

## SOME OBSERVATIONS ON THE SOLIDIFICATION OF HELIUM

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Visual observations of the solidification of helium have been made. The difficulty of obtaining large blocks, even under conditions when the crystallization process can be controlled, is noted.

IN all experiments on the properties of solid helium, it has usually been obtained under conditions where the apparatus itself (the pressure chamber) is connected to the external apparatus that produces the gas pressure by means of a narrow capillary. This method came under criticism in Cwilong's work<sup>[1]</sup> on the determination of the solidification point of helium in a glass piezometer, the pressure in which was produced by a change in volume of a metal bellows connected to it. The melting curves obtained by Cwilong below 1.8° K are considerably different from the curves obtained by Keesom and Keesom<sup>[2]</sup> and by Swenson<sup>[3]</sup> by the blocked capillary method. Unfortunately, Cwilong did not carry out observations on the characteristics of the solidified helium and confined himself only to the determination of the melting point, which was registered by the cessation of motion of magnetic needles placed inside the piezometer.

Keesom,<sup>[4]</sup> who first used a glass piezometer, could not for some reason observe the boundaries between the liquid and solid helium.

Since we wished to investigate the motion of electric charges in solid helium,<sup>[5]</sup> we naturally had to be sure of the quality of the crystals studied. For observation of the solidification process we initially used a glass test tube (Fig. 1a) made of No. 29 glass of diameter  $\frac{7}{9}$  mm, the upper part of which was sealed to an iron-chromium stopper (N47KhB). This seal (in contrast with a 3S5 seal to kovar) stood up to any number of coolings and heatings without cracking. A 0.5 mm diameter platinum wire was sealed into the bottom of the tube.\* The tube was connected through a 0.9/1.6 mm diameter capillary, shielded by a vacuum jacket, to a helium gasifier† by means of which a

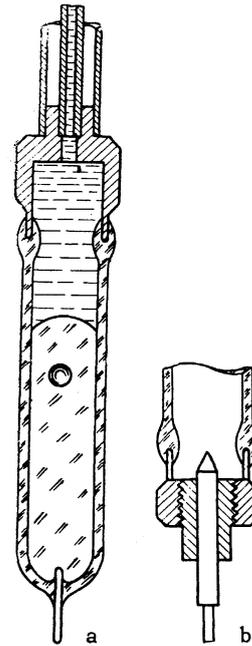


FIG. 1

relative pressure up to 100 atm could be produced and maintained.

Since the thermal conductivity of iron-chromium is very low, the test tube in apparatus already used in studying the motion of charges was provided with an iron-chromium stopper, through which a 2 mm diameter copper conductor of cold was passed (Fig. 1b).

With a favorable choice of illumination, the boundaries between the solid and liquid helium are very well visible, so that the solidification process could be recorded on motion picture film (Fig. 2).

The means by which the liquid solidifies depends on many factors, difficult to control because the heat generated on solidification must be led away through the two-phase system produced, the overall thermal conductivity of which varies within wide limits.

\*The platinum wire (Fig. 1a) had no noticeable effect on the duration of the crystallization process.

†I would like to thank A. I. Filimonov and V. K. Tkachenko for the possibility of using the gasifier set up by them.

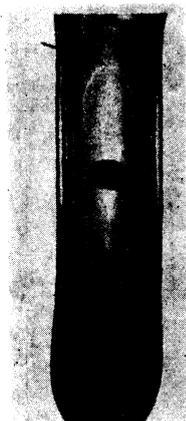


FIG. 2

It is difficult to obtain macroscopic blocks of solid helium even when the process is carried out very slowly, for it is very difficult in practice to achieve conditions under which the heat removal is through the lowest part of the apparatus. Even under conditions when the capillary transmitting the pressure is insulated from the ambient by vacuum shielding, it is not always possible to prevent the formation of centers in it.

In order to avoid this we even had to resort to heating the part of the capillary inside the vacuum shield (40 mm in length) which was in the immediate neighborhood of the tube. This procedure, however, produces at the same time a temperature gradient in the tube itself.

The following types of crystallization are generally observed.

1. If the pressure is increased relatively rapidly ( $\sim 0.1$  atm/sec) blocking of the capillary is observed and then an instantaneous solidification of the whole volume of the tube, which in this case appears to be filled with a mass reminiscent of "wet" snow. This mass only becomes transparent gradually when the pressure is increased considerably.

2. Sometimes on reaching the solidification point a "hail" of solid spherulites starts to precipitate in the liquid, and these gradually fill the whole volume of the tube.

3. For a sufficiently slow increase of the pressure ( $\sim 0.01 - 0.05$  atm/min) the formation of an ideally transparent crystal starts at the surface of the copper rod let into the tube, and the layer of solid helium formed duplicates the contours of the cold conductor, as though marking out a surface of constant temperature. As the crystal grows its boundary deforms and the phase boundary, which crosses the tube by its continuous upward movement, becomes uneven. Formation of a crystal at the surface of the cold conductor is eased if a temperature gradient is produced in

the liquid filling the tube, by heating the capillary or, correspondingly, by lowering the level of the liquid helium in the external bath. However, even when these measures are taken, the growth of several regular spherulites starting from the side walls of the tube is observed, which in the end usually leads to the blocking off of some volume of the liquid, which because of the small compressibility of solid helium cannot be transformed into the solid state, even upon considerable increase in pressure.

4. Apparently the purity of the helium also affects the crystallization process. Generally, in the first experiments with newly assembled apparatus, blocking of the capillary takes place more easily and also the formation of centers of crystallization, leading to the falling out of "hail."

We carried out some experiments with  $\text{He}^3$  along with the observations of the crystallization of  $\text{He}^4$ .

We did not observe any special features in the solidification of  $\text{He}^3$ , but we have the impression that the formation of large blocks in  $\text{He}^3$  is appreciably easier and that the whole solidification process proceeds more regularly. We used apparatus with electrodes<sup>[5]</sup> in the experiments with  $\text{He}^3$ . After the formation of a crystal at the cold conductor and the subsequent gradual filling of the apparatus with "ice," the appearance of a transparent solid phase in the interelectrode region was invariably observed. We intend to study the solidification of  $\text{He}^3$  in more detail later.

A report has recently appeared<sup>[6]</sup> on the observation of a new modification of solid  $\text{He}^4$ . There was no evidence of this new modification in our observations on the crystallization process. This can be explained, however, by the very limited region of existence of the new crystalline modification and the low accuracy in the measurement of pressure in our experiments.

In conclusion I would like to thank B. N. Esel'son for discussion and D. I. Vasil'ev for help in the experiments.

<sup>1</sup>B. M. Cwilong, *Phys. Rev.* **88**, 135 (1952).

<sup>2</sup>W. H. Keesom and Miss A. P. Keesom, *Leiden Comm.* **20**, 224d, 224e (1933).

<sup>3</sup>C. A. Swenson, *Phys. Rev.* **79**, 626 (1950).

<sup>4</sup>W. H. Keesom, *Leiden Comm.* **17**, 184b (1926).

<sup>5</sup>A. I. Shal'nikov, *JETP* **41**, 1059 (1961), this issue, p. 755.

<sup>6</sup>J. H. Vignos and H. A. Fairbank, *Phys. Rev. Lett.* **6**, 265 (1961).