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SINGLE-CRYSTAL ALUMINATES - A NEW GENERATION OF SCINTILLATORS FOR SCANNING ELECTRON MICROSCOPES AND TRANSPARENT SCREENS IN ELECTRON OPTICAL DEVICES

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Abstract

Scintillators and transparent screens are described which are based on single crystals of yttrium aluminium garnet (YAG) and yttrium aluminium perovskite (YAP) activated by trivalent cerium. Their relative efficiency is compared with the powder P 47 scintillators. The detective quantum efficiency (DQE) coefficient was measured as 0.8 for YAG (versus 0.6 for P 47). Their advantage is the high resistance to radiation damage. Decay time of the luminescence is suitable for TV frequencies (YAG is 80 ns, YAP is 40 ns). It is possible to use single-crystal scintillators in devices with ultrahigh vacuum conditions without difficulty. Spectral characteristics of YAP are matched to the maximum spectral sensitivity of photomultipliers with photocathodes S 11 or S 20. The use of photocathode S 20 is preferable for YAG which has a longer wavelength of characteristic emission (560 nm). YAG can be used as a transparent screen in various electron optical devices. Its efficiency is lower than that of powder screens, but it has a high resolution. It is capable of withstanding high power loading of the electron beam. The thinnest screen produced to date is 35 μm. Other parameters are identical with those of scintillators. YAG and YAP single crystals can be machined in different forms suitable for implementation of the backscattered electrons (BSE) detector. A double detector has been developed for secondary electrons (SE) or BSE detection which is connected to one photomultiplier. The detector makes it possible to choose detection of signal electrons of different types and can be used in any SEM.

KEY WORDS: Scintillator, single-crystal, yttrium aluminium garnet, yttrium aluminium perovskite, transparent screen, secondary electrons, backscattered electrons.

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Introduction

A scintillation photomultiplier system[12] is most usually used for detection of secondary and backscattered electrons (SE, BSE, respectively) in a scanning electron microscope (SEM). In comparison with other systems, such as the semiconductor detector diode and channel multiplier, the scintillator/photomultiplier system is the most effective detector of signal electrons in SEM, but to work properly, all its components (scintillator, light-guide, photomultiplier) must be carefully chosen to ensure high efficiency, high DQE coefficient, large bandwidth, large dynamic range, long lifetime and applicability in ultrahigh vacuum conditions. The scintillator seems to be the least perfect element in the system while the light-guide and the photomultiplier can be chosen from a wide assortment of commercially produced parts or they can be easily adapted.

The performance of the scintillator/photomultiplier system was the subject of interest of many laboratories[15,18,23,30]. The most complete survey of all aspects considered in this field was presented by Pawley[19]. He pointed out the importance of noise properties of scintillator detectors, degradation of plastic scintillators and suggested possible progress in the use of phosphor P 46 (yttrium aluminium garnet). Comins et al.[8] introduced the DQE coefficient of a scintillation system according to[10] and studied the properties of scintillators based on powdered yttrium silicate activated by cerium (see phosphor P 47) — considered as the best one to date — also later studied by Baumann[4] and Volbert[29]. Reimer[20,21] described the possibilities of using the powder scintillator P 47 in modified scintillation photomultiplier systems in order to obtain new information on the studied object.

In the present paper basic properties of a new generation of scintillators based on single crystals of yttrium aluminates are described and a comparison with the other scintillators used to date is made.

Among yttrium aluminates it is yttrium aluminium perovskite (YAP:Ce^{3+}) and yttrium aluminium garnet (YAG:Ce^{3+}) activated by trivalent cerium which possess the best scintillation properties. Some advantages, such as low phosphor noise, high resistance to radiation damage, long lifetime and possible application in ultrahigh vacuum conditions, can be ascribed to the monocrystalline structure of these materials. The efficiency is comparable with that of powder phosphor P 47 and short luminescent decay time has been achieved by special techniques of single crystal preparation which are different from those used by Robbins[22].

Comparison of scintillation materials

Early SEMs used plastic scintillators[11]. A disadvantage of this type of scintillator is a relatively fast decrease of efficiency with total collected charge. At present, plastic scintillators have been almost completely replaced by inorganic powder scintillators using yttrium silicate activated by trivalent cerium (P 47)[4,8,29]. It has a high quantum efficiency of 6 %[14] and fast decay time of luminescence ($\tau_{1/e}$) of 50 ns[19]. Powdered yttrium aluminium garnet activated by trivalent cerium (phosphor P 46) is also used to a smaller extent.

Inorganic powder scintillators are less suitable for applications in ultrahigh vacuum conditions. They always require a flat light-guiding substrate and it is difficult to adapt their form for use in backscattered electron detectors[28]. Moreover, they contain a binder and often a surface organic film that degrades with use, shortening its lifetime. On the other hand, they have a sufficiently high efficiency, short decay time, are spectrally well matched to the photomultiplier and are cheap.

scintillator	structure	charact. emission nm	radiant efficiency %	decay time[1] ns	lifetime[2] h.	DQE coefficient[3]	reference
plast. NE 102 A	amorph. block	416		2.4	0.9	0.37	19 15, 8
plast. film	amorph. film	416		2.4	10	0.45	19 8
glass (Li) Ne 901 (OS1)	amorph.	395		75	10 >230	0.10	19 18
anthracene	single crystal	447		30			13
CaF_2 (Eu)	single crystal	435		1000	5×10^3	0.22	19
CeP_5O_{14}	small crystals	335		12			6
P 47	polycryst. powder	380– –415	6-8	50	100	0.39 0.6-0.7 0.25-0.39	19 4 14, 8
P 46	polycryst. powder	560	4	70 75	2×10^4	0.28	7 19
YAG:Ce^{3+}	single crystal	560		80	$>10^4$	0.8	1 this paper
YAP:Ce^{3+}	polycryst. powder	380	7	30			28
YAP: Ce^{3+}	single crystal	380		40	$>10^4$		2 this paper

Tab. 1. Comparison of various scintillation materials. 1 - decay time (a time interval after stopping excitation during which the emission intensity decreases to 1/e value, e being the base of the natural logarithm). 2 - lifetime (time to reach 50 % of the original efficiency at 5×10^{-9} electron beam current). 3 - DQE (detective quantum efficiency of Everhart-Thornley detector).

Some other scintillation materials such as glass activated by lithium[18], calcium difluoride[19], cerium pentaphosphate[6], phosphor P 15[16] and anthracene[13] were also investigated. However these materials are not advantageous for use in SEM for various reasons. A survey of basic properties of some scintillators is given in Table 1. Parameters listed there may serve for basic orientation when a suitable scintillator is to be chosen. It is true that the data of the authors may be influenced by the quality of materials used, by technology available for their treatment, by optical properties of detectors, by method of measurement and the like. Nevertheless, the table shows clearly that powder P 47 and single crystals YAG:Ce^{3+} and YAP:Ce^{3+} are the most suitable materials for use as SEM scintillators. The luminescent efficiencies of YAG:Ce^{3+} and YAP:Ce^{3+} are only known for their powder form. In comparison with phosphor P 47[14] the value of YAP:Ce^{3+} is slightly higher[27], the value of YAG:Ce^{3+} powder (phosphor P 46) is by one third lower[7]. With regard to the structure and purity of single crystals it may be supposed that their radiant efficiency will be higher than that of their powder form.

