The molecular drag gauge as a calibration standard

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The National Institute of Standards and Technology has used molecular drag gauges for six years as transfer standards in the high-vacuum range, 1×10^{-4} to 1×10^{-1} Pa. We report on the experience gained with these gauges and, in particular, on their long- and short-term calibration stability, on factors affecting accuracy, on the predictability of the effective accommodation coefficient, and on factors affecting the stability of the offset correction.

I. INTRODUCTION

The use of the spinning rotor or molecular drag gauge (MDG) has grown rapidly since its introduction as a commercial instrument in 1982. The range of the MDG conveniently overlaps the higher pressure end of the ionization gauge range and the lower pressure end of the capacitance diaphragm gauge (CDG) range, its linearity is superior to ionization gauges at higher pressures, it does not have the thermal transpiration problems of CDG's, and its operating principle promises stability superior to other gauges in the high-vacuum range. These features have prompted the use of MDG's to calibrate other high-vacuum gauges. The range of this application and the accuracy of the results will be limited at lower pressures by the stability of a necessary zero or offset correction, at higher pressures by viscous flow effects, and at all pressures by the accuracy and stability of the calibration constant or effective accommodation coefficient. We have used MDG's as transfer standards since receipt of a prototype unit in 1981, repeatedly calibrating them against our primary standards[1-3] and then using them to calibrate ionization gauges between 10^{-4} and 10^{-1} Pa (10^{-6} and 10^{-3} Torr). Based on this experience, we present here an analysis of the probable accuracy and stability of the MDG in the high-vacuum range, along with the results of experiments aimed at improving the stability of the offset correction.

II. OPERATION OF THE MOLECULAR DRAG GAUGE

The design and operation of the MDG has previously been described in detail.[4,5] In short, a small rotor, generally a steel bearing ball 4.5 or 4.76 mm in diameter, is magnetically levitated, spun up to ~400 Hz by an inductive drive, and allowed to coast. The ball is contained within a small tube, called a thimble, connected to the vacuum system. The rotational period of the ball can be determined by timing the signal induced in a set of pick up coils by the rotating component(s) of the ball's magnetic moment(s). Gas molecules colliding with the ball will slow it at a rate determined by the pressure P of the gas, its molecular mass m and temperature T, and the coefficient of momentum transfer σ between the gas molecules and the ball's surface. This latter factor is known as the effective accommodation coefficient, and it would have a value of unity for a perfectly smooth ball with complete momentum transfer. The rotation of the ball is also slowed by a pressure-independent residual drag (RD), caused by eddy current losses in the ball and surrounding steel structures; the rotation of the ball will be affected as well by temperature changes, which cause the ball diameter and moment of inertia to increase or decrease.

In the molecular flow regime, where gas–gas collisions are negligible, the pressure can be determined from

$$P=\frac{\pi\rho a\bar{c}}{10\sigma}\left[-(\dot{\omega}/\omega)-\mathrm{RD}-2\alpha\dot{T}_b\right], \qquad (1)$$

where ρ is the density of the rotor, a is the radius of the rotor, and $\dot{\omega}/\omega$ is the fractional rate of slowing of the rotor. $\bar{c}$ is the mean gas molecular velocity, α is the linear coefficient of expansion of the ball and $\dot{T}_b$ is the rate of change of the ball's temperature. All of the terms in the first part of the equation can be readily determined except for the effective accommodation coefficient σ, which depends on the structural roughness of the ball's surface (as opposed to the roughness caused by microscopic layers of adsorbed gases) and the molecular interaction between the gas molecules and the surface of the ball. This factor must either be determined by calibration of the MDG against a pressure standard, or estimated from previous experience with other balls. Changes in the ball's surface will affect this factor and degrade the accuracy of the gauge.

The pressure-independent residual drag RD, which typically is equivalent to nitrogen pressures between 10^{-4} and 10^{-3} Pa and may vary from ball to ball and from day to day, is compensated by an offset correction. The offset correction is determined by measuring the rate of rotational decay at "zero pressure" ($<10^{-6}$ Pa). Included in this experimental approximation to the residual drag are changes in the rotation rate caused by a changing ball temperature. Subsequent changes in the rate of temperature change will cause an error in this correction. In order to minimize such errors it is desirable to minimize the last term of Eq. (1).

