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Abstract

Based on the simultaneous measurement of length and transit time of a specimen under pressure, we have developed a new way of determining absolute high pressure. The mercury freezing point at 0°C has been determined with this new method at 7571.2±1.6 bars. Accurate high pressure calibration of secondary pressure gauge such as manganin gauge is possible with this method. The maximum deviation from linearity of the present gauge studied is 11.6±.1 bars between atmospheric pressure and the mercury freezing point at 0°C.

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If the linear compressibility of a material is isotropic, it is easy to show that the pressure can be expressed in the form

$$P = P_1 - 3\rho_1 \ell_1{}^3 \int_{\ell_1}^{\ell} \left(\frac{1}{\tau_\ell{}^2} - \frac{4}{3\tau_s{}^2}\right)\left(\frac{1}{1+\Delta(P)}\right)\frac{d\ell}{\ell^2} \qquad (1)$$

where

$$\Delta(P) = \frac{\beta^2 B^s T}{\rho C_p}\,. \qquad (2)$$

Here ρ_1 and ℓ_1 are the density and length at atmospheric pressure P_1, τ_ℓ and τ_s are transit times for [111] longitudinal wave and [100] shear wave respectively, β is the volume thermal expansion coefficient, B^s is the adiabatic bulk modulus, T is the absolute temperature and C_p is the specific heat of the material. It is obvious then from equation (1), the pressure can be determined if length ℓ and transit times τ_ℓ and τ_s are measured at the same temperature and pressure.

We choose silicon as our specimen for the following two reasons: (1) because $\Delta(P)$ is quite small ($\Delta(P) \leq 0.0011$ for $0 \leq P \leq P_{Hg}$, P_{Hg} is the freezing pressure of mercury at 0°C) and moreover can be accurately evaluated, (2) because excellent single crystals of silicon can be grown and hence the material can be expected to behave reversibly.

The fractional length change measurement is accurate to 4×10^{-8} and utilizes a meter long specimen; it is described elsewhere.(1,2) Briefly, end positions of specimen are defined by special H-shaped soft magnetic cores which are located with two linear variable differential transformers (LVDT) external to the pressure vessel by a null technique. The length change is measured by a Fabry Perot interferometer with laser beams traveling in a vacuum bellows, between mirrors aligned with centers of LVDT on carriages. The entire system along with ultrasonic pressure vessel are immersed in a constant temperature both controlled to 2×10^{-3}°C. The initial length ℓ_1 at atmospheric pressure is measured interferometrically against a standard length. The fractional error of the original length of the specimen ℓ_1 is 1×10^{-5}.

The silicon specimens used for ultrasonic measurements were made from single crystals of the same purity as that used in the length measurement. The transit times were measured with pulse superposition technique. The reproducibility of transit time measurement was within 2×10^{-5}.

After data for length measurement and ultrasonic transit time were collected, analysis was done by nonlinear least square analysis for fitting Murnaghan equation and Simpson's rule was utilized for integration to obtain P vs ℓ data. Meanwhile, we obtained simultaneously the calibrations for manganin gauge.

The mercury freezing point at 0°C, P_{Hg}, was then determined using a pressure arrest method (zero rate change of pressure versus time at transition point). It was determined to be 7571.2± 1.6 bars.

The quadratic dependence of pressure on the ratio of change of manganin resistance and initial resistance $(\frac{\Delta R}{R_1})$ was fitted. The maximum deviation of the gauge from linearity about midway between P_1 and P_{Hg} was determined to be $\Delta P_M = 11.6\pm.1$ bars.

Table 1 tabulates the value of ΔP_M observed by different workers. Direct comparison of ΔP_M may not be meaningful since it is conceivable that ΔP_M varies with the manganin sample. It is, however, also conceivable that ΔP_M is not very strongly dependent on the sample (assuming its purity and fabrication are nearly the same) as Bridgman proposed but rather dependent on the inaccurate measurement of pressure. At this point we do not believe the answer is known. To compare the relative reliability of the non-linearity or ΔP_M measured by our pressure gauge and that measured by the free piston gauge, let us assume a random error of 2 bars at each pressure for the free piston gauge. Thus the value of ΔP_M would be known to at best ±1 bar. The random error of an individual reading in the present system is 0.04 bars or less. Consequently, ΔP_M can easily be measured to 0.05 bars. It is important to know ΔP_M accurately in many applications. For example, if the pressure derivative of the bulk modulus of iron is measured by length change, an error of 2 bars in ΔP_M would give an error of nearly 10% in B_o'.[1]

Values of the 0°C mercury freezing pressure are given in Table 2. It should be noted that the Committee on Fixed Points Near Room Temperature at the Symposium on Accurate Characterization of the High - Pressure Environment held at the National Bureau of Standards, Gaithersburg, Md. in 1968 designated 7569±2 bars as the accepted value for the 0°C mercury freezing pressure.(3,4)

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Table 1. Values of ΔP_M

ΔP_M (bars)	Source
-10	Harwood[5]
-11	NBS[6]
-21	Zeto and Vanfleet[7]
-21	Boren, Babb and Scott[8]
-11.6±.1	Present Work

Table 2. The 0°C Mercury Freezing Pressure

Pressure (bars)	Source
7565.4 ± 3.7	Newhall, Abbot and Dunn[9]
7569.2 ± 1.2	Dadson and Greig[10]
7571.0 ± 1.2	Yasunami[11]
7571.2 ± 1.6	Present

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