Preparation and evaluation of P-47 scintillators for a scanning electron microscope

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Received 8 March 1978, in final form 8 June 1978

Abstract Plastic scintillators commonly in use in the secondary-electron detectors of scanning electron microscopes are known to deteriorate rapidly, particularly under conditions of operation at high electron beam currents. A simple method is described for the production of scintillators from P-47 phosphor powder which is known to have excellent durability. The performance, evaluated in terms of the signal-to-noise ratio, detective quantum efficiency and relative light output, is correlated with the surface coverage of the phosphor layers, and compared with some commercial plastic and powder scintillators.

1 Introduction
The secondary-electron detector used in most scanning electron microscopes is based on the design of Everhart and Thornley (1960), as shown in figure 1. Low-energy secondary electrons emitted from a sample are attracted by a positively biased grid and accelerated toward the front surface of a scintillator held at a positive potential of about 10 kV. The scintillator is attached to a light pipe which transfers the generated photons to a photomultiplier tube (PMT), thus producing an amplified electrical signal. From a study of the signal-to-noise ratio at the PMT output and the detective quantum efficiency and relative light output, it is possible to correlate the performance of the detector with the system being limited by the inherent shot noise in the primary-electron beam plus noise introduced in the secondary-emission process. This behaviour was expected if, on average, each secondary electron produced substantially more than one photoelectron at the photocathode of the PMT.

Much of the subsequent published work has concentrated on increasing the light output from the scintillator by using different preparation methods and materials. To overcome difficulties in machining and polishing the shaped plastic scintillator tips used in the original design, Hatzakis (1970) developed a technique for applying a thin layer of plastic scintillator material, from solution, directly on to the light pipe and obtained enhanced light output. Taylor (1972), using this same type of thin-film scintillator, showed that the light intensity reaching the PMT could be further increased by the use of a quartz light pipe. Secker et al (1973) extended the study to include phosphor powder scintillators (with the powder referred to as 'an oxide of phosphorus'), showing that a further increase in light output was possible. Unfortunately, none of these studies included any signal-to-noise ratio measurements and it is not clear, therefore, that these improvements have in fact improved the detector performance.

Pawley (1974) carried out a systematic study of various scintillator materials measuring the light output (relative to a plastic scintillator), the signal-to-noise ratio and the deterioration of the system in terms of the light output, with exposure to the electron beam. The plastic material was shown to deteriorate very rapidly and Pawley was unable to obtain any signal-to-noise data for this reason. It was concluded that one of the most promising materials was the P-47 type phosphor (yttrium silicate, 1% Ce), as scintillators from this material produced the highest light output, the best signal-to-noise ratio and had a hundredfold increase in lifetime measured against the plastic material (lifetime being defined as the time for the light output to decrease to 50% of its initial value).

No systematic study of the production parameters for phosphor powder scintillators has been presented to date. Pawley (1974) describes, in outline, how to prepare the layers by allowing the powder to settle from a water suspension on to the light pipe or a glass disc. This is then covered with a layer of collodion or Formvar after which aluminium is evaporated on to the unit. Subsequent baking at temperatures of 620 K was suggested for removal of the organic film. The only data on the important parameter of phosphor layer thickness are the recommendations that 'the thinner the layer the better' and 'a good compromise is about 10 crystal layers'. It was reported, in addition, that these devices showed poor mechanical strength especially under conditions of vacuum cycling and this limited their useful life. Secker et al (1973) also reported a similar preparation method for powder scintillators using collodion as a binder, but again no details of phosphor layer thickness were presented.

The present work describes a simple preparation technique for P-47 phosphor powder scintillators. The performance of the scintillators is assessed in terms of the signal-to-noise ratio of the detector output, the detective quantum efficiency and the relative scintillator light output. These measurements are then correlated with the surface coverage of the phosphor layers. The scintillators are also compared in performance with the standard plastic devices of both the disc and thin coated-layer type as well as a commercial powder scintillator.