An MDG user should be concerned with the short-term repeatability of the MDG and long-term stability of the effective accommodation coefficient after the gauge is calibrated. We discuss below the short- and long-term performance of MDG's used in our laboratory. If the MDG is not calibrated the user needs to estimate a value for the effective accommodation coefficient. We present the range of effective accommodation coefficients we have determined for "smooth" bearing balls. We also present the results of attempts to decrease the temperature-induced perturbations of the offset correction. We do not discuss corrections to Eq. (1) necessary at higher pressure (above $\sim 10^{-1}$ Pa) to account for gas viscosity effects. These have been discussed elsewhere.[6]

III. SHORT-TERM STABILITY

Because of its ease of use within the range of the MDG we generally calibrate ion gauges against several MDG's after first calibrating the MDG's against our primary standard. Changes in these calibrations are largely due to uncertainties in the primary standard, random errors in the gauge readings, and the short-term instability of the offset correction.

As a measure of short-term stability, 14 MDG's were calibrated in nitrogen against the primary standard, two to nine times each, at a pressure of 5×10^{-3} Pa over a two-month period. The results of these calibrations are presented in Fig. 1. Each horizontal line represents the mean value of the effective accommodation coefficient at that pressure and the box represents one standard deviation of the individual values about the mean. For some of the rotors (e.g., number 9), the standard deviation is small enough ($<0.1\%$) to appear as a horizontal line. Gauges 1–4 used 4.0 mm diam rotors which were apparently too small to be reliably suspended by all controllers; interactions between the suspension and the rotor appears to cause anomalously large variations in gauge readings. The performance of the other gauges clearly varied, with standard deviations varying from 0.02% to 0.28%. The pooled standard deviation for the 4.5 mm balls, gauges 5–14, was 0.16%.

IV. LONG-TERM STABILITY

For many users, rotors are purchased with calibration certificates or are calibrated periodically as part of a quality assurance program. For these users, long-term stability is important. Several NIST-owned MDG rotors have been in use for periods in excess of 1 yr. The long-term performance for these rotors may be seen in Fig. 2, a plot of the percent changes from an initial calibration in nitrogen as determined by repeated calibrations. The rotors are vacuum baked at 200–250 °C prior to each calibration. For clarity, each point in Fig. 2 represents the average of several points taken close together in time. One rotor in particular (indicated by line A) has been calibrated 106 times in nitrogen at 5×10^{-3} Pa over a five-year period and the calibration is seen to have systematic changes with a range of 2.4%. The net shift in the calibration of this ball over the 5-yr period has been $\sim 1.5\%$. It should be noted that generally two or more of these rotors are calibrated simultaneously as check standards; changes in the calibrations of different rotors are largely uncorrelated, indicating that the calibration changes are primarily due to changes in the effective accommodation coefficient and not due to changes in the primary standard.

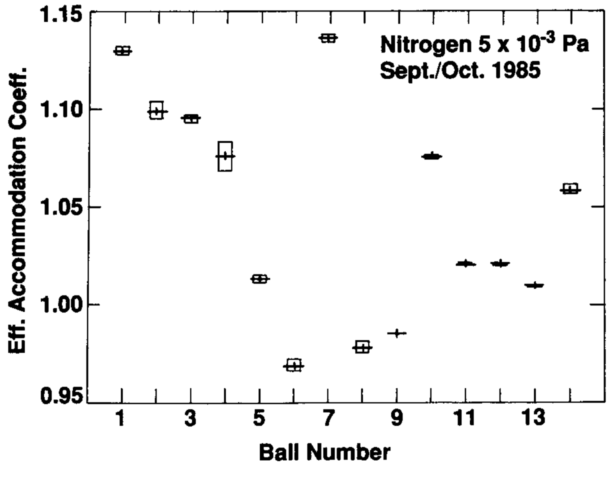

FIG. 1. Effective accommodation coefficients for 14 MDG rotors as determined by calibration against the NIST primary high-vacuum standard. The cross is the mean value and the box represents one standard deviation about the mean.

