Crystal Growth of Solid $^3$He

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Crystal growth of $^3$He in restricted and bulk geometries is described. A striking feature is the growth of single crystals inside a metallic power of 0.3 μm mean grain size. Comparison of the intensity of the $(1, 1, 0)$ and $(2, 0, 0)$ Bragg reflections gives a direct estimate of the zero-point motion. No temperature dependence of the Debye-Waller factor is observed.

1. INTRODUCTION

The aim of this paper is to describe the crystal growth of solid bcc $^3$He realized as part of work intended to perform neutron experiments down to the millikelvin range. The search for $^3$He single crystals is made by the observation of $(1, 1, 0)$ Bragg reflections. In order to increase the thermalization of solid $^3$He with the cooling agent (a copper nuclear demagnetization stage), the choice was made to dissolve the $^3$He atoms inside a sponge of fine metallic powders (copper or silver). Results obtained with these “restricted” geometries are compared with experiments performed on a bulk geometry. Finally, the observation of the $(2, 0, 0)$ reflection gives an estimate of the mean displacement of the $^3$He atoms due to the strong zero-point motion of this quantum solid.

2. EXPERIMENTAL CONDITIONS

Experiments were performed using a rapidly cooled dilution refrigerator where the holder sample can reach 70 mK in less than 3 h from room temperature. The thermometry utilized carbon resistors (Allen-Bradley 100 Ω for $T > 1$ K and Speer 100 Ω for $T < 1$ K) and a germanium bolometer. Slow linear temperature sweeps with a cooling velocity $v = \Delta T/\Delta t$ were monitored by a microprocessor using the output of an ac resistor bridge. The incoming $^3$He is injected into the experimental chamber.
by a fine capillary tube (2 m length, 0.1 mm diameter), which is thermalized at different cooling points.

For the restricted geometry, the $^3$He target was a boatlike copper container filled with copper or silver sintered powder of respective nominal mean grain sizes 1 $\mu$m and 700 Å. The thickness of the boat was 0.5 mm; a thin Cu foil (0.05 mm) and filling capillaries were soldered with tin onto the top of the container (Fig. 1a). The copper powder was sintered at 550°C during 3 h under a hydrogen atmosphere; since this temperature leads to some annealing of the "bulk" copper plate and foil, a good mechanical stiffness was simultaneously realized with the copper support. The silver powder was sintered at 200°C during 30 min. Electron microscope studies show that after this procedure the grain distribution is rather homogeneous, with a mean size of 3000 Å. In contrast to the copper powder case, auxiliary mechanical supports of Zycral were necessary to maintain the pressure (Fig. 1b).

For the bulk geometry, an indium-sealed target was designed as shown in Fig. 1c. The correct $^3$He thickness (0.1 mm) was achieved by polishing the upper Zycral part of the cell.

The minimum on the melting curve at $P \approx 29$ atm and $T \approx 0.32$ K rules out the performance of experiments at different molar volumes by changing the pressure at room temperature. Along the path of the incoming capillary of $^3$He, there will be a blocking as the temperature decreases. The solidification of the fluid is then performed at constant volume along a course shown in Fig. 2. The liquid reaches the melting curve at A (temperature $T_A$) and the capillary is plugged. From A to B (temperature $T_B$), the experiment is still performed on the melting curve, but the temperature decrease of 400 mK corresponds to a pressure drop of 10 bar. Finally, the pressure in the cell becomes quasiconstant, since the thermal expansion vanishes.

The molar volume of $^3$He in the target was determined by measuring the transmission $N$ of $N_0$ incoming neutrons. Accurate measurements can be achieved due to the huge neutron absorption cross section $\sigma_{abs}$ of $^3$He (see Ref. 4):

$$\sigma_{abs}/\lambda = 2962 \text{ barn/Å}$$

$N$ is related to $N_0$ by the absorption coefficient:

$$N = N_0 e^{-\alpha}, \quad \alpha = A\sigma_{abs}x/V$$

where $A$, $V$ and $x$ are, respectively, Avogadro's number, the molar volume, and the effective thickness of the target. In order to determine $V$, $\alpha(V)$ was calibrated using the pressure–molar volume relation\textsuperscript{5,6} of liquid $^3$He, where the pressure $P$ can be measured at room temperature. For the chosen wavelength ($\lambda \approx 1$ Å), a typical thickness is 0.1 mm.