2 Scintillator preparation
2.1 P-47 phosphor powder scintillators
The scintillators in the present study were produced by a process of settling of the phosphor from suspension under gravity on to thin glass discs for mounting on the end of the...
light pipe, as shown in figure 2. The phosphor powder (Riedel-De Haén Blue FS, No. 54063), which had a particle size distribution as shown in figure 3, was ultrasonically dispersed in a low-concentration solution of gelatin in distilled water. This suspension was applied in the form of a drop, limited by surface tension to cover the whole disc, and this was allowed to dry completely in air. The gelatin acted as a binder for the powder to increase the mechanical strength of the layer. In

Figure 2  Diagram of the P-47 phosphor powder scintillator and light pipe mounting.

Figure 3  P-47 phosphor powder particle size distribution measured using a Coulter counter and shown as a histogram. The distribution is approximately log-normal in form as indicated by the curve.

order to produce devices with varying layer thicknesses and binder contents, the concentrations of the constituents in the suspension were varied, and the results will be presented for the scintillators in terms of the screen density expressed in units of mass per unit area on the disc.

The second production stage is the application of the aluminium coating over the powder layer. This coating has the dual function of providing an acceleration electrode for the secondary electrons and also acting as an optical reflector increasing the proportion of the generated photons which enter the light pipe. Diakides (1973) has shown that the highest light output is obtained from such a device when the aluminium layer is applied by the 'float technique'. Following this approach, the aluminium layer was produced by vacuum evaporation on to a glass slide covered with a pre-evaporated parting layer of 150-200 nm of NaCl, the layer thicknesses being determined by means of a quartz-crystal film thickness monitor. After appropriate circles had been scribed in the coating, the aluminium discs were floated off on to the surface of distilled water and picked up on the phosphor-coated glass discs.

2.2 Other scintillators investigated

In order to evaluate the phosphor powder scintillators, three other scintillator/light pipe combinations were used in this study. These were a standard plastic disc scintillator (JEOL Ltd), a hemispherically tipped light pipe directly coated with phosphor powder and used as supplied (JEOL Ltd), and a quartz light pipe coated with plastic scintillator from a solution of the plastic material in toluene (supplied by M E Taylor Engineering, Maryland, USA).

3 Assessment of the scintillator performance

The instrument used in the present work to evaluate the scintillators was a JEOL JSM U3 scanning electron microscope fitted with an Everhart-Thornley detector having a 19 mm diameter light pipe and operating under normal conditions with a collecting grid voltage of 420 V and an accelerating potential of 10 kV.

The fundamental performance of the SEM is limited by random fluctuations in the number of electrons falling on a picture element of the specimen (Oatley 1972). It has been shown by Rose (1948) that for the average human eye to distinguish an area of brightness \( B \) from an adjacent area of brightness \( B + \Delta B \), the relation

\[
\frac{S}{N} \geq \frac{B}{\Delta B}
\]

applies, where \( S \) is the signal corresponding to the brightness \( B \) and \( N \) the noise signal superimposed thereon. In assessing the performance of the detector system, therefore, it is the signal-to-noise ratio which is the important criterion. As mentioned earlier, much of the published work has concentrated on the optimisation of the scintillator light output, terming this the 'scintillator efficiency'. Provided, however, that the light output per incident electron is sufficient to produce several photoelectrons from the photocathode of the PMT, it is unlikely that increased light output will result in any useful improvement in performance. A better criterion for determining the efficiency of a detector system is the so-called 'detective quantum efficiency' (DQE), which gives a measure, for the detector, of the fraction of incoming secondary electrons actually contributing to the signal and is defined operationally as

\[
\text{DQE} = \frac{(S/N)_\text{real}^2}{(S/N)_\text{ideal}^2}
\]

where 'real' indicates the actual device and 'ideal' indicates an ideal detector (Jones 1959). The DQE has a maximum value of unity. In the present case, the ideal detector would have, at its output, the signal-to-noise ratio of the incident secondary electrons from the sample, which must be evaluated in terms of the shot noise in the incident beam plus the noise introduced in the secondary-emission process. Following Everhart et al (1959), the signal-to-noise ratio for these secondary electrons can be written as

