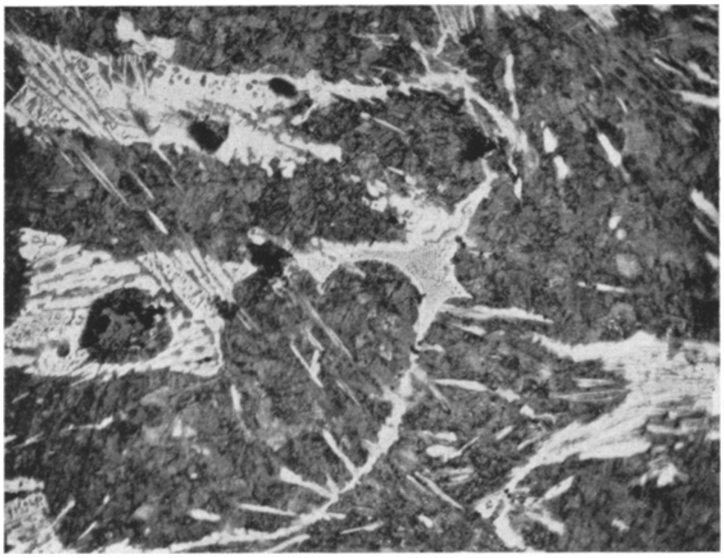

Figure 1 (b) Microstructure of an alloy of Fe-3% C-2% Si-0.03% S. Nital etch (× 200).

tures are close to those expected for the alloys used. Thus, the problems of the introduction of elements into liquid iron baths are overcome.

It is well established that graphite morphology is affected by the presence of impurity elements, and at present the technique is being used to study the effect of impurity elements on the graphite morphology of hypo-eutectic cast irons.

Figs. 1a and 1b show the results of some preliminary experiments on the effect of sulphur on graphite morphology. Fig. 1a shows the structure obtained on melting a sample prepared from very pure iron, 3% C and 2% Si. The graphite is here present as nodules. The addition of 0.03% sulphur leads to a tendency to form the Fe-Fe_3 C eutectic and changes the morphology of any graphite which may be present as seen in fig. 1b.

In summary, there are several advantages of this powder technique over more conventional methods of melting on a laboratory scale.

(i) versatility – any system whose components can be prepared as powders can be studied, e.g. Fe-C-Si, Al-Si, Cu-Zn.
(ii) high purity materials may be used.
(iii) speed – the time required for the Fe-C-Si samples from powder to solidified specimen is only 15 min.
(iv) stricter control of composition.

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Evidence for AC and DC Electroluminescence of CdS: Cu Powder Phosphors

Electroluminescent (EL) CdS powder phosphors have not been produced before [1], though chemically similar ZnS powder has been a well-known electroluminophor for more than thirty years. In fact, as has been shown [2], even (Zn, Cd)S powder, produced by the usual methods for preparing EL ZnS powders, ceases to exhibit EL when containing more than 50 g mol % of CdS. On the other hand CdS in crystal form shows EL under dc excitation [3, 4].

We have developed a method for the preparation of CdS:Cu powder phosphors which exhibit visible ac and dc EL in electric fields of about 10^5 V/cm, at room temperature. Results presented in this note also suggest a relationship between EL brightness and variations of non-stoichiometry of CdS:Cu powder phosphors.

The CdS was activated exclusively with copper in order to ensure the simplicity of the system. We added (1 to 6) × 10^{-3} g atoms of copper per mole CdS, which is more than the solubility of copper in CdS at room temperature (about 10^{-5} g atoms of copper per mole CdS [5]). This is the usual practice because the precipitation of copper on defects in powder microcrystals after firing is assumed to be essential for the EL exciting mechanism.

The preparation process begins with the pre-treatment of pure CdS powder which, in our case, was a 5N grade "Koch Light Laboratories" CdS powder. This powder is placed in a semi-sealed quartz tube, evacuated up to 10^{-3} torr, and fired for 0 to 20 min at 750°C. The vacuum pump remains in operation throughout the firing process. After firing, the tube is cooled in air. We found that from the beginning of the firing process evaporation of cadmium takes place from CdS. Cadmium, which condenses on the cold region of the quartz tube, is removed from the tube after several different periods of evaporation, and weighed on a microbalance. The identity of evaporates was verified by Debye-Scherrer X-ray analysis. After some 3 min of firing, when the amount of evaporated cadmium reached ~ (3 to 4) × 10^{-3} g atoms per mole CdS, evaporation of CdS itself also begins. After about 20 min of firing the amount of evaporated cadmium is totalling ~ 9 × 10^{-3} g atoms per mole CdS, and the evaporation rate of cadmium is greatly reduced, while CdS continues evaporating.

The CdS powder samples, that had sustained various periods of evaporation, are mixed with the respective amount of copper, and then hammer-milled for ~ 10 min to ensure homogeneity. The powders are placed in quartz tubes which are then evacuated up to 10^{-3} torr and sealed, fired for 1 h at 950°C and cooled in cold water. Firing in argon atmosphere is found to give the same effect as firing in vacuum.

