their hexagonal axes parallel to one of the four body diagonals of a cube indicates that they were all formed from one larger single crystal of the body-centered cubic β phase by a transformation of 'oriented' character, as was observed already by Weerts (1932). Each hexagonal crystal is formed from the β-phase crystal by the displacement of the atoms mainly in the direction of one of the cube diagonals of the cubic structure. There are eight different sets of such displacements, two for each cube diagonal. They correspond to the transformation to hexagonal crystals having identical structures but different orientation in space. The transformation evidently takes place with equal utilization of the four different possibilities of spatial orientation (four body diagonals) with the result that one single crystal of the β phase yields a multitude of hexagonal crystals with an apparent cubic symmetry.

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Formation and Identification of Thallium Single Crystals

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A crucible is described in which a metal can be cooled from the melt while subjected to a strong temperature gradient. By this method single crystals of thallium were obtained when the gradient exceeded 20° C. cm.$^{-1}$. A simple X-ray method is described for determining whether the sample is a single crystal and for finding the hexagonal axis. In some samples a body-centered cubic phase was observed with a lattice constant 3% smaller than the value for the high-temperature phase.

Introduction

Certain nuclear experiments which have been on the way in this institution require single crystals of thallium in the shape of spheres approximately 6 mm. in diameter. A knowledge of the direction of the hexagonal axis is also necessary. Because of the short lifetime of the radioactivity induced in the material, it is essential that the single crystal be grown and its crystal orientation be determined in a matter of a few hours.

Upon cooling, thallium undergoes a martensitic transformation from b.c.c. to h.c.p. (Lipson & Stokes, 1941). In other similar cases it is generally accepted that, even if the high-temperature phase is in the form of a single crystal, polycrystalline material will result upon cooling. However, in the case of thallium it has been found (Dehlinger, 1932; Rao & Subramaniam, 1936) that a single crystal is obtained if the cooling is sufficiently slow. This method could not be used in the present case, because of the length of time involved in the treatment. The present paper reports on single crystals obtained from a melt cooled at a relatively large rate in a strong temperature gradient.

Method and results

A cylindrical carbon crucible was made of two halves lightly pressed together (Fig. 1). The thallium was filled into a cavity *b* in one of the pieces. A 2 mm. duct directed the melt into a spherical hollow *h*. The outer diameter of the crucible was considerably constricted in this region. The lower base of the crucible was seated on a metal disk *A*, which rested on a hot plate. A similar metal disk *D* rested upon the upper surface of the crucible. This disk was hard-soldered to a copper tube *E*, which could be heated with a heating coil. A brass tube *F* carrying a cooling coil was hard-soldered to the hot copper tube. The cooling coil could be soft-soldered to the brass tube at different distances from the joint so that the heat

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carried away in the water could be widely varied. By adjusting the voltages of the heaters, the temperatures of disks A and D could be independently regulated. The temperatures were measured with thermocouples

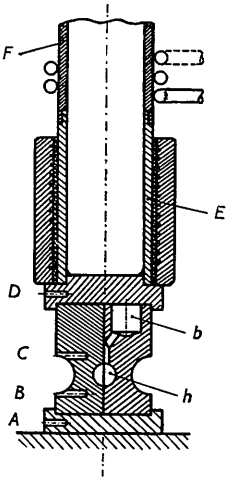

Fig. 1.

inserted in holes at points A, B, and D. In addition, the temperature difference between points B and C could be measured with two thermocouples in a differential circuit. These two junctions were electrically insulated from the crucible by painting the wires with Sauereisen cement.

The preparation of the sample was begun by rapidly heating both plates to about 100° C. above the melting point of the thallium. Then the heating of disk D was interrupted so that a temperature difference between B and C was rapidly established. The highest temperature gradient obtained was 120° C. cm.$^{-1}$. By reducing the heat input at A, the temperature of the specimen could be lowered at any desired rate, while maintaining the temperature gradient fairly constant. After the metal cooled below the transformation point, the crucible was taken out and put on a thick metal plate to cool quickly. It was found that the two halves of the crucible could be separated and the spherical pellet extracted without difficulty. If the surface was scratched during this operation, the sphere was etched in hot nitric acid.

By experimenting on a number of samples, it was found that when the cooling occurred at a rate of 2° C. min.$^{-1}$ single crystals were obtained if the temperature gradient was larger than 20° C. cm.$^{-1}$. No particular correlation between the hexagonal axis and the temperature gradient was noticed. In cases where the temperature gradient was lower than 12° C. cm.$^{-1}$ single crystals were also obtained but in addition a quantity of microcrystalline material could be detected.

In order to ascertain if the pellet is a single crystal an unusual X-ray method was devised because of the strong X-ray absorption in the sample. The method consisted in mounting the pellet on a goniometer head and taking a rotating crystal pattern with Co K radiation (40 kV., 10 mA., 10 min.). At this wavelength the (006) reflection spots appear at a scattering angle of about 154°, i.e. in a back-reflecting direction. If the entire sphere is a single crystal, then only four spots are generated, provided that the hexagonal axis made an angle greater than 77·5° with the rotation axis. By taking oscillation patterns with gradually smaller amplitudes, the position of the pellet which gives one of the four (006) reflections can be found. A simple calculation gives the necessary adjustment of the goniometer head in order to orientate the hexagonal axis in any desired direction. The intensity of a (006) reflection is sufficient for the spot to be observed on a fluoroscopic screen. Probably with a wide pencil of X-rays (for example 2 mm.) the photographic method could be completely dispensed with, in which case the whole operation of finding the orientation could be reduced to a matter of a few minutes.

Certain of the pellets showed a curious behavior under etching. It was noticed that part of their surface layer reacted with nitric acid much more slowly than the remainder and this led to the formation of a protruding shiny surface. X-ray diffraction revealed that it consisted of a polycrystalline b.c.c. phase. On the other hand, filings taken from the shiny surface gave the normal hexagonal pattern. This showed that the shiny material is a quenched high-temperature phase which is unstable, and spontaneously transforms while undergoing the filing process. The lattice constant for the cubic material was found to be 3·77 Å, i.e. about 3% smaller than the value reported for the high-temperature phase. This difference in the lattice constant is far too large to be attributed to thermal expansion. It might be accepted as an indication that the high-temperature phase can exist at normal temperatures only when the thallium has formed an alloy with some other element (possibly carbon) resulting in a smaller lattice constant.

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