Letters to the Editor

A single crystal of YAG – new fast scintillator in SEM

The scintillator is the basic element of an electron detector in a scanning electron microscope (SEM). It transforms the energy of secondary or backscattered electrons (produced by the interaction of the primary-electron beam with the specimen) into photon energy. The generated light quanta are detected by a sensitive, spectrally adapted photomultiplier. The scintillator and photomultiplier form a detection system which should meet the following demands: high quantum efficiency, low noise, high bandwidth, large dynamic range and long lifetime. Not all scintillation materials used to date comply with these requirements.

The main disadvantage of plastic scintillators is their short lifetime (Hatzakis 1970, Pawley 1974). Lithiumactivated glass has a low efficiency and a short lifetime, too (Marshall and Stephen 1972, Pawley 1974). Single-crystal sodium iodide is not suitable because it is very hygroscopic. It can hardly be used in vacuum because of its high vapour pressure at room temperature (Faber 1967, Porter *et al* 1966). Single-crystal calcium fluoride has a long decay time (Pawley 1974), yttrium silicate powder possesses a limited lifetime (Pawley 1974), and efforts to grow a single crystal of cerium pentaphosphate to a sufficiently large size have not yet been successful (Bimberg *et al* 1975).

Pawley (1974) described yttrium aluminium garnet (YAG) activated by cerium as a promising material for scintillation purposes. Until recently polycrystalline powders or small single crystals have been used. This letter presents basic results obtained with a single crystal of YAG: Ce³⁺ of 25 mm diameter and 60 mm high from which scintillators of arbitrary shape and size can be ground. Single crystals of this size were prepared by growth from a melt in an atmosphere comprising a special gas mixture and at well correlated crystal growth rates and solid-liquid interface temperature gradients. They contain about 1% of Ce3+ acting as the activator in the garnet crystal lattice. However, cerium doping causes a yellowish colouring involving undesirable changes of the absorption spectral response which is a limiting factor for the practical applicability of large-size crystals. Therefore, the single crystal was cut into discs of 0.2-1.0 mm thickness and of up to 25 mm diameter. The lateral area of the disc and the base on which the secondary electrons are incident were carefully polished, the other base adjacent to the light pipe being mat-ground. The reason for this treatment is to improve the reflection and the penetration of light into the light pipe and to increase the efficiency by 40-60% compared to the state with both bases polished.

Absorption of light in a thick layer of the single crystal causes a reduction in efficiency, particularly in a hemispherical scintillator ('fish eye') in which the compact single crystal is especially thick. For this type of scintillator it is therefore better to use the powder produced by crushing the block single crystal, by thermal treatment and by sorting out the grains of uniform size.

The powder is deposited on a hemispherical polymethylmethacrylate substrate by the usual sedimentation technique. Further, it is necessary to cover the deposited powder with an organic film, building a carrier medium for the thin aluminium film which serves for the maintenance of the scintillator surface at +7-12 kV. The organic film causes mechanical failures which shorten the lifetime. This is a disadvantage compared with the compact single crystal. Nevertheless, the advantages of the hemispherical surface, i.e. the increased efficiency and the fact that the lifetime is shortened by no other effects, rank this scintillator among the progressive ones. Moreover, the monocrystalline form of each grain of this highly efficient basic material (YAG) is the greatest advantage.

The high efficiency and long lifetime of both types of scintillator based on YAG will become evident in comparison with those of other fast scintillators used previously.

Figure 1 shows the efficiencies and lifetimes of a plastic block scintillator, a plastic thin film, a powder scintillator

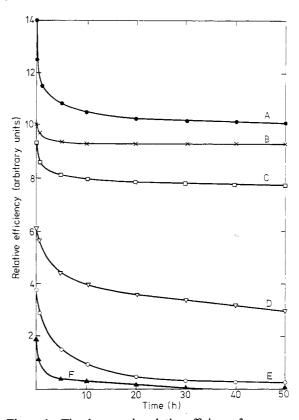


Figure 1 The decrease in relative efficiency for some scintillation materials under electron beam irradiation. Curve A, powdered $YAG:Ce^{3+}$ on hemispherical substrate; B, single-crystal disc of $YAG:Ce^{3+}$; C, powdered $YAG:Ce^{3+}$ on flat substrate; D, powdered yttrium silicate ($Y_2Si_2O_7:Ce^{3+}$) on flat substrate; E, plastic film on flat substrate; F, plastic disc.

Letters to the Editor

based on yttrium silicate $(Y_2Si_2O_7:Ce^{3+})$, usually marked as P47, and of the two types of monocrystalline YAG described here.

Measurements were made in an adapted electron microscope under the following conditions: incident beam energy E = 10 keV, current density $J = 4 \times 10^{-8}$ A cm⁻², an electron beam diameter of 3 mm, a polymethylmethacrylate light pipe, and a Tesla VUVET 61PK412 photomultiplier with S-11 photocathode ($\lambda_{max} = 460$ nm). The relative efficiencies of both types of YAG scintillator are higher with the use of a photomultiplier having an S-20 photocathode, which is more sensitive even at longer wavelengths in the visible spectrum. The wavelength maximum of the light emitted from YAG activated by cerium lies in the region of 550 nm while the other scintillators mentioned emit the light in the blue region. Nevertheless, even under these spectrally poorly matched conditions the efficiency is always higher than that of other scintillators.

However, the main success achieved is the long lifetime which is not influenced by physical changes during electron irradiation. If contamination effects are eliminated the monocrystalline scintillator disc can operate for the whole lifetime of the electronic circuit elements of an SEM. The short decay time which is less than 50 ns makes it possible to use standard television scanning frequencies.

† Institute of Scientific Instruments of	R Autrata†
the Czechoslovak Academy of Sciences	P Schauer†
Královopolská 147	Josef Kvapil‡
61264 Brno	Jiří Kvapil‡
Czechoslovakia	
‡ Monokrystaly Turnov	
Leninova 175	
511 01 Turnov	
Czechoslovakia	

22 December 1977

References

Bimberg D, Robbins D J, Wight D R and Jeser J P 1975 CeP₅O₁₄, a new ultrafast scintillator *Appl. Phys. Lett.* **27** 67–8

Faber K 1967 Nachweis von Elektronen von 2 bis 40 keV mit Szintillationskristallen

Z. Angew. Phys. 23 240-3

Hatzakis M 1970 A new method of forming scintillators for electron collectors

Rev. Sci. Instrum. 41 128

Marshall D C and Stephen J 1972 Secondary-electron detector with an extended life, for use in a scanning electron microscope

J. Phys. E: Sci. Instrum. 5 1046-7

Pawley J B 1974 Performance of SEM scintillation materials Proc. 7th A. SEM Symp. (Chicago: IITRI) pp 27–34

Porter F T, Freedman M S, Wagner F, Sherman Jr and Sherman I S 1966 Response of NaI, anthracene and plastic scintillators to electrons and the problems of detecting low-energy electrons with scintillation counters *Nucl. Instrum. Meth.* **39** 35–44 J. Phys. E: Sci. Instrum., Vol. 11, 1978. Printed in Great Britain