Preparation of single crystals

Yttrium aluminates - is a general designation for compounds of yttrium, aluminium and oxygen. From among them, yttrium aluminium garnet (YAG) - $Y_3Al_5O_{12}$ and yttrium aluminium perovskite (YAP) - $YAlO_3$ are of the greatest importance for scintillation purposes. Both the single crystals are activated by trivalent cerium. Among elements of rare earths cerium has the shortest decay time.

Single crystals YAG:Ce^{3+} and YAP:Ce^{3+} are grown by the Czochralski method in a protective atmosphere of 98 % Ar and 2 % H_2 in a molybdenum crucible. For example, YAG ($Y_{3-x}Ce_xAl_5O_{12}$, where $x = 0.015$) is grown from a melt containing oxides in the ratio $(Y + Ce)_2O_3 : Al_2O_3 = 2.95 : 5$ up to $3.03 : 5$. Cerium doping is advantageous up to concentration 3×10^{-2} at %. At higher concentrations of Ce^{3+} ions, the distribution coefficient decreases rapidly and the cathodoluminescent efficiency does not increase any more[3]. The growth rate is 1.5 mm h^{-1}, the temperature at a distance of 5 cm above the melt level is 1690 ± 30 °C. The grown single crystals are 10 - 15 cm long and of 30 - 40 mm diameter. If the protective atmosphere contains water vapour (1×10^{-2} vol. % of H_2O) the single crystals will contain molybdenum ions. The ions of iron and other impurity dopants if present in the single crystal are removed by additional temperature treatment[17].

Conditions of perfect growth of scintillation single crystals are: 1. The single crystal must be grown from a melt with an excess of Y^{3+} ions. 2. The concentration of impurity dopants in the single crystal must not exceed 10^{-4} wt %. Fe, Ir, Pt, Cr are considered as the most effective luminescence quenchers. 3. The concentration of Ce^{3+} ions in the single crystal should be higher than 5×10^{-3} wt % . The reason for this condition is to suppress formation of quenching colour centres and to achieve a perfect structure growth of the single crystal. 4. A trace amount of Mo influences luminescence efficiency favourably. 5. Parameters of luminescence are improved by additional treatment of the single crystal in an atmosphere of hydrogen or oxygen.

Additional treatment of the single crystals plays a very important part in improving their scintillation properties. In the case of a scintillator for SE detection it is implemented as follows: An as grown single crystal, e.g. YAG:Ce^{3+}, is cut into discs of 0.5 - 1.0 mm thickness and of up to 25 mm in diameter. One base of the single-crystal disc is carefully polished, the other one is mat-ground. The discs are then annealed in oxygen at a temperature of 1500 °C for 10 hours. As a result of this treatment the stable colour luminescence centres decompose. This effect causes a slight decrease of luminescence efficiency on the one hand but on the other hand it shortens the decay time from the initial 150 ns to the final 80 ns. The increase of luminescence efficiency is achieved by treating the discs in a moisture hydrogen atmosphere in a closed molybdenum crucible. The Mo ions, probably in a reduced Mo^{3+} state, increase the luminescence yield of Ce^{3+} ions considerably. The disadvantage of the hydrogen treatment is the prolongation of the decay time up to 180 ns. For these reasons it is necessary to introduce a certain optimum between the described ways of treatment.

The optical matching of the single-crystal scintillator with regard to reduction of light losses during light transfer from the scintillator into the light-guide was discussed in detail in our previous paper[24].

Spectral properties

The single-crystal scintillator used in the Everhart-Thornley detector performs two important functions simultaneously. It is an efficient source of the light signal in an appropriate wavelength region and a light-guide which does not absorb light just in the wavelength region of its own emission. For this reason emission and absorption spectra of single-crystal scintillators are of great importance not only for determination of the physical model of luminescence, but also for the considered application.

Absorption and emission cathodoluminescent spectra of single-crystal scintillators of YAG:Ce^{3+} and YAP:Ce^{3+} are shown in Fig. 1. The emission spectra were measured at pulse incident beam of energy 10 keV and of current density 4×10^{-8} A cm^{-2}. The light emitted by the single crystal was detected after passage through a mirror monochromator by means of a photomultiplier (EMI 9558) and a lock-in nanovoltmeter. The curves of the emission spectra are corrected for the spectral sensitivity of the photomultiplier used and normalized to their maxima. The absorption spectra were measured with a spectrophotometer Specord UV VIS (VEB Carl Zeiss Jena). They are corrected for reflectance and shown as light transmission in Fig. 1 .

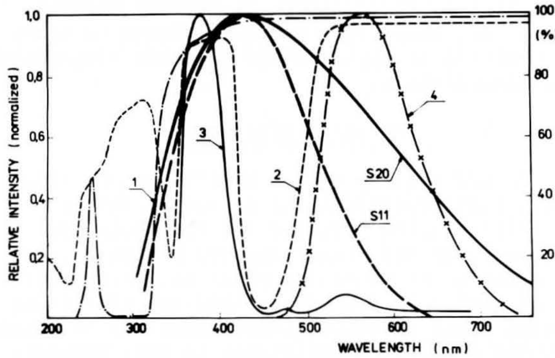

Fig.1. Absorption and emission spectra of single-crystal scintillators with cathode radiant sensitivity of used photomultipliers (S 11, S 20). 1 - transmission of YAP:Ce^{3+}, 2 - transmission of YAG:Ce^{3+}, 3 - emission of YAP:Ce^{3+}, 4 - emission of YAG :Ce^{3+}.

The absorption spectrum of YAG:Ce^{3+} shows a broad absorption band with its maximum at 460 nm and a narrow band at 340 nm. The absorption spectrum of YAP:Ce^{3+} has only one absorption band lying at energies close below the absorption edge (260 - 330 nm). The emission spectrum of YAG:Ce^{3+} is characterized by an outstanding broad emission band (FWHM 175 nm) with the maximum at the wavelength of 565 nm. The characteristic emission band of the single crystal of YAP:Ce^{3+} lies at the boundary of the ultraviolet and the visible spectrum region with the maximum at the wavelength of 378 nm and FWHM of 50 nm.

From absorption and emission spectra shown in Fig. 1 the value of the so-called self-absorption which precisely means absorption of its own emission can be determined. The knowledge of this value is important especially for applications in which the emitted light passes through a thick layer of a scintillator as e.g. with a certain type of detector of backscattered electrons. The self-absorption can be evaluated by numerical integration of the product of emission intensity and the mean transmissivity along all paths of the light beam inside the single crystal under the assumption that the luminescent centres are emitting uniformly in all directions and that only that part of photons is utilized which is not incident on the side walls of the specimen[25]. The self-absorption calculated in this way amounts to 9 % with YAG:Ce^{3+} and 20 % with YAP:Ce^{3+} (specimen ø 9.6 × 0.5 mm). It follows from this that YAP:Ce^{3+} should be used in applications that do not necessitate thick scintillators.