\[
\left( \frac{S}{N} \right)_\text{ideal} = \left( \frac{I_0}{2e\Delta f} \right)^{1/2} \frac{1}{(1 + b)^{1/2}}
\]

where \( b = (\sigma - \bar{\sigma})^2/\bar{\sigma}^2 \), with each primary electron having a probability \( p(\sigma) \) of producing \( \sigma \) secondary electrons, \( I_0 \) is the incident beam current, \( e \) the electronic charge, and \( \Delta f \) the bandwidth of the measuring instrument. The accepted
Preparation and evaluation of P-47 scintillators for an SEM

An estimate of $b$ for incident beam energies of 20 kV is about 4 (Thornton 1968). If, however, $\sigma$ is randomly distributed about $\bar{\sigma}$, then $b$ reduces to $1/\bar{\sigma}$, although Oatley (1972) indicates that this assumption should be treated with some reserve.

In using equation (3), assuming random secondary emission, the value of $\bar{\sigma}$ should represent only secondary electrons emitted by the specimen. In reality there will be other contributions to the signal reaching the detector from secondary electrons produced in the sample chamber by backscattered electrons, as well as from some backscattered electrons themselves (Everhart et al 1959). For the purpose of evaluating the ideal signal-to-noise ratio, and thus the detector $DQE$, a measured value of the secondary-electron current $I_{se}$ reaching the detector is used for $\bar{\sigma}$.

The signal and noise values for the detector fitted with the various scintillators were measured experimentally at the PMT output by means of a DC voltmeter and a true RMS voltmeter respectively, the latter having a noise-equivalent bandwidth $\Delta f$ of 27 kHz.

4 Results

4.1 Effect of production parameters on scintillator performance

The gelatin binder content of the phosphor layer primarily determines the mechanical strength of the powder scintillators, but in excess can degrade the performance. Scintillators having no binder could be produced, but were very fragile, with the aluminium coating showing no adhesion. Figure 4 shows the effect of the binder content on the light output of a series of scintillators. There is little influence on output for a binder content up to about 2 g m$^{-2}$, but thereafter a rapid decrease occurs. Based on this information, all the scintillators to be discussed later were produced with 1.6 g m$^{-2}$ gelatin content corresponding to the use of a gelatin concentration of 0.05 wt % in aqueous solution. With this preparation, the adhesion of the aluminium layer was excellent and it seems likely that the re-immersion procedure used to float on the layer results in a softening of the binder and subsequent bonding of the aluminium to the phosphor layer.

Aluminium layers covering a range of thicknesses (50–200 nm) were made, but because of damage resulting during flotation, continuous films on the scintillators of less than about 70 nm could not be consistently produced. The light output for scintillators with 200 nm aluminium films was found to be about 25% less than for corresponding scintillators made with a 100 nm film, consistent with expected energy losses and backscattering in the thicker film. For these reasons aluminium films in the range 90–100 nm were used in preparing the scintillators to be described later. It is estimated that for these films 5% of the signal electrons are backscattered (Reimer and Krefting 1976), and that the transmitted electrons suffer a 5% average energy loss, based on the Thomson–Whiddington Law (Oatley 1972).

A series of scintillators of various screen densities was produced by adjusting the phosphor concentration in the suspension. Figure 5 shows the output signal-to-noise ratio of the Everhart-Thornley detector fitted with the different scintillators, all measured under identical instrumental operating conditions. The performance of the detector improves up to a phosphor screen density of about 12 g m$^{-2}$ after which the signal-to-noise ratio remains effectively constant. The detector output signal, for constant instrumental conditions, gives a relative measure of the light output from the scintillator and this is shown in figure 6 for all the scintillators. There is little influence on output for a binder content up to about 2 g m$^{-2}$, but thereafter a rapid decrease occurs. Based on this information, all the scintillators to be discussed later were produced with 1.6 g m$^{-2}$ gelatin content corresponding to the use of a gelatin concentration of 0.05 wt % in aqueous solution. With this preparation, the adhesion of the aluminium layer was excellent and it seems likely that the re-immersion procedure used to float on the layer results in a softening of the binder and subsequent bonding of the aluminium to the phosphor layer.

