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Observations on whiskers and skeletal crystals in PbSe layers by scanning electron microscopy

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Whiskers and skeletal crystals of PbSe are obtained on substrates of (1 0 0) germanium. The production conditions and peculiarities of their structures are discussed. SEM observations show that the whiskers are prism-shaped possessing rectangular or square cross-sections with lengths up to 190 μm and widths of the surrounding walls up to 26 μm. Hollow pyramidal crystals with their apexes connected to the layer surface by a whisker are described, as well as hollow crystals with an open or a closed surrounding surface. Certain aspects of the growth mechanism of skeletal crystals and whiskers are discussed.

1. Introduction

Certain photoelectric properties of the compounds of the group of lead chalcogenides and PbSe in particular (e.g. photosensitivity in the infra-red) and the possibilities of using them in semiconductor devices are the reasons for their intensive study and an ever-growing interest in them.

In contrast to the numerous studies on the semiconductor properties of layers of these compounds, the structure and particularly the surface morphology of the layers are discussed in a very limited number of scientific communications. The detailed investigations of the initial growth stage carried out by Yagi *et al.* [1], Palatnik *et al.* [2–5] and Kossevich *et al.* [6] are, to a certain extent, an exception to this. Semiletov [7] only notes the existence of long whiskers in epitaxial PbSe layers.

Some results of our studies on the morphology of the surface of PbSe layers containing whiskers and skeletal crystals are given in this paper. We are not aware of any published data concerning observations of similar PbSe layers.

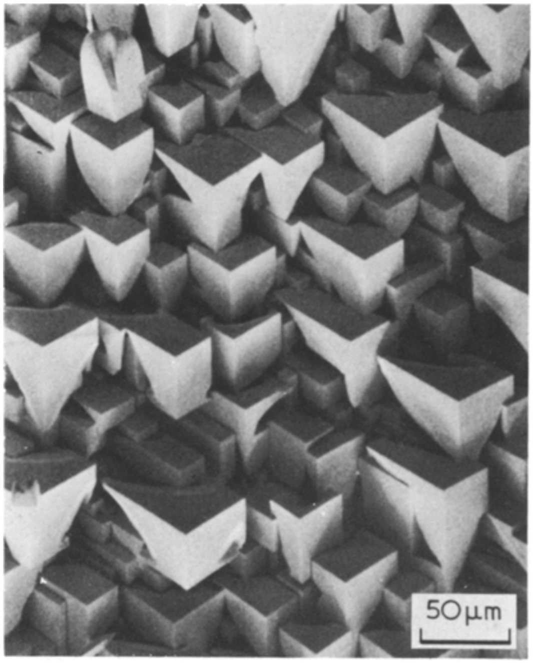


Figure 1 PbSe layer surface.

2. Experimental details

PbSe layers, were obtained from a vapour phase in an open system in the presence of 99.999% pure argon, using apparatus similar to that described by Igarashi [8]. Tablets of polycrystal PbSe, 99.999%

pure, 16 mm diameter and 3 mm thick, were used as the source material. The layers were deposited on germanium substrates with (1 0 0) orientation, the distance between the source and substrate varying between 5 and 10 mm. The source temperature was maintained in the range of 700 to 740° C and that of the substrate between 480 and 510° C; the flow of argon was 0.5 litre h^{-1}.

The topography of the layers and the morphology of the whiskers and skeletal crystals obtained on the surface of the layers were studied by a JSM-S1 scanning electron microscope using the secondary electron mode and an accelerating voltage of 10 kV. The magnifications used were between 300 and 2000.

3. Results and discussion

As a rule the complete surface of the PbSe layers was covered with whiskers and skeletal crystals. In some cases where the distance between the source and the substrate was less than 5 mm, layers with a characteristic surface were obtained, as shown in Fig. 1.

The whiskers grown on the surface of PbSe layers are prism-shaped with a rectangular or square cross-section with the dimensions 190 μm long and surrounding walls 26 μm wide (Fig. 2). The whiskers usually lie parallel to each other (Fig. 2a) but this is not observed at some points on the layer surface (Fig. 2b). In the cases where it was possible to examine the base of the whiskers it was established that near the layer surface the whiskers become smaller, tending towards a smaller prism or a cylindrical shape (Fig. 3).

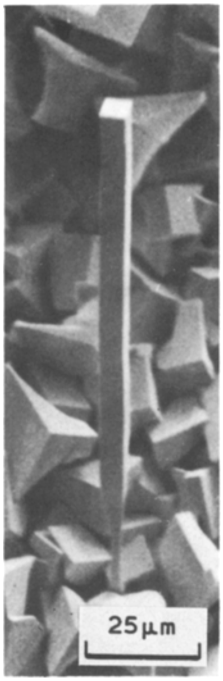


Figure 3 Connection of the whiskers with the layer.