In Fig. 1 two curves are plotted which correspond to the normalized radiant sensitivity of photocathodes S 11 and S 20 of the photomultiplier. Photocathodes of these types are most frequently used in the scintillation photomultiplier system of SEM. It can be seen from their shape and from the shape of emission characteristics of the considered single crystals that it is the YAP:Ce^{3+} scintillator which is well matched to both types of photocathodes, while YAG:Ce^{3+} necessitates using the photomultiplier with the S 20 photocathode to prevent greater losses. All these circumstances should be taken into account in applications of these single-crystal scintillators.

Relative efficiency

A sufficiently high scintillator efficiency is the basic condition for a good performance of the scintillation detector and the whole SEM. The efficiency of the transformation of energy, electron-photon, is generally given as radiant efficiency quoted in percentage. Radiant efficiency of some types of scintillator materials is listed in Table 1 . Radiant efficiency is not, however, a suitable quantity for comparison of scintillators' quality because in practice it does not imply technological aspects of their preparation. These aspects can be involved in efficiency expressed as a relative quantity related to a suitable frequently used phosphor. The relative efficiency comprises losses caused by optical matching, by limited spectral transmittance of the light-guide, losses across the photocathode of the photomultiplier and losses due to insufficient technological optimization of the scintillator. The values of relative efficiencies were related to phosphor P 47 (Sylvania Chemical, USA, No. 23347-20, surface density of powder was 2 mg cm^{-2}, aluminium layer 50 nm).

The comparison of relative efficiencies of single-crystal scintillators of $YAG:Ce^{3+}$ and $YAP:Ce^{3+}$ and some powder scintillators based on phosphor P 47 follows from Fig. 2 which shows the dependence of the light output signal from a scintillator on the accelerating voltage of the electron beam. Measurements were made with a continuous primary electron beam focussed into a spot of 3 mm diameter and current of 3×10^{-9} A. The light emitted by the scintillator was guided through a quartz light-guide to the photomultiplier with the S 20 photocathode. Values used for comparison were recorded after three hours from the beginning of excitation. In the graph of Fig. 2 these values are corrected for spectral sensitivity of the photomultiplier used with regard to the maximum of the characteristic emission spectra of individual scintillators. Relative efficiency of the single-crystal scintillators is aproximately comparable with the efficiency of scintillators using powder phosphor P 47 (Riedel-de Haën No. 54063) and it is higher than that of scintillators using phosphor P 47 (Sylvania No. 23347-20). The curve valid for the scintillator Planotec (PLANO, but the original scintillator was covered with an organic film and an Al layer) shows a very steep slope, however at energies lower than 5 keV its light output signal is lower than that of single crystals of $YAP:Ce^{3+}$ or $YAG:Ce^{3+}$. A still steeper slope was found with the scintillator NPRL P 47 (according to Thirlwall). The light output signal of single-crystal scintillators increases linearly with the accelerating voltage which may be even higher than 40 kV. With the powder scintillator P 47 (surface density 2 - 5 mg cm^{-2}) the line slope begins to decrease at higher accelerating voltages. The reason for this non-linearity consists in the low surface density of grains of the powder P 47 and in the decreased absorption of electrons in the phosphor. The optimum of the surface density of powder scintillators depends on the value of the accelerating voltage[9]. Single-crystal materials are highly resistant to electron radiation damage. They have a great specific density ($YAG:Ce^{3+}$ - 4.55 g cm^{-3}; $YAP:Ce^{3+}$ - 5.35 g cm^{-3}) and can be made in different thicknesses (the minimum limit is 30 µm for the time being). Their linear dependence of the light output signal on the accelerating voltage in the range of 40 - 100 kV is of great importance if they are used as transmission screens in TEM.

The efficiency of transformation of energy in the scintillator depends on the efficiency of the luminescence centre represented by an atom of trivalent cerium and on the efficiency of transfer of excitation energy to this luminescent centre or away from it. It is well known that the efficiency of transfer of excitation energy depends above all on the concentration of luminescent or defect centres. We discuss this problem elsewhere[3].

Detective quantum efficiency

For testing the quality of a scintillator it is also necessary to measure its noise properties. If the signal/noise ratio (SNR) of the detector signal in SEM or STEM is too low, the resolution of the details on the observed subject will be bad. The general effort is then to prepare a scintillator with the best possible SNR. The noise of the detector system is influenced not only by the scintillator which is a part of the chain: scintillator, light-guide, photocathode, multiplier, video amplifier. An excellent analysis of this problem was made by Baumann and Reimer[5]. The scintillator is the most important part of this chain. Only it determines how many photons will be excited from one incident electron and how effectively they will be reflected to the light-guide.

The performance of the scintillation electron detector is evaluated in terms of DQE[8], which is defined as the ratio of the square of the SNR of the output from the detector to the square of the SNR that the detector would produce under ideal conditions.

$$DQE = (SNR)^2_{out} \; / \; (SNR)^2_{ideal} \qquad (1)$$

The DQE values measured by various authors for electron detectors using P 47 scintillators differ from each other. Pawley[19] achieved the value of 0.39 , Comins et al.[8] 0.25 - 0.39 according to phosphor screen density, Baumann et al.[4] about 0.6 or 0.7, and Comins and Thirlwall[9] above 0.8, all for 10 - 20 kV accelerating potential. The reason for these discrepancies may result from different technological performances of the samples (this includes light yield differences for P 47 powders of various producers), from different measurement conditions, precision, and from different properties of the individual parts of the detection system. The results of these authors are very valuable for developing a better method of measurement[4, 9], and especially also for the relative comparison of several scintillators within one measuring device. From this point of

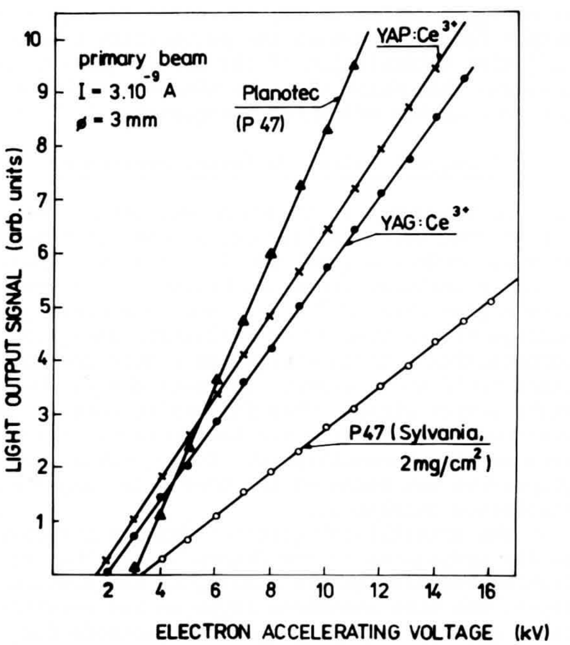

Fig.2. Light output signal of single-crystal and P 47 powder scintillators plotted as a function of accelerating voltage. I - electron beam current, ∅ - diameter of primary electron beam.