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scintillators studied. For the P-47 scintillators there is a broad maximum in light output signal centred on a screen density of about 14 g m\(^{-2}\) with decreased output for the thicker phosphor layers. As found by Pawley (1974) the light output from the P-47 scintillators was about 2-3 times that produced by the plastic material.

To determine the DQE values of the detector fitted with different scintillators, the signal-to-noise measurements were carried out using a 25 kV primary-electron beam and a beam current, measured in a Faraday cage, of 1.0 × 10\(^{-10}\) A, normally incident on a polished copper specimen. In addition, data were required for \(\delta_{\text{eff}}\), the mean effective ratio of the number of secondary electrons to the number of primary electrons, for use in equation (3), and these were obtained in two ways. Firstly, the current collected by an electrode fitted inside and connected to the grid of the detector was measured directly. The second method involved a technique similar to that of Colby \textit{et al} (1967) where the specimen current was measured initially with the specimen held at \(-60\) V, and the detector grid at \(420\) V to attract all the emitted secondaries, and then with the detector grid grounded and the sample set at a potential high enough to re-collect the secondary emission (120–150 V in the present system). The difference in these values of specimen current, assuming that the backscattered electron emission remains unaffected, represents the maximum electron current which could reach the detector. The value obtained for \(\delta_{\text{eff}}\) from the direct measurement was 0.27±0.01, and from the second method 0.28±0.01. The mean value was then used in equation (3) for \(b\) (= 1/5) to evaluate the ideal signal-to-noise ratio with the assumption that the secondary-emission process is random.

Table 1 gives the experimental signal-to-noise values for the detector with the various scintillators studied, as well as the DQE values. Pawley (1974) measured the signal-to-noise ratio of an Everhart-Thornley-type detector fitted with a range of different scintillator materials by allowing the primary beam to strike the scintillator directly. DQE values have been calculated from the data and are included for completeness. No direct correlation between the signal-to-noise ratio values of the two sets of data should be made as they were obtained under different operating conditions and using a different noise bandwidth. The DQE values, on the other hand, agree well for the P-47 scintillators.

<table>
<thead>
<tr>
<th>Scintillator material</th>
<th>Type</th>
<th>((S/N)_{\text{measured}})</th>
<th>((S/N)_{\text{ideal}})</th>
<th>DQE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Present work</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Plastic</td>
<td>Disc</td>
<td>30.2±0.5</td>
<td>49.8</td>
<td>0.37</td>
</tr>
<tr>
<td>Plastic</td>
<td>Thin-film</td>
<td>33.5±0.5</td>
<td>49.8</td>
<td>0.45</td>
</tr>
<tr>
<td>P-47 powder</td>
<td>4 g m(^{-2})</td>
<td>25.0±0.5</td>
<td>49.8</td>
<td>0.25</td>
</tr>
<tr>
<td>P-47 powder</td>
<td>8 g m(^{-2})</td>
<td>29.2±0.5</td>
<td>49.8</td>
<td>0.34</td>
</tr>
<tr>
<td>P-47 powder</td>
<td>12 g m(^{-2})</td>
<td>30.0±0.5</td>
<td>49.8</td>
<td>0.36</td>
</tr>
<tr>
<td>P-47 powder</td>
<td>16 g m(^{-2})</td>
<td>30.2±0.5</td>
<td>49.8</td>
<td>0.37</td>
</tr>
<tr>
<td>P-47 powder</td>
<td>24 g m(^{-2})</td>
<td>31.0±0.5</td>
<td>49.8</td>
<td>0.39</td>
</tr>
<tr>
<td>Phosphor powder</td>
<td>JEOL</td>
<td>25.0±0.5</td>
<td>49.8</td>
<td>0.25</td>
</tr>
<tr>
<td>Pawley (1974)</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>P-47 powder</td>
<td>Not stated</td>
<td>6.4</td>
<td>10.2</td>
<td>0.39</td>
</tr>
<tr>
<td>P-46</td>
<td>Single crystal</td>
<td>5.4</td>
<td>10.2</td>
<td>0.28</td>
</tr>
<tr>
<td>CaF(_2)(Eu)</td>
<td>Single crystal</td>
<td>4.74</td>
<td>10.2</td>
<td>0.22</td>
</tr>
<tr>
<td>Glass (NE 901 or GS 1)</td>
<td></td>
<td>3.3</td>
<td>10.2</td>
<td>0.10</td>
</tr>
</tbody>
</table>

