PHYSICS

Use of an Electron Beam for detecting Superconducting Domains of Lead in its Intermediate State

SUPERCONDUCTING domains have been detected in lead by means of an electron beam technique previously used to detect magnetic domains¹. A spectroscopically pure cylinder of lead, 1.1 cm long and 0.28 cm in diameter, was mounted with its axis vertical in a modified horizontal electron diffraction camera. A 20-kV electron beam was focused about 15 cm from the cylinder and was deflected at 50 c/s in a vertical direction by means of suitable coils, so as to produce an electron shadow of the lead cylinder. Fig. 1c shows a photograph of a shadow taken with the lead in its normal state. In this case the smooth edge is merely a geometrical projection of part of the curved surface of the cylinder. The cylinder, which could be heated electrically by a non-inductively wound heater, was attached to the outside of a metal vessel containing liquid helium. A uniform magnetic field could be applied to the lead by means of a pair of small Helmholtz coils. These were mounted symmetrically on either side of the lead with their common axis horizontal and perpendicular to the mean direction of the undeflected electron beam.

Fig. 1. Electron shadows of the surface of a lead cylinder, showing lead in the following states: (a) superconducting, (b) intermediate, (c) normal, (d) intermediate

The electron shadow shown in Fig. 1a was taken after the applied field had been increased from zero to 37 oersteds. When the field was further increased to 40 oersteds the shadow edge ceased to be smooth (Fig. 1b). It became smooth again for fields in excess of about 50 oersteds (Fig. 1c).

These observations are consistent with the following explanation. At and below 37 oersteds the lead was superconducting. At 40 oersteds the lead transformed to the intermediate state² in which alternate lamellae of superconducting and normal lead were perpendicular to the axis of, and approximately periodically distributed along, the cylinder. Since the applied field was expelled from the superconducting parts, a concentration of magnetic flux occurred in the normal regions. The resulting periodic distortion of the previously uniform field led to the 'corrugated' electron shadow shown in Fig. 1b. Above about 50 oersteds the intermediate state transformed to the normal state. Taking 50 oersteds as the critical field this would indicate that the results so far described were obtained with the lead at about 6.95° K (ref. 2).

Fig. 1d was obtained by cooling the lead in a field of 37 oersteds from a temperature at which the cylinder was in its normal state to 6.8° K. The irregularities in the electron shadow may again be explained by assuming that the applied field was distorted because the lead was in its intermediate state. The distance between adjacent minima

on the shadow in Fig. 1d corresponds to distances varying from 0.08 to 0.29 mm at the specimen. It follows that these distances give the order of the periodicity of the superconducting domains in lead under the conditions of the experiment. The variation of this periodicity as a function of applied field and temperature has not yet been studied in detail. A full account of the work and details of the apparatus will be published in due course.

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¹ Blackman, M., Haigh, G., and Lisgarten, N. D., *Nature*, **179**, 1288 (1957).
² Shoenberg, D., *Superconductivity* (Cambridge Univ. Press, 1952).

Gamma-ray Spectrometric Determination of Cesium-137 in Sea-water by using Ammonium Molybdophosphate as Scavenger

A NUMBER of methods have been proposed for determining very low concentrations of caesium-137 in sea-water by γ -ray spectrometry. The sodium potassium cobaltinitrite method^{1,2} and the nickel ferrocyanide method³ were proposed by several workers for identifying traces of caesium-137. Special considerations should be made on the purity and the size of the caesium sample to be counted and the simplicity of the procedure to be suited for shipboard operations.

The ammonium molybdophosphate method was used by Yamagata and Yamagata⁴ originally for the radiochemical determination of caesium-137 in biological materials, the precipitate of ammonium molybdophosphate being made *in situ* in a sample solution. Meanwhile, the work of Smit⁵ revealed the ion-exchange properties of ammonium molybdophosphate, and the use of solid ammonium molybdophosphate by adding to sample solution as a specific scavenger was made by Yamagata *et al.*⁶ in the neutron activation analysis of common caesium and by Miyake *et al.* in the determination of caesium-137 in sea-water⁷.

The best conditions for scavenging concerning temperature, acidity, the quantity of carrier caesium and the quantity of ammonium molybdophosphate were checked by Yamagata *et al.*⁶. The experiments on a small scale showed about 0.1 per cent quantity of the ammonium molybdophosphate (wt./vol.) was enough to recover more than 95 per cent of caesium labelled with caesium-137 in sea-water in acid conditions. However, the large-scale operations on 100–200 l. of sea-water by this method resulted in only 70–80 per cent of recoveries with the addition of 5 mg quantity of carrier caesium per litre⁸. The reduction in recoveries might have been caused by losses in the large-scale operations, and the addition of carrier caesium as well because of the limited ion-exchange capacity of the ammonium molybdophosphate.

To overcome the difficulties involved in the addition of appreciable amount of carrier caesium for checking the recovery in full-scale operation, the radioactive caesium-132 was examined for its use as a spike. This radioisotope was produced by a (γ, n) reaction on the purified caesium chloride using a linear accelerator at the Japan Atomic Energy Research Institute. It had specific activities of about 1 mc./g caesium when used as a spike and several hundred μ mc. were applied to 100 litres of sea-water sample. This means the addition of only less than 1 μ g of the stable caesium to 50 μ g of naturally occurring caesium in the sea-