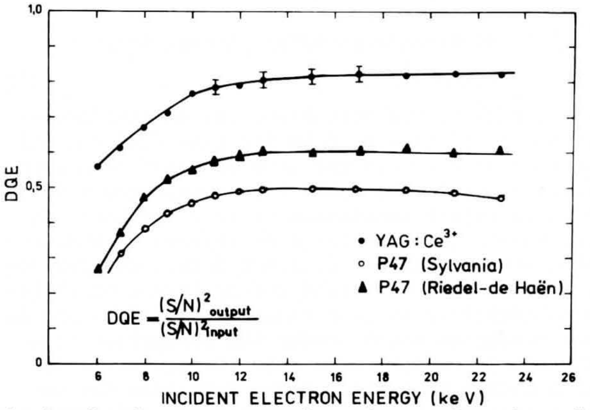

Fig.3. The detector DQE plotted as a function of the incident primary electron energy. Powder screen density of P 47 2 mg cm^{-2}.

view we can understand the aim of our measurement that is to find out the DQE value of the YAG:Ce^{3+} single-crystal scintillator compared with the powder P 47 scintillator within identical measurement conditions.

The experimental arrangement used for DQE measurement consisted of a standard scintillation electron detector in an adapted TEM. The primary electron beam excited directly the scintillator inside the Faraday cage through a diaphragm. The light from the scintillator approached the photocathode (S 20) of the photomultiplier through the straight quartz light-guide (in the axis of the primary beam). A dc voltmeter, RMS voltmeter and oscilloscope were connected to the photomultiplier output. Using this apparatus it was possible to adjust the current of the incident electron beam on the scintillator, to change its accelerating voltage, then to adjust the photomultiplier gain and to read the output signal $(S)_{out}$ on the dc voltmeter. Then the RMS voltmeter showed the noise value $(N)_{out}$ in the output signal. The ratio $(S/N)_{out}$ was used for the DQE calculation. The ratio $(S/N)_{ideal}$ was evaluated from the formula:

$$(S/N)_{ideal} = (S/N)_{input} = \sqrt{\frac{I_B}{2e\Delta f}} \qquad (2)$$

where I_B = the primary beam current (1.2×10^{-10} A), e = 1.6×10^{-19} coulombs and Δf = the bandwidth of the RMS voltmeter (500 kHz). The dependence of DQE coefficient on the incident electron energy is shown in Fig. 3 . The DQE value for the single crystal YAG:Ce^{3+} is about 0.8 , while the value for phosphor P 47 (Riedel-de Haën) is about 0.6 . Although the light output signal of P 47 phosphor (Riedel-de Haën, Scintillator Planotec, Fig. 2) is for the accelerating voltage 10 kV slightly higher in comparison with the single crystal YAG:Ce^{3+}, the DQE value is lower. The reason for this effect cannot be yet precisely given. We can only assume the difference in the number of backscattered electrons, in the scintillator homogeneity, in the relative variances of the transmission probability of the photons and in the energy distribution. The resolution of this problem is the subject of our interest. The DQE value was found to be influenced at the same type of scintillation material by its quality. A strong connection between DQE and the light output signal in this case can be seen in Fig. 2 and Fig. 3 for P 47 phosphors. The DQE value of the single-crystal scintillator YAG:Ce^{3+} is dependent upon the technology of the single crystals preparation and on the way of their treatment.

Time characteristics

By time characteristics of scintillators we mean the rise time and the decay time of the cathodoluminescent process. It is the decay time which is critical when using the scintillator in a SEM. If television frequencies higher than 10 MHz are to be used this decay must be shorter than 100 ns.

Only such materials are convenient to the crystal lattices of which ions of trivalent cerium can be built-in as activators. A simple diagram of energy levels of the ground and the excited state of trivalent cerium shows the simple recombination processes and phosphors activated by trivalent cerium work very fast. However this short decay time of cathodoluminescence of many phosphors is unfortunately affected by the presence of defect centres on which energy levels carriers are being trapped during the transfer of excitation energy to the luminescent centres. Their release is characterized by a certain time constant which influences the overall rate of the cathodoluminescent process in the scintillator which results in the non-exponential course of the decay of cathodoluminescence. The time parameters of single-crystal aluminate scintillators cannot be simply characterized by a single decay time, but are determined by a multicomponent equation

$$I(t) = (1-A_1-A_2)\exp(-t/\tau_1) + A_1\exp(-t/\tau_2) - A_2\exp(-t/\tau_3) \quad (3)$$

where $I(t)$ is the normalized cathodoluminescent intensity ($I(0) = 1$), t is the time from the end of excitation, τ_1 is the time constant of radiative transitions in the activator (luminescence decay time at direct excitation of an activator), τ_2 and τ_3 are time constants of release of excitation energy carriers from the defect centres localized at two different energy levels which become effective at room temperature and A_1 and A_2 are constants which (under the assumption that the energy capture rate into the activator is much higher than the capture rate into the defect centres and the energy release rate from the activator is much higher than the release rate from the defect centres) determine the probability of excitation energy transfer to the activator through the defect centres so that the constant $1-A_1-A_2$ determines the probability of direct excitation of the activator.

The cathodoluminescent decay characteristics of the single-crystal scintillators YAG:Ce^{3+} and YAP:Ce^{3+} are shown in Fig. 4 . The results were obtained at pulse excitation using electron beam pulse duration of 10 µs and repeated frequency of 1 kHz. The decay time of the measuring device was 5 ns. Constants τ_1, τ_2, τ_3, A_1 and A_2 can be obtained from the curves using a graphical-numerical method[26]. For YAG:Ce^{3+} the following values were determined: τ_1 = 85 ns, τ_2 = 360 ns, τ_3 = 2.8 µs, A_1 = 0.23 and A_2 = 0.11. For YAP:Ce^{3+} τ_1 = 20 ns, τ_2 = 70 ns, τ_3 = 1.5 µs, A_1 = 0.29 and A_2 = 0.12. If shorter excitation pulses ($\Delta t < 10$ µs) are used constants A_1 and especially A_2 decrease since the time of excitation becomes comparable with the time of capture of excitation energy carriers into the defect centres so that the probability of energy transfer through the defect centres decreases. For the sake of comparison P 47 phosphor (Sylvania) has a single exponential decay characteristic with a constant τ = 38 ns.