The small-grained fractions of the resulting CdS:Cu powders are then extracted. The mean grain diameter is ~6 to 8 μm, as measured under a metallurgical microscope. These fractions are mixed with Araldite, an organic dielectric, in a weight ratio of about one part of CdS to two parts of Araldite. The mixtures are placed between two equal metal electrodes with an interspace of 0.2 mm.

The measurements of integrated EL brightness (B) dependence upon mean voltage (V) of this CdS:Cu powder phosphors show [6] that B/V relation follows the well-known [7, 8] EL relations, $B \propto \exp(-c/\sqrt{V})$ under ac excitation, and $B \propto V^a$ under dc excitation over three to four orders of brightness magnitude. The agreement with this basic EL relations confirms that the reported effect on CdS:Cu powders is really EL. The coefficients, $c \approx 200 - 220\ V^{1/2}$, and $a \approx 3.5$, are not significantly different for different pre-treatment of samples. The EL spectra [6], which are very similar for all CdS:Cu samples, show maxima in the green, orange and deep red regions.

Our measurements also provide evidence that the integrated brightness intensity of the samples depends upon the amount of evaporated cadmium during the pre-treatment operation (fig. 1). The brightness intensity measurements were carried out with the EMI 9558 QB photomultiplier having S-20 spectral response. The EL CdS:Cu cells operated under 1200 V of ac 600 Hz voltage supply, at room temperature. The cell geometry, grain density, mean grain diameter and copper concentration of different samples were standardized as far as possible.

It is necessary to see whether the fig. 1 dependence remains unchanged for any exciting voltage and frequency and at any temperature, or whether it is correct only under the conditions indicated above. The dependence of brightness intensity upon ac and dc voltage and ac frequency is shown to be very similar for the CdS:Cu samples with different pre-treatment, i.e. the fig. 1 dependence would remain unchanged for measurements at any given voltage and frequency. The ratio of dc to ac EL brightness intensity is also shown not to vary much with different CdS:Cu samples, so that for dc EL fig. 1 behaviour remains correct. Besides, EL brightness is known not to change much with changing temperature [9]. Therefore, we believe that the integrated brightness intensity versus evaporated cadmium amount dependence, shown in fig. 1, is generally correct for the ac and dc EL of CdS:Cu powder phosphors.

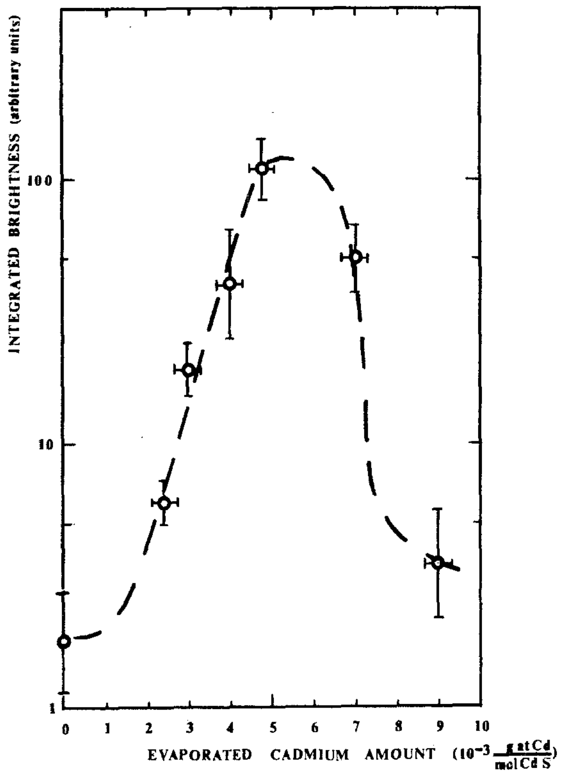

Figure 1 Integrated brightness of CdS:Cu EL powders against evaporated cadmium amount during CdS pre-treatment. Measurements carried out at room temperature, under 1200 V and ac 600 Hz voltage supply for EL cells.

It seems that the only factor causing such variation of EL brightness, is the non-stoichiometric change of CdS powders resulting from the pre-treatment operation. Since analyses of CdS stoichiometry are not yet fully completed nor are EL processes known in sufficient detail, we are not in a position to offer an interpretation of the obtained result. However, as regards this result we would like to point out that according to the preliminary experiments of Vecht *et al* [10], the variation of non-stoichiometry of ZnS powders appears to have a great effect on EL processes.

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Field-Ion Microscopy of Aluminium and Plasma-Anodised Alumina Films

Field-ion microscopy has been used to study a wide range of materials [1-4], but neither aluminium nor alumina has been amenable to the technique. Aluminium presents difficulties because of its low evaporation field (Brandon [5] quotes a calculated value of 1.8 V/Å) and its ease of plastic deformation; however, low-field imaging and field-etching techniques offer some hope of success. Alumina has an excessively high resistivity, and cannot therefore be imaged as a bulk specimen; however, a thin anodised film might be suitable, as has been shown by Southworth [4] for wet-anodised iridium. Recent work by the present author [6] and by Boyes *et al* [7] has shown that despite the experimental problems, both aluminium and alumina can be studied by field-ion microscopy.

A specimen of 99.99% pure aluminium wire was electro-polished into the required needle