4.2 Effect of operating conditions on scintillator performance

Following Everhart and Thornley (1960) and Pawley (1974), the variation of the signal-to-noise ratio as a function of the accelerating voltage applied to the aluminium layer on the scintillators is shown in figure 7(a). The signal-to-noise ratio reaches a constant value above an applied potential of about 5 kV illustrating that the 10 kV potential normally applied is well within the plateau region of the curve. Figure 7(b) shows...
the corresponding dependence of the output signal plotted as a function of the mean energy $E_m$ of the electrons transmitted through the aluminium film, calculated using a law of the Thomson–Whiddington type (Oatley 1972), and given by

$$E_m = \left( E_a^2 - A x \right)^{1/2}$$

where $E_a$ is the acceleration potential, $A = 1.1 \times 10^{11} \text{ (eV)}^2 \text{ cm}^{-1}$ (for aluminium) and $x$ (cm) is the aluminium layer thickness. It was found for a range of P-47 scintillators that the output signal varied as $E_m^{1.6 \pm 0.2}$, consistent with Pawley's results (1974).

For completeness, the detector output signal $S$ and the signal-to-noise ratio were measured as functions of the electron beam current $I_b$ incident on a polished copper sample. Figure 8(a) shows that $S$ is proportional to the beam current in the range $10^{-11} - 10^{-11} \text{ A}$ for both the P-47 and plastic scintillators. This is in contrast with the results of Secker et al (1973) which indicated differing behaviour for these two types of scintillator. The signal-to-noise ratio of the system, as plotted in figure 8(b), showed a linear dependence on $I_b^{1/2}$ in agreement with equation (3).

![Figure 8](image-url)

Figure 8 (a) The dependence of the detector output signal fitted with a P-47 scintillator (■) screen density $16 \text{ g m}^{-2}$ and a thin-film plastic scintillator (●) on the primary-beam current $I_b$ incident normally on a polished copper sample. (b) The signal-to-noise ratio plotted as a function of $I_b^{1/2}$.

5 Discussion

In producing scintillators two aspects are important for optimum performance. Firstly, the active phosphor material must cover the whole surface of the scintillator so that as high a proportion as possible of the incident electrons contribute to the signal since losses of this kind directly reduce the DQE of the detector. Secondly, as stated previously, the light output per electron detected must be high enough to ensure that no significant statistical noise is introduced in the subsequent conversion at the photocathode of the PMT.

Unlike plastic scintillators where the whole surface is usually active, the phosphor powder scintillators, produced by the settling process, suffer intrinsically from incomplete coverage particularly at low screen densities. This can be illustrated by imaging the surface of a phosphor layer in the

![Figure 9](image-url)

Figure 9 Cathodoluminescence images of P-47 scintillators without aluminium covering. (a) Screen density $8 \text{ g m}^{-2}$, (b) screen density $16 \text{ g m}^{-2}$.
cathodoluminescence mode in the SEM where the uncovered regions will show as dark patches, as can be seen in figures 9(a) and (b). Bodó and Hangos (1953) and Weiszburg and Hangos (1959) have derived an expression to calculate the coverage of a settled screen for a given particle size distribution, particle density and screen density, and figure 10 shows the theoretical curve for the present phosphor. This calculation indicates that 99% coverage should be achieved at a screen density of 14 g m\(^{-2}\). With lower screen densities, more electrons will be ineffective because they will strike uncovered areas, and thus a reduction in the signal-to-noise ratio will occur. The signal-to-noise ratio measurements given in figure 5 can thus be explained in terms of the phosphor surface coverage in the P-47 scintillators.