Skeletal structures were also observed on the surface of PbSe layers. These structures vary greatly in shape, therefore we shall discuss only a few of the more typical ones: (a) the skeletal

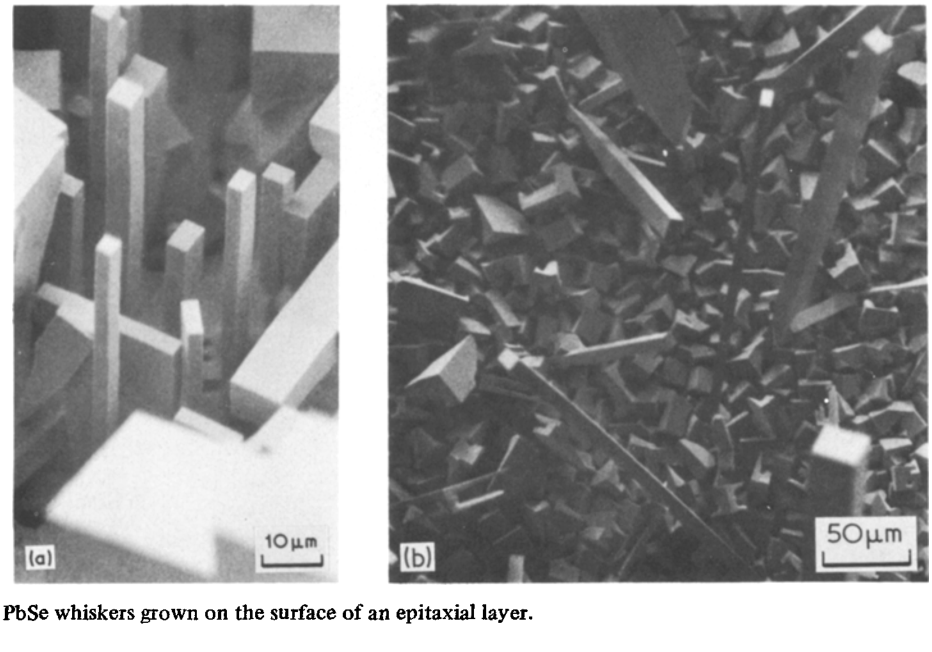


Figure 2 PbSe whiskers grown on the surface of an epitaxial layer.

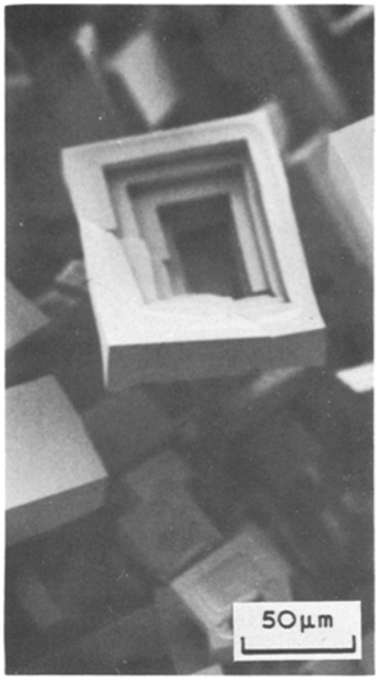


Figure 4 Skeletal crystal consisting of a whisker and a hollow four-walled pyramid.

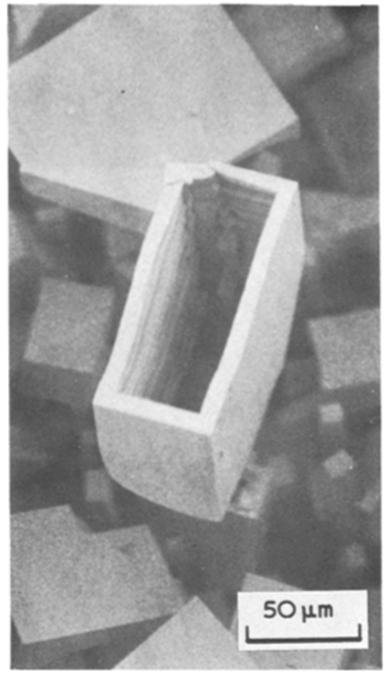


Figure 5 Hollow crystal with closed surrounding surface.

crystals shown in Fig. 4 are shaped like a hollow pyramid turned with its apex towards the layer surface and connected to it by a whisker; (b) hollow crystals with a closed or an open surrounding surface (Figs. 5 and 6 respectively). A characteristic feature of the skeletal crystals is their inner surface which consists of steps of approximately 2 μm (Fig. 7).

The prismatic forms have rectangular cross-section, in which the PbSe whiskers (a habit typical of cubic crystals) are obviously determined by the basic structural modification in which PbSe crystallizes, namely a lattice structure of the type of sodium chloride.

