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Solidification of helium, at room temperature under high pressure (*)

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Résumé. — La cristallisation de l'hélium 4He a été observée à 297 K et 115 ± 2 kilobars. Cette valeur est proche
de celle donnée par l'équation de Simon, extrapolée de mesures à 5 kilobars. La cellule diamant utilisée est d'une
conception nouvelle qui permet l'étude de tous les gaz inertes entre 4 et 500 K jusqu'à 1 mégabar.

Abstract. — Melting of 4He has been observed at 297 K and 115 ± 2 kilobars. This value is close to that derived
from Simon's equation, extrapolated from 5 kilobar data. The diamond cell we used is a new design and allows
inert gases to be studied between 4 and 500 K up to 1 megabar.

1. Introduction. — Melting curves at high pressures and temperatures, and, in general thermodynamic
parameters of matter at high densities are of interest
in numerous fields of research, especially as diamond
anvil techniques now reach into the megabar range
(100 GPa) or more.

Helium is of particular interest for the study of
melting parameters since its critical point is the
lowest of all elements. Its melting curve has only been
measured up to 14 kilobars at liquid N₂ temperature,
and, in this range, is quite well represented by Simon's
equation. Over 40 years ago, L. D. Landau showed
the impossibility of a terminal critical point on a
melting curve, but switching of the transition from
first to second order [2] should not be completely
ruled out. There is also a possibility of a temperature
maximum [3] on the melting curve which would
indicate the approach of a triple point and of a solid-
solid transition [4], although it has been proved that
both of these conjectures do not hold for elements
in the argon and sodium classes [5].

Outside the field of fundamental thermodynamics,
to be a convenient pressure transmitting medium:
there is evident need for better knowledge of the
behaviour of gases (fluids) at very high densities,
particularly hydrogen (deuterium, tritium), helium
and their mixtures. In the field of very high pressure
research in the diamond cell, helium has been thought

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in the bomb and on the piston are then gradually decreased, preserving the same differential pressure, until the former is zero. The cell is then extracted from the bomb and contains a significant mass of helium (~ 10^{-7} grams). The internal pressure can be varied by varying the low pressure backing the piston, which remains attached, by a capillary, to the low-pressure generator through the whole process. Details of this setup as well as experimental procedures will be fully described in a later paper [10].

In our experiments, we used U-grade helium with impurity content below 20 ppm. Crystallization comes about rapidly upon compression and the value of pressure is not well defined. The equilibrium point was therefore determined at the coexistence of the solid and liquid phases, during slow decompression. Direct microscopic observation showed the melting of large (50 × 30 µ) crystals during 15 min. Those conditions can be considered as isothermal in view of the thickness (~ 20 µ) of the cell, and thermal conductivity of the anvils.

The equilibrium pressure is 115 ± 2 kilobars at 297 ± 0.4 K.

The refractive index difference between the two phases is quite weak which is not unexpected for helium. Crystals were observable only under oblique illumination. No anisotropy is apparent under crossed polarizers.

3. Conclusions. — The index difference of the two phases which renders the crystals visible clearly points to a difference in density between the solid and the fluid : the transition is thus of the first order up to 115 kilobars.

Room temperature (297 K), is over 50 times the critical temperature for $^4$He. Comparison with Simon’s equation with the parameters quoted in ref. [12] gives a predicted value of 115 kilobars, which fits well with our value of $115 \pm 2$ kilobars. The figure shows a comparison of this equation with existing data. Thus, the extrapolation of this type of melting curve can be safely used above 100 kilobars for helium and, a fortiori, for other elements verifying Simon’s equation whose critical temperature is higher.

There is no evidence for a solid-solid phase transition in helium up to 120 kilobars : the melting curve shows no inflexion point that could be linked to proximity of a triple point. No index anisotropy in the solid could be observed, which points to a situation where helium has retained its face-centred cubic low-pressure structure.

No broadening of the ruby luminescence has been observed, in the solid phase. This confirms the expected plasticity of solid helium at high pressures. Moreover, the refractive index being low, grain boundaries are weak and transparency of the medium is good : solid helium seems to be a convenient pressure-transmitting medium for optical measurements above 100 kilobars, especially in view of its chemical inertness in comparison with classical ethanol-methanol mixtures [9].

Finally, concerning high pressure technology, our setup is applicable to the study of all fluids which are chemically compatible with the cell materials, in the megabar range and between 4 and 500 K.

References