If the concentration of Ce^{3+} ions in YAG and YAP increases, the probability of the direct excitation of the activator also increase and the probability of activator excitation by energy transfer through the defect centres decreases. Consequently, constants A_1 and A_2 also decrease and the decay characteristics approach more the single exponential characteristics having the decay time τ = 85 ns which is determined by the radiative transition rate in the activator. Unfortunately, the Ce^{3+} ions concentration in YAP:Ce^{3+} and especially in YAG:Ce^{3+} is limited by technology available for the preparation of single crystals. Therefore, the undesirable effect of the defect centres on the decay of YAG:Ce^{3+} and YAP:Ce^{3+} cannot be limited by an activator concentration higher than 1×10^{-2} at %. The only possibility how to limit the effect of the defect centres on the decay characteristics is to anneal the single crystals in an oxidizing atmosphere at a temperature of approximately 1500 °C. It is true that this technique does not increase the probability of direct excitation of the activator, but it decreases the probability of energy transfer to the activator through the defect centres, because the probability of non-radiative deexcitation of the defect centres increases. This means that the single crystal cathodoluminescence efficiency decreases.

Electron radiation damage resistance

To be useful as an SEM scintillator a material must have a sufficiently long lifetime. It must produce a light yield even after irradiation by incident electrons having current density higher than 10^{-8} A cm^{-2} and accelerating voltage higher than 30 kV. Radiation damage of a scintillator becomes evident as a decrease in its light yield which results in lower SNR in the light output signal. This failure is usually compensated by increasing the beam current or the gain on the photomultiplier. Consequently, noise properties get worse or the beam gets larger and resolution decreases.

The scintillator lifetime depends not only on its resistance to the damage by electron radiation but also on the quality of the aluminium layer. The thin aluminium layer on the scintillator surface is employed as an electrode for acceleration of secondary electrons. It prevents formation of the static charge during detection of backscattered electrons and reflects the emitted photons in the direction to the light-guide. This last effect is more important with the smooth surface of single crystals than with coarse surfaces of powder scintillators.

Scintillator lifetime depends on parameters of incident electrons and on their current density and accelerating voltage. The evaluation of radiation dose is complicated by non-uniform energy distribution of electrons in the penetration depth of the scintillator and by the fact that the electron flux is not uniformly distributed over the surface of the scintillator, especially if a hemispherical scintillator is used. At specimen current of 10^{-11} - 10^{-9} A as is usually used in SEM the radiation doses rates range within 10^{-1} - 10^{1} Mrad h^{-1}. Pawley[19] showed that doses of this range cause a rapid shortening of lifetime of plastic scintillators especially. At 1 Mrad h^{-1} the efficiency of plastic scintillators decreases to 50 % of their initial value after one hour. In some applications, for example electron channeling patterns, electron beam lithography or scanning transmission microscopy, currents of 1×10^{-7} A and higher are used. The proportionally higher damage rates require frequent replacement of scintillator.

Glass and powder scintillators are not damaged by ionization caused by the impinging electrons and therefore show a higher resistance to electron beam damage than the plastic scintillators. Powdered phosphors such as P 47 possess a good radiation resistance, but low mechanical resistance. Powdered scintillators are covered either with an aluminium foil or a thin aluminium layer deposited on an organic film. Due to frequent alteration of vacuum-air medium and due to contamination, the aluminium cover may easily be injured and the scintillator damaged. An exception is the powder scintillator P 47 (Plano) working without any aluminium cover.

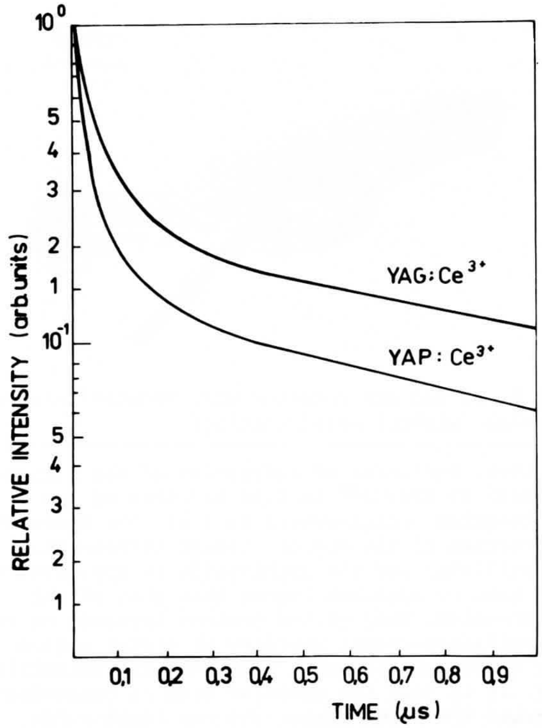

Fig.4. Luminescent decay characteristics of single-crystal scintillators. Excitation pulse duration 10 μs, concentration of Ce^{3+} ions 1×10^{-2} at %. Treated in oxygen and hydrogen atmosphere at 1500 °C, 3 h .

The lifetime of powdered scintillators containing the phosphor P 47 depends on the current density of the excitation electron beam. Using current densities of 1×10^{-5} A cm^{-2} at our measurements the light output signal decreased by as much as 50 % in the first three hours. During the next hours the decrease is shown as can be seen from Fig. 5 . At lower current densities (3×10^{-7} A cm^{-2}) or at current densities frequently used in SEM the situation is more favourable. In the first 5 hours the decrease amounted to 13 %, the situation for the next hours is shown in Fig. 5 . 50 % light output signal was recorded after approximately 200 hours. The overall performance depends on the working conditions of the SEM.

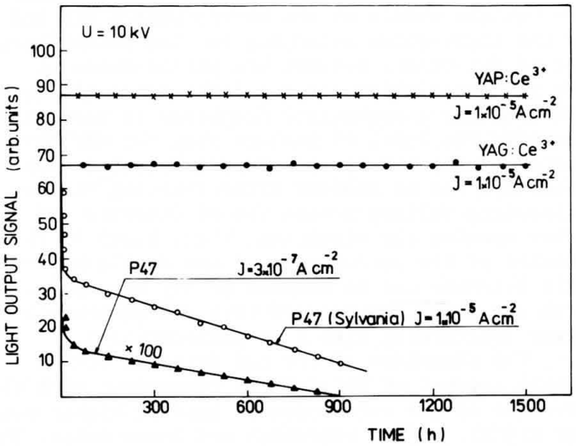

Fig.5. Light output signal versus excitation time of electron primary beam with 10 keV energy. J - current density of electron primary beam incident on the scintillator, U - accelerating voltage.

Single-crystal scintillators have excellent resistance to electron beam damage. The crystal lattices of YAG and YAP are so resistant that practically no decrease appears in the course of some thousands of hours using incident primary beam energy of 10 keV and current density 1×10^{-5} A cm^{-2} (see Fig. 5). In the first three hours the light signal output of the single-crystal scintillator decreases by 2 - 3.5 %, depending on the way of additional treatment of the crystal, but afterwards there is no further change. A 2 % decrease recorded after approximately 5000 hours is rather due to contamination effects. If the aluminium layer on the upper polished base of the scintillation disc is contaminated, aluminium can be dissolved in a 20 % solution of potassium hydroxide and the scintillator recoated with 50 nm of aluminium. In this way a scintillator can be used indefinitely. Powder scintillators cannot be recoated easily.