The skeletal crystals shown in Fig. 6 are similar to those described by Fujisaki *et al.* [9] in CdS. The rest of the structures have many features in common with the hollow crystals described in [10].

Certain peculiarities of the growth mechanism of the skeletal crystals shown in Figs. 4 to 6 have also been discussed for similar CdTe hollow crystals [10]. The detailed study of the initial growth phase of lead chalcogenides carried out by Palatnik and co-workers [2–5] and Kossevich *et al.* [6] shows that even at that early stage conditions favourable to the growth of the structures we have examined are created. Suitable places for

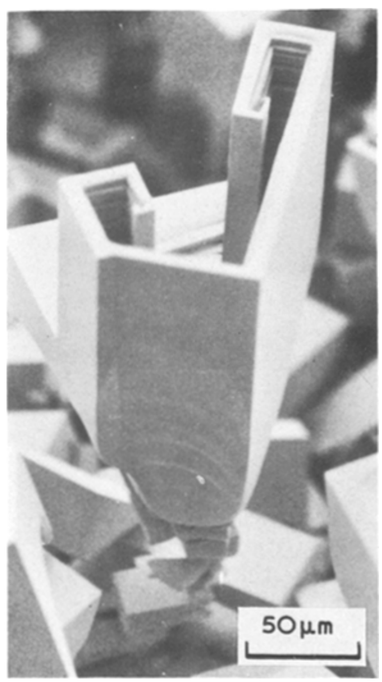
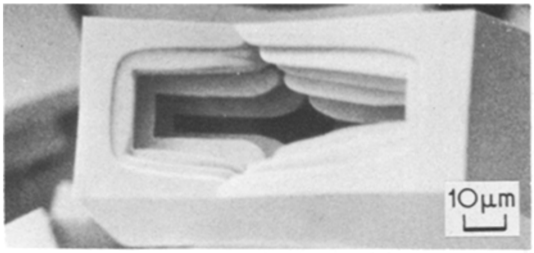
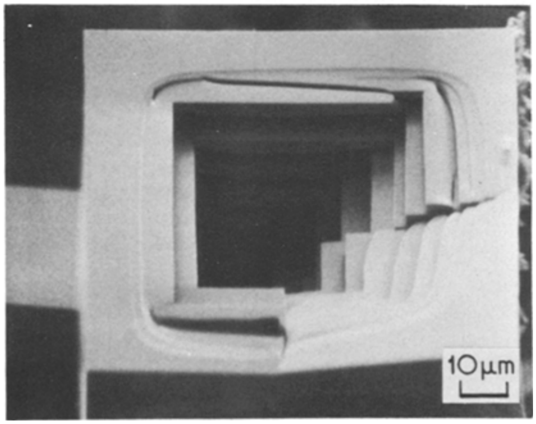


Figure 6 Hollow crystal with open surrounding surface.

the growth of whiskers are the microcrystallites (which exhibit a marked tendency to grow rapidly in height) which are described by these authors. Another condition favouring the growth of PbSe whiskers is the growth of microcrystallites without coalescence observed by Palatnik and co-workers





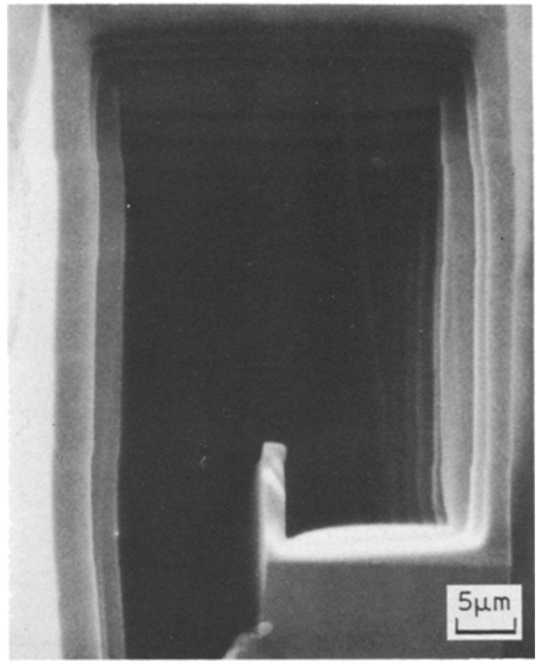


Figure 7 Inner surface of hollow PbSe crystals.

[2–5] and Kossevich *et al.* [6]. Under definite conditions this may favour a more rapid growth in one direction only. The formation of micropores in the layers may initiate the growth of hollow crystals [4, 10].

The step-like inner surface of the hollow crystals is typical of skeletal structures and the mechanism of their formation is examined by Chernov and Boudourov [11].

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