The light output, although increasing with surface coverage at low screen densities, begins to decrease again for screen densities greater than about 15 g m\(^{-2}\), and this can be attributed to light absorption and scattering losses in the thicker layers. It is significant that once complete coverage is attained, there is no decrease in the signal-to-noise ratio corresponding to that in the light output, indicating that subsequent conversion processes are not limiting the performance. The optimised scintillators for the phosphor particle size distribution used in the present work will have a screen density in the range 14-25 g m\(^{-2}\), where the signal-to-noise ratio has reached the plateau region. Where light pipe losses are more significant, it may be preferable to use screen densities nearer to 15 g m\(^{-2}\) where the light output is at a maximum. It seems likely that the expression of Bodó and Hangos can be used to predict the optimum coverage parameters for any given size distribution of phosphor powder.

From the DQE values in table 1, it is clear that the best performance was obtained with the detector fitted with a new thin-film plastic scintillator, with slightly inferior performance being obtained for the new disc-type plastic scintillator and P-47 powder devices (screen density greater than 15 g m\(^{-2}\)). Pawley's data indicate that both CaF\(_2\)(Eu) and glass (NE 901 or GS 1) scintillators have DQE values considerably lower than the other types and thus do not appear to be suitable substitutes. The values obtained for the plastic scintillators are contrary to the conclusion by Pawley (1975) in that the signal-to-noise ratio for the detector fitted with these devices is not in fact lower than with the P-47 scintillator despite its lower luminescence efficiency. The present result indicates that no significant deterioration in performance can be associated with the conversion at the photocathode of the PMT with the lower light output of the new plastic scintillator.

The measured DQE value for the detector fitted with any of the scintillators does not approach the theoretical value of unity. Electron losses at the collection grid and scintillator contact ring together with backscattering from the aluminium layer may account for some of this deviation from ideal performance. A further reason could be that only the DC PMT output current and its fluctuations were measured, whereas the output is in fact made up of individual pulses. If these pulses have a broad amplitude distribution, then the measured output could be dominated by the fraction of electrons giving rise to higher-amplitude output pulses, and this would lead to a low measured DQE.

Pawley (1974) has clearly illustrated the rapid deterioration of the light output from the plastic materials especially under high-beam-current conditions, and thus it is expected that the signal-to-noise ratio will deteriorate when the photocathode conversion begins to influence the noise seriously. The P-47 phosphor, on the other hand, shows a hundred-fold improvement in lifetime relative to the plastic and thus, provided that mechanical stability of these powder scintillators is good, it can be expected to be more suitable for instruments operated under wide-ranging beam current conditions. The present P-47 scintillators have been used extensively in the JEOL JSM U3 microscope, and no mechanical deterioration, in particular damage to the aluminium layer, has been observed even after six months of use. This instrument does, however, have a specimen airlock and thus vacuum cycling is minimal compared with the system described by Pawley.

6 Conclusions
It has been shown that the performance of the P-47 phosphor powder scintillators can be correlated with the surface coverage of the powder and that under optimised conditions the performance is only slightly inferior to a new thin-film plastic scintillator. The present method of preparation produces devices with the good mechanical properties necessary for use over an extended period.

Acknowledgments
The authors are indebted to Dr J Langmore of the MRC Laboratory of Molecular Biology, Cambridge for his critical refereeing of the manuscript and his constructive comments, in particular, regarding the evaluation of the detector in terms of the DQE concept, and the suggested mechanism for the low measured DQE values in terms of the pulse amplitude distribution in the output. Thanks also go to Mr C F van Huyssteen for his assistance in determining the phosphor particle size distribution and Dr B P J van Oorschot for useful discussions on the noise measurements.

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