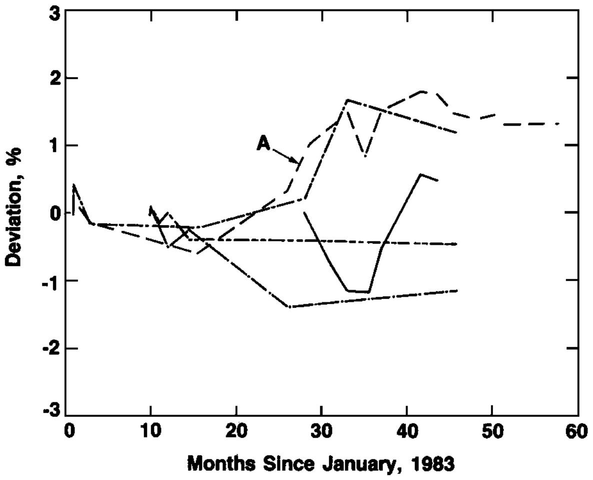

FIG. 2. Changes from initial values in the effective accommodation coefficients of five NIST rotors. Line A is discussed in the text.

We believe that the data in Fig. 2 are typical of the performance of rotors that have received "good" treatment. The care with which the rotor is treated will influence its long-term stability because the surface characteristics of the rotor determine the effective accommodation coefficient for a given gas. Smooth rotors may become scuffed or scratched if allowed to strike the thimble repeatedly while spinning or may become pitted or corroded if exposed to corrosive gases. We have observed several cases where markedly poorer performance has been obtained. After we observed a change of 8% in the effective accommodation coefficient of one rotor, examination revealed that it had become badly corroded (probably between calibrations). To avoid these problems, some users prefer to use uniformly etched rotors with the expectation that additional abrasion of the surface will not have a large effect. However, it has been shown that the calibrations of these rotors can also change with time if their surfaces become polished during use. Decreases of $\sim 4\%$ have been seen in the effective accommodation coefficients in argon of etched rotors; these variations appear to be due to mechanical polishing when the rotors were shipped unconstrained under vacuum in stainless-steel thimbles.[7] Changes in the effective accommodation coefficients in hydrogen for these same rotors were as large as 8%. We have found the effective accommodation coefficients of one group of rotors to decrease 0.5%–1.5% after baking at 150–250 °C, in one case after each of three different bakes. This appears unique to certain rotors as several rotors baked and calibrated at the same time showed no significant changes.

V. USING UNCALIBRATED ROTORS

There may be users who must or can tolerate uncalibrated rotors. In this case, as Fremerey argues,[6] the MDG can serve as a primary standard although with a relatively large uncertainty. Of greatest concern in this case is the accuracy with which the value of the effective accommodation coefficient can be predicted. Earlier work by Fremerey has shown that the maximum value that the effective accommodation coefficient can have for a completely roughened rotor is 1.27.[6] Although the effective accommodation coefficient could in theory be much less than unity, the smallest value we have observed for bearing balls is 0.97, so we can state that any steel bearing ball can confidently be expected to have an effective accommodation coefficient for nitrogen between 0.97 and 1.27. If a -3%, $+27\%$ uncertainty is tolerable (e.g., if one is measuring corrosive gases and needs an approximate pressure) the user need look no further.

However, if one limits the search for an acceptable rotor to "smooth" (as fabricated), steel bearing balls, the range of values is much smaller. The effective accommodation coefficients for 68 smooth rotors calibrated at the National Institute of Standards and Technology (NIST) for nitrogen are shown in Fig. 3. The effective accommodation coefficients range from 0.97 to 1.06. These are initial values of the effective accommodation coefficients as determined by calibration against the NIST primary high-vacuum standard. Most of these rotors were submitted by outside calibration customers and, although most of them are probably "as fabricated," we do not know the history of each nor do we know whether they were "stainless" 440C (6%–18% Cr content) or "normal" SAE 52100 (1.3%– 1.6% Cr) steel. These data are limited enough that at this time, we can only say that it appears that the effective accommodation coefficients of smooth bearing balls are not likely to differ by more than $+6\%$ or -3% from unity.

The sensitivity of the MDG varies with the square root of the molecular weight of the gas. This effect is accounted for by entering the molecular weight as an input parameter in the control unit. However, there is a further dependence of the effective accommodation coefficient on gas species. This is small for most gases but must be taken into account for accurate work. Some gases, such as hydrogen, helium, and neon, may have effective accommodation coefficients which differ from those for nitrogen and argon by several percent. We have observed differences between hydrogen, nitrogen and argon as large as 3%. The size of these differences and the relative magnitude of the effective accommodation coefficient for different gases for a given rotor will depend on the relative importance of tangential and normal momentum transfer. That is, the effective accommodation coefficient for hydrogen may be smaller than that for nitrogen for one ball but larger for another, and the magnitude of the difference may vary from essentially zero to a few percent. Early work by Comsa *et al.*,[4,8] Comsa *et al.*,[9] and Fremerey[5] illustrates this dependence.