Application of single crystals of $YAG:Ce^{3+}$ and $YAP:Ce^{3+}$

Transmission cathodoluminescent screens

The single crystal of $YAG:Ce^{3+}$ emitting green-yellow light is a suitable material for transmission cathodoluminescent screens. For this purpose discs of 35 μm - 0.5 mm thickness and 10 - 40 mm diameter (Fig. 6) were cut from the single crystal. The disc dimensions can be chosen with regard to the intended application. Their relative efficiency is half of the one of the high-quality transmission powder screen (EMI 214, 1 mg cm^{-2}, particle 1 - 2 μm), but it is three times higher in comparison with the vacuum deposited transmission screens (e.g. $ZnSiO_4:Mn$). The light output signal of $YAG:Ce^{3+}$ is mainly determined by the primary electron beam voltage and its current intensity and is not affected by the thickness and size of grains as in the case of powder screens. The dependence of light output signal on the accelerating voltage of the electron beam shown in Fig. 2 is also valid for $YAG:Ce^{3+}$ screens.

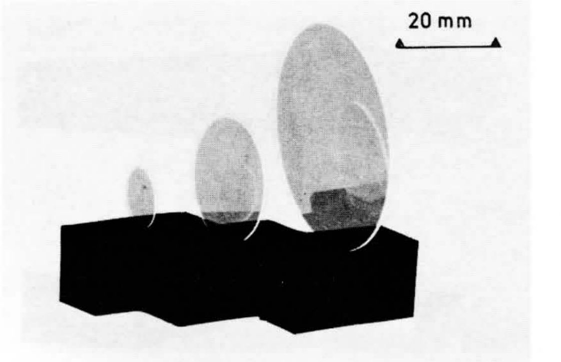

Fig.6. $YAG:Ce^{3+}$ single-crystal transparent screens.

The advantage of using a single crystal as a TEM screen is its extremely high resolution which is dependent only on the electron scattering in the single-crystal phosphor ($YAG:Ce^{3+}$ density- 4.55 g cm^{-3}, $YAG:Ce^{3+}$ refractive index - 1.84 at 560 nm) that can be retained during further optical process of imaging. For instance, the image can be recorded photographically by focussing through the screen. Different types of converters of the electron image using conventional optics can also be designed without special requirements on the screen thickness. However, if used in connection with fibreplates the screen must comply with certain demands. On the basis of evaluation from indexes of refraction and angles of reflection it can be found that for a fibreplate of 10 μm fibre diameter a 10 μm thick screen is suitable. For the time being it is still technologically difficult to achieve this thickness. We succeeded in producing a 30 μm thick screen of 20 mm diameter in a mechanical way.

Scintillators for BSE detection

Single crystals of $YAG:Ce^{3+}$ and $YAP:Ce^{3+}$ like a plastic scintillation material can be machined into arbitrary forms. The lifetime of plastic scintillators is limited by incident electron radiation damage, the lifetime of single-crystal scintillators is unlimited. Therefore, they can be used with advantage for BSE detection in an arrangement similar to that suggested by Robinson[23] and also used by Thirlwall and Comins for powder phosphor P 47[28].

Several types of detectors with single-crystal scintillators were made, having for example 2 × 2 cm surfaces and with fibre optics light-guides with a possible working distance of 4 mm, with two built-in detectors intended for recognizing various contrast mechanisms which can be moved from the horizontal position into the vertical one. In Fig. 7 and 8 two detectors in a non-metallized state are shown. Owing to their simple construction and properties they could be of interest for users of SEM.

Fig. 7 shows a BSE detector with a single-crystal scintillator of $YAG:Ce^{3+}$ in the form of a 3 mm thick disc with a hole of 3 mm diameter in the middle allowing the passage of primary electrons and with a light-guide cemented on one half of the side wall of the disc. In this type of arrangement transfer of photons from the scintillator into the light-guide causes some difficulties. The index of refraction of the single-crystal of $YAG:Ce^{3+}$ is 1.84 and that of the light-guide (acryl-resin) is 1.52. The index of refraction of the optical cement between the scintillator and the light-guide is approximately the same or somewhat higher than that of the light-guide. Many of the photons incident on the scintillator-cement boundary at higher angles are therefore reflected back into the scintillator. It is just the circular form of connection between the scintillator and the light-guide which reduces the number of photons incident at a high angle onto the boundary and increases the probability of their transfer into the light-guide. The detector is closely pressed under the polepiece so that the object can be viewed at a working distance of as little as 4 mm.

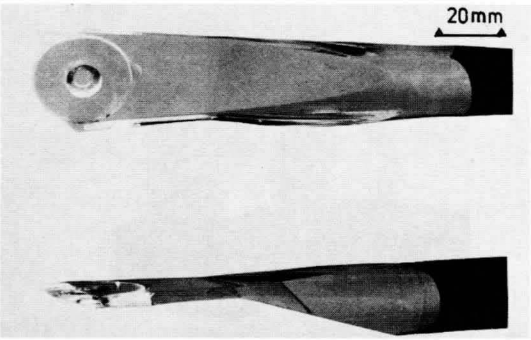

Fig.7. BSE detector with $YAG:Ce^{3+}$ single crystal (without metal coating).

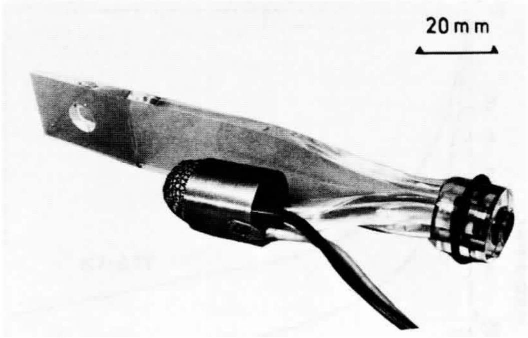

Fig.8. SE and BSE detector with $YAG:Ce^{3+}$ single crystal (without metal coating).

Fig. 8 illustrates a double detector with single crystals of $YAG:Ce^{3+}$ used for SE or BSE detection with one photomultiplier. The BSE detector is composed of a single-crystal square plate of $YAG:Ce^{3+}$ on whose side wall an interference layer of λ/4 thickness from Al_2O_3 is deposited to ensure a better transfer of photons from the scintillator into the light-guide. It is cemented onto a flat light-guide forming a circle in the direction of the photomultiplier. Through the middle of the circle passes the rod of the light-guide belonging to the scintillator for SE detection. Between the photocathode of the photomultiplier and the light-guide of the BSE detector a mechanical diaphragm is used which prevents the inlet of photons from the BSE detector at the moment when SE are detected. BSE detection can be started after removing the accelerating voltage across the SE detector and after opening the diaphragm. Figs. 9 and 10 show results of the performance of the double detector. This detector can be adapted to any microscope with one video channel and with one photomultiplier (preferably with S 20 photocathode).

