OBTAINING TEMPERATURES OF \(14 \div 6^{\circ}\) K IN SOLID HYDROGEN BY PUMPING VAPOR ABOVE ITS SURFACE\

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Abstract

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OBTAINING TEMPERATURES OF 14 ÷ 6° K IN SOLID HYDROGEN BY PUMPING VAPOR ABOVE ITS SURFACE*

A widely used and convenient method for obtaining temperatures below the boiling temperatures of nitrogen, hydrogen, and other low-boiling substances by pumping vapor above a liquid is limited from below, for most of these substances, by the crystallization temperature (the vapor pressure at the triple point is rather high—tens and even hundreds of millimeters of mercury). Further lowering of the pressure for the cooling of a solid, in order to cover temperature intervals in which there is no liquid, has not become widespread.

We were interested in the possibility of obtaining temperatures in the interval bounded by the critical temperature of helium (5.2° K) and the melting temperature of hydrogen (14° K). The present communication gives results on obtaining temperatures down to 6° K in the bulk of solid hydrogen by pumping off its saturated vapors with an adsorption pump cooled by liquid hydrogen, in an apparatus analogous to those used for obtaining a temperature of 0.7° K ($p \cong 2.3 \cdot 10^{-3}$ torr; heat influx $Q \sim 3 \cdot 10^4$ erg/sec; pumping speed $w \sim 300$ l/sec) in liquid He^4, and a temperature of 0.34° K ($p = 5.4 \cdot 10^{-3}$ torr; $Q \sim 600$ erg/sec; $W \sim 75$ l/sec) in liquid He^3 (^{1,2}). Reducing the heat conduction to values $\sim 10^2$ erg/sec and realizing pumping speeds of diffusion and adsorption pumps up to 300 l/sec made it possible to lower the pressure above He^3 to $2 \cdot 10^{-5}$ torr ($T = 0.21°$ K) (^{3,4}).

The possibility of cooling solid hydrogen by pumping vapor above it is determined by: a) the saturated-vapor pressure, which one must have the means to maintain; b) the heat of sublimation, since the cooling is produced by sublimation of the substance, and by the heat capacity; c) the thermal conductivity, which determines the rate of temperature transfer from the surface into the volume and the magnitude of the gradients along the solid column. The vapor pressure above solid hydrogen in the interval $5 \div 14°$ K lies within $2 \cdot 10^{-5} \div 54$ torr; it is easy to calculate that, to maintain a pressure of $2 \cdot 10^{-5}$ torr above solid hydrogen with a heat influx of 10^3 erg/sec, a pumping speed of 100 l/sec is necessary, i.e., the range of pressures and pumping speeds required to cover the interval $14 \div 5^\circ$ by pumping hydrogen was attainable with adsorption and diffusion pumping of helium.

A comparison of thermophysical properties shows that hydrogen is a more effective refrigerant than helium. The heat of sublimation of hydrogen (~ 250 cal/mole at the triple point) exceeds the heat of evaporation of He^4 by more than an order of magnitude; therefore it is easier for the system to cope with heat influxes. The relation between the heat of evaporation and sublimation and the heat capacity of H_2 in the interval $20.4 \div 5^\circ$ K is such that $\sim 25\%$ of the substance is expended on "self-cooling," while $\sim 1.5\%$ is accounted for by the solid (when the temperature of He^4 is lowered from 4.2 to 0.9° K, $\sim 40\%$ of the substance evaporates) (5,6).

The study of adsorption isotherms of hydrogen by charcoal at hydrogen temperatures and the experiments carried out show that, as in the case of He, the use of an adsorption pump makes it possible to provide the necessary parameters in a simple compact device, since at pressures of $10^{-2} \div 10^{-5}$ torr the adsorbent capacity is $2 \cdot 10^2 \div 10^2$ cm^3/g (7).

A negative property of normal hydrogen is the ortho-para conversion, with a heat of occurrence of 254 cal/mole. The conversion rate in solid hydrogen is estimated as 1% per hour at high concentrations of orthohydrogen, which leads to heat evolution of $\sim 4 \cdot 10^4$ erg/sec $\cdot$ mole. When parahydrogen is used, this additional heat evolution is absent.

The use of parahydrogen is useful for another reason as well: in the range $5 \div 11^\circ$K in parahydrogen, the establishment of thermal equilibrium over the volume occurs considerably faster than in normal hydrogen. An estimate by the formula $x^2 = vt$ ($x = 1$ cm; $[v] = \text{cm}^2/\text{sec}$; $[t] = \text{sec}$) gives, at $5 \div 11^\circ$K, propagation times of the temperature wave of $\sim 10 \div 20$ sec in n-H_2 and $10^{-2} \div 1$ sec in n-H_2 (9).

