 \mu m$ thick polycarbonate film, when fitted as described above, makes it suitable for routine use in x-ray spectrometers, the transmission factors of commercially available thin films were measured for the K-radiation of some light elements, and compared with the calculated transmission factors of pure 1 µm thick polypropylene (table 1). The films were coated with the thin layer of aluminium needed to make them conducting when used as flow counter windows. The transmission was obtained by measuring the intensity of crystal reflected fluorescent radiation, before and after attenuation with one thickness of film, using a flow proportional counter with pulse-height selection. The pulse-height selector was set to eliminate radiation arising from higher order reflections from the analysing crystal and enough counts were obtained to produce a relative counting error of less than 2% for NaKa and less than 1% for the

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Table 1	Percentage of radiation transmitted by films	

Film	SKα (5·37 Å)	SiKα (7·12 Å)	AlKα (8·34 Å)	MgKα (9·89 Å)	NaKα (11·91 Å)	
6 μm Mylar	77	58	43	26	13	
$3\frac{1}{2} \mu m$ Mylar	88	76	66	53	37	
$2 \ \mu m$ polycarbonate	91	85	78	67	56	
1 μ m polypropylene	99	97	96	93	88	

other radiations. The mass absorption coefficients of Heinrich (1966) and a density of 0.92 (Natta and Corradini 1960) were used to calculate the transmission of polypropylene. Table 1 confirms that, of the commercially available films, 2 μ m thick polycarbonate has the largest transmission for the radiations of light elements. The measured transmission of polypropylene stretched to 1 μ m thickness is smaller than the calculated values in table 1. Henke (1965, figure 11) showed transmissions of 90% for MgK α and 84% for NaK α ; Caruso and Kim (1968) say that transmission for AlK α is greater than 80%. Possible reasons for the differences between observed and calculated values are the effect of the aluminium coating required to make the film conducting when used as a counter window, the effect of impurities in the polypropylene and uncertainties in the mass absorption coefficients. For most light element analytical problems the convenience of using commercially available 2 μ m polycarbonate films of consistent thickness and quality probably outweighs the advantage of the extra transmission of 1 μ m polypropylene films that have to be made by the user.

References

Caruso A J and Kim H H 1968 Rev. Sci. Instrum. 39 1059-60

Heinrich K F J 1966 The Electron Microprobe Ed. T D McKinley K F J Heinrich and D B Wittry (New York: Wiley) pp. 296-377

Henke B L 1965 Advances In X-ray Analysis Vol. 8 Ed. W M Mueller G Mallett and M Fay (New York: Plenum Press) pp. 269-84

Natta G and Corradini P 1960 Nuovo Cim. (Suppl.) 15 40-57 (see also Structure Reports 1960 24 p. 574)

Corrigendum

Channel multiplier life tests in ultra-high vacuum by D G Smith J. Sci. Instrum. 1967 44 1053-5

In figure 1 the mean output current was incorrectly evaluated and should therefore be ignored. The results and conclusions drawn are not affected by this mistake.