The advantage of the BSE detector with a single crystal of $YAG:Ce^{3+}$ in comparison with the detector of the semiconductor type is higher output signal, higher bandwidth and lower noise. It operates well at specimen currents similar to those used in SE detection, it can be employed to give an image with similar SNR characteristics. It enables us to use a very short working distance with simultaneous SE detection. The improved

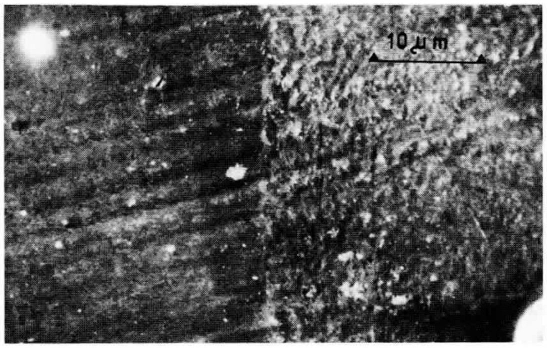

Fig.9. Secondary electron image. Left-hand side - copper, right-hand side - Cu-Zn alloy, specimen current 1×10^{-10} A, accelerating voltage 10 kV, tilt 0°, polished surface.

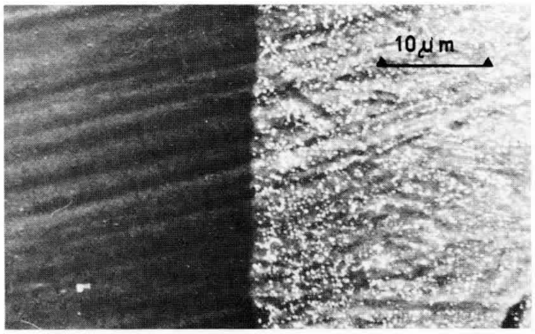

Fig.10. Backscattered electron image. The same position as Fig. 9 . Left-hand side - copper (atomic number 29), right-hand side - Cu-Zn alloy (mean atomic number 29.2), specimen current 1×10^{-10} A, accelerating voltage 25 kV, tilt 0°.

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construction, high light output signal and low noise properties give images with displayed resolution similar to that which is achieved with the SE detector. It has a long lifetime, TV scan speed capability and simple switch-over observation of both the normal SE and the BSE image.

Fig. 11 shows a drawing of the BSE detector with a single crystal of YAP:Ce^{3+} which is more appropriately adapted to a photomultiplier with any photocathode. The light transfer from the scintillator into the light-guide is here better solved, which increases the light output signal of the detector.

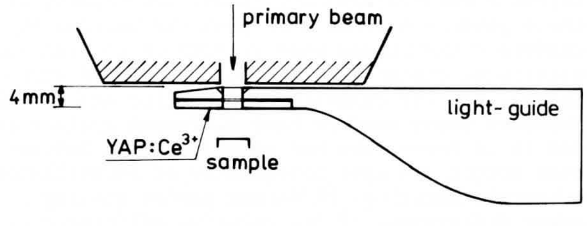

Fig.11. BSE detector with YAP:Ce^{3+} single crystal.

Acknowledgements

The authors wish to thank Professor Armin Delong, the director of the Institute of Scientific Instruments of the Czechoslovak Academy of Sciences in Brno, for his support and creation of favourable conditions for this work. Thanks are also due to Dr. Noli (PLANO, Marburg, Germany) for his providing us with the scintillator Planotec and further material help and to Dr. Thirlwall (National Physical Research Laboratory, Pretoria) for the NPRL P 47 scintillator and valuable information.

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Discussion with Reviewers

N.R.Comins and J.T.Thirlwall: Is it possible to produce P 47 phosphor in single-crystal form?
Authors: Silicates have no defined point of the hardening. The melt is of high viscosity and it is therefore very difficult to grow a single crystal by the Czochralski method. Silicates form a vitreous mass where the single-crystal structure is improbable. It is sure that it is impossible to use the reducing atmosphere reguired for the maintaining of trivalent cerium. Nevertheless, we intend to carry out experiments in this respect.

J.B.Pawley: Is the radiant sensitivity of S-11 and S-20 photocathodes shown in Fig. 1 measured with respect to quantum efficiency or energy efficiency?
Authors: Radiant sensitivity of S-11 and S-20 photocathodes shown in Fig. 1 is expressed with respect to quantum efficiency. Radiant sensitivity (mA/w) = q % λ(nm)/124, where q = quantum efficiency.

B.Volbert: In the literature (your reference 4 and 8), the optimum layer thickness is about 1.7 mg cm^{-2} for 10 keV electrons. Why does the relative efficiency of the 2 mg cm^{-2} scintillator in Fig. 2 show a linear increase? I expect a broad maximum around 12 or 14 keV.
Authors: We would like to point out, that the relative efficiency parameter in Fig. 2 was replaced by the light output signal with respect to the reviewed copy. The optimum thickness of the powder scintillator from the point of view of technology is affected by the powder grain size and its distribution, homogeneity of surface covering, quantity of binder, adhesive and transmissive properties of binder and so on. The literature suggests optimum thickness of 1.5 - 2.0 mg cm^{-2} for the energy of about 10 keV under conditions which do not specify all effects in greater technology details. Linear dependence of the light output for powder density of 2.4 mg cm^{-2} and accelerating voltage 3 - 20 kV was obtained ref. 4. Values of 2.0 mg cm^{-2} and 1.7 mg cm^{-2} differ from the point of view of other effects only slightly. Nearly linear dependence in Fig. 2 of this paper is with respect to different technological aspects in a surprisingly good agreement with the result given in the literature previously.

B.Volbert: How do you explain the large difference in Fig. 2 between the two P 47 scintillators in relative efficiency if the layer thicknesses are nearly the same?
Authors: Several P 47 powder phosphors of different manufacturers were tested. The quality of these powders differs most probably because of different techniques used (purity of initial raw materials, concentration of Ce^{3+}, applied atmosphere, time of annealing and cooling and so on). Moreover these powders have different grain size and it is known this has an effect upon luminescent output and upon homogeneity of scintillator substrate covering. Different powder quality makes differences in the relative efficiency of the scintillators even when maintaining the same

layer thickness and the same technology of scintillator preparation.

N.R.Comins and J.T.Thirlwall: In your experimental technique you adjusted the PMT gain during the process of measuring the output signal and noise to compensate for changing the incident electron beam energy. Because this adjustment must affect in some degree the relative variance of the PMT gain, as shown in the analyses of references 9, 5, it must influence the signal-to-noise ratio. Have you considered this in your measurement?
Authors: The way of measuring the PMT gain was based on the method by Pawley[19] (pp. 31 - 32) and on the method 2.), 3.), described by Baumann and Reimer[5] (p. 146). The PMT gain was adjusted for each value of the electron beam accelerating voltage, presented in Fig. 3 . The mentioned DQE values can be loaded with a measurement error. Nevertheless, our aim was to find the relation between YAG:Ce^{3+} and P 47, in which possible measurement error does not appear.

N.R.Comins and J.T.Thirlwall: In stating that the bandwidth of the RMS voltmeter was 500 kHz have you included the influence of the PMT/head amplifier and distributed capacitance on this value?
Authors: RMS voltmeter bandwidth was the only frequency limiting factor of the whole measuring equipment.