Figure 1 shows a diagram of the apparatus for carrying out the measurements described. In Dewar vessel 1 with liquid hydrogen at temperatures $20.4 \div 14^\circ$K there were a carbon adsorption pump 2 (with 50 g of activated birch charcoal) and a small glass Dewar 3, into which hydrogen ($10 \div 15$ cm^3) was poured from an external Dewar by means of valve 4 or was condensed through the pressure-measurement system. The pumping speed of the pump and, consequently, the temperature obtained were varied by a fine-control valve 5, whose seat diameter was 20 mm. This device provided pumping speeds up to 32 l/sec. The temperature was measured simultaneously at the surface and in the depth of the solid hydrogen by carbon resistance thermometers*, calibrated at hydrogen and helium temperatures and by the transition temperature to the superconducting state of lead indicators ($T_k = 7.22^\circ$K). The distance between

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Fig. 1

Figure 1: Fig. 1

the thermometer-lead-indicator pairs was 40 mm. The leads were constantan ($d = 0.08,\ l \sim 200$ mm).

Usually the hydrogen was brought into the solid state by pumping the vapor from the small Dewar 3 with a fore-vacuum pump and, depending on the solidification rate and other conditions, was a matte or semitransparent fairly homogeneous mass.

Fig. 1

The pressure-measurement system consisted of a mercury differential manometer and McLeod gauges. The elasticity of the saturated vapor over H_2 for the interval $5 \div 10$°K was calculated from the formula proposed by Brickwedde and Scott for temperatures $10 \div 14$°K (5).

The following results were obtained: at an external-bath temperature of 20.4°K, pumping of normal hydrogen achieved a temperature of 6.36 ± 0.05°K ($p = 2.5 \cdot 10^{-3}$ torr).

From the amount of substance evaporated during 10 hours of operation of the apparatus, the heat influx was estimated and found to be $4.4 \cdot 10^{4}$ erg/sec (mainly the heat of conversion and heat conduction through the glass).

[*] The carbon thermometers were kindly provided by N. N. Mikhailov (Institute of Physical Problems, Academy of Sciences of the USSR).

In a column of solid n-H_2 there existed a small temperature gradient. This gradient at a temperature of 7.22°K was determined from the temperature difference at the surface of the solid at which the upper and lower lead temperature indicators passed into the superconducting state, and amounted to $0.1 \div 0.02$°K; at the minimum temperature it was equal to 0.2°K. In a number of experiments, in order to reduce gradients over the volume, corrugated copper foil 0.3 mm thick with a pitch of 3 mm was used (area of contact with the hydrogen ~ 50 cm^2). In this case the temperature drop between the upper and lower thermometers was less than 0.02°K. For the same purpose, and also to eliminate heat evolution due to ortho–para conversion, parahydrogen was used.

The release of 5 mW of power (total heat input ~ 10 mW) by a heater wound on the cold finger led to an increase in temperature by ~ 0.5°K.

At an external-Dewar temperature of 14°K, a temperature of 6.17 ± 0.05°K was obtained in solid n-H_2 ($p = 1.44 \cdot 10^{-3}$ torr); when n-H_2 was used, a temperature of $6^\circ \pm 0.05$°K was reached ($p \cong 10^{-3}$ torr).

This temperature is not limiting, since it was obtained with a comparatively large heat leak and with a pump of medium pumping speed.

Thus, by a new method the temperature interval bounded by the critical temperature of He^4 and the triple point of H_2 has been spanned: temperatures of $14 \div 6$°K were obtained in the volume of solid hydrogen by pumping saturated vapors above its surface.

To provide the necessary pumping rates, a carbon adsorption pump cooled by liquid hydrogen was used.

It has been shown that the temperature in the volume of solid H_2 differs only slightly from the temperature of the surface from which the vapors are pumped; under certain conditions (use of parahydrogen, etc.) the temperature gradients in the solid are so small that the possibility is opened of measuring the temperature of objects placed in hydrogen from the vapor pressure above the refrigerant.

Application of this method makes it possible to extend the temperature range in which other substances may be used. In particular, in one of the measurements a pressure of 10^{-2} torr was obtained above the surface of solid nitrogen when the adsorption pump was cooled with liquid nitrogen.

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