N.R.Comins and J.T.Thirlwall: You have commented, in your section on DQE measurements, on the inconsistency between the DQE value obtained for P 47 and YAG:Ce^{3+} scintillators in view of their light output signal values. Have you considered analysing the data in the form used in reference 9 (plotting reciprocal DQE against reciprocal signal) in order to attempt to identify the reason for this apparent discrepancy? If so, what conclusions could be drawn?
Authors: Plotting reciprocal DQE against reciprocal signal is linear. However we do not dare to identify the reason for the discrepancy between the light output signal and the DQE without further measurements and analysis. We have pointed out some possibilities in the text of this paper.

N.R.Comins and J.T.Thirlwall: The light output signals of the P 47 scintillators were measured after 3 hours of irradiation at a current density of 4×10^{-8} A cm^{-2} - at a time when the signals as shown by the life-test data were about 50 % of their initial values. This represents several months of typical SEM operation. Can you present DQE data measured at an earlier stage in the life of the P 47 scintillators and compare it with the YAP:Ce^{3+} material?
Authors: DQE data measured after 15 minutes of the lifetime remain the same for YAG:Ce^{3+} as shown in Fig. 3 . In comparison with the initially commented value (P 47 Riedel-de Haën) we obtained the DQE coefficient by nearly a tenth higher for P 47 under these conditions. The period of three hours was used in order to stabilize the apparatus parameters and perhaps to approximate average time conditions for the applications in SEM.

B.Volbert: In Fig. 5 the P 47 scintillators light output signals are decreasing to nearly zero after approx. 1000 hours of operation. There are commercially available scintillators which do not show this decrease at comparable current densities. Is it possible that the binding material of the preparation causes this strong decrease and is then Fig. 5 really representative for P 47 powder scintillators?
Authors: In SEM conditions such a damage of a scintillator as illustrated in Fig. 5 does not occur. Higher currents than those usually applied in SEM were used to demonstrate a more intensive course of damage. If the current is decreased (curve $J = 3\times10^{-7}$ A cm^{-2}) the radiation damage also decreases. Naturally, the effect of the quantity and quality of the binder influences also the damage. We used the binder of standard concentration of about 0.15 mg cm^{-2} and standard quality (Formvar, PMMA, silicate). With respect to various technological aspects the shape of the curve can be influenced to a certain extent. Nevertheless great differences are not supposed.

It is necessary to note that even the contamination in the applied vacuum (5×10^{-3} Pa) plays a certain role in damage, depending on the current density of the electron beam.

R.B.Bolon: Is the decay time of the exciting pulse sufficiently long so as to require some form of deconvolution in order to determine the decay time of the Ce^{3+} centres? Would you comment on the precautions that you took and any data processing you might suggest?
Authors: The decay time of the measuring system was 5 ns as mentioned in the paper. It has been found by deconvolution of the obtained decay characteristics that the measurement error due to the decay of the measuring system is lower than 2.5 % in the region from 20 ns upwards. It is a quite convenient accuracy for determination of the decay time of all three components, that is also for the decay time of Ce^{3+} centres. Therefore, the plotted curves are not corrected for decay of the excitation pulse and this must be considered only at interpretation in the region $t < 20$ ns.

J.C.H.Spence: For many U.H.V. applications it is important that a detector be bakeable. To what temperature can your detectors be safely heated?
Authors: YAG or YAP scintillators can be heated up to a temperature of approximately 1200 °C without change of their qualities. Using oxygen atmosphere, temperature of up to 1000 °C is recommended. Naturally the light-guide of quartz glass should be used and the scintillator should not be covered with aluminium at higher temperatures.

J.C.H.Spence: Do the authors plan any experiments to measure the spatial resolution of the single crystal screens, using for example, the differentiated edge response method described in your reference 10?
Authors: Spatial resolution is one of the basic parameters of cathodoluminescent screens. We plan to make the measurements (e.g. according to D.G. Taylor, Photo-Electronic Image Devices,

Proceedings of the Third Symposium held at Imperial College, London, p. 395, Academic Press, 1966). But at present we have to solve more important problems in the field of scintillators. We would welcome cooperation with any working place equipped with the devices for measuring spatial resolution. (We would supply a suitable screen.)

J.C.H.Spence: Is it possible to bond the photocathode of a television tube directly onto this material in order to avoid the problems of interfacing your single-crystal screens to fibre-optics?
Authors: If the single-crystal material is to form the input face of a TV tube, there is still one problem to solve. The single-crystal screen should be very thin in order to limit the optical scatter. The mechanical strength of this material, e.g. of 50 µm thickness and 20 mm diameter is insufficient. Even when it is easy to deposit a material of the photocathode on the single-crystal screen, for mechanical reasons it is impossible to replace the input glass face of the TV tube by this material. Fibre-optics as carrying medium is necessary.

N.R.Comins and J.T.Thirlwall: A geometric compromise must be reached in designing a compact yet efficient light-guide for BSE detection by the mode illustrated in Figure 7. Please supply more details of the use of fibre optics for this purpose.
Authors: The BSE detector applying the fibre optics consists of a scintillation plate of square shape, the side wall of which is of 1.7 × 1.5 mm area. A beam of fibres (single fibre diameter of 50 µm) is bonded perpendicularly in the same way as the light-guide in Fig. 7. The fibres are encapsulated in a terminal of rectangular shape. From here the beam passes into a circular profile and is enclosed in a connector on the other end. With respect to the small height of the side wall (1.5 mm) certain losses occur during the light transfer from the scintillator into the light guiding fibres, the total effectiveness of the detector is convenient. This type of detector which has many advantages (multifunction operation) as well as its performance will be described in a later paper.

N.R.Comins and J.T.Thirlwall: The comparison of the performance of a YAG:Ce^{3+} BSE detector with the semiconductor detection system should be made in terms of DQE and noise figure rather than "sensitivity". Do you have any comparative data of this type for the two systems?
Authors: At this time no exact data relating to DQE are available. In the near future we are going to measure the performace of YAG:Ce^{3+} of the BSE detector in terms of DQE. Up to now we have compared the "sensitivity" by which we mean the specimen current required for the obtainment of the same detector output of semiconductor and scintillator types. The result is given in the paper. Apart from that we have observed resolution of the material contrast and time characteristics. Noise properties were observed on the basis of the resulting image quality using the same sample only.

R.B.Bolon: Where can I buy some YAG:Ce^{3+} and YAP:Ce^{3+} for use in my instruments?
Authors: Our single-crystal scintillators based on YAG:Ce^{3+} will be supplied by the PLANO Company (Friedrichsplatz 9, D-3550, Marburg, Germany) and the EBTEC Company (120 Shoemaker Lane, P.O. Box 465 / Agawan, Mass. 01001 U.S.A.).

N.R.Comins and J.T.Thirlwall: What is the commercial cost of a YAG or YAP scintillator in comparison with the P 47 items available?
Authors: As far as we know, the price list is in the stage of preparation. The price of single-crystal scintillators will be certainly higher than that of P 47 scintillators and will differ according to size, shape and kind of application.