VI. STABILITY OF THE OFFSET CORRECTION

A considerable amount of work has been done to understand the factors controlling the stability of the offset correction.[10–13] At low pressures, it is the stability of the offset cor-

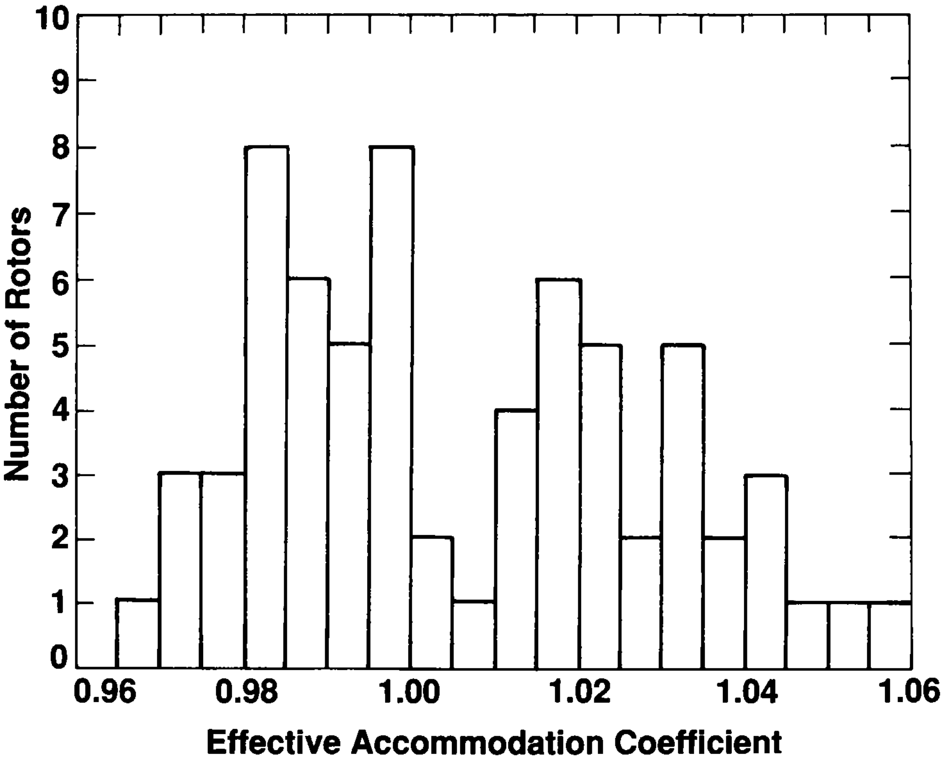

FIG. 3. Distribution of effective accommodation coefficients for 68 smooth, steel bearing balls used as rotors. Data were taken at NIST in nitrogen gas at 5×10^{-3} Pa.

rection which determines both the lowest pressure at which the MDG may be used and the short-term (hours) stability of the gauge. Several factors will cause instabilities in the offset correction. Changing the attitude (level) of the suspension head will alter the position of the ball in the suspension field and alter the residual drag.[13] Vibrations can cause perturbations in the suspension control system that may feedback to the rotor, causing second-order changes in the frequency, the signal-to-noise ratio of the rotor pickup signal will affect the imprecision of the rotation period measurement, and changes in the rotor orientation with frequency, discussed below, will alter the residual drag. However, in most cases the primary source of instability in the offset correction is temperature changes of the rotor, with corresponding changes in its moment of inertia, causing variation in rotor speed. The temperature of the rotor will vary with ambient temperature changes and with operation of the inductive drive circuit. Ambient temperature changes are damped by the long time constant (typically 1 h) of the radiative heat transfer between the thimble and rotor. Thus, longer term changes are the most disruptive. Large temperature changes occur when a rotor is accelerated from rest by the inductive drive. The suspension head, thimble, and rotor are heated, with the thimble and suspension head becoming hotter than the rotor. When the drive is turned off, the thimble and head, which are in contact with the atmosphere, begin to cool but the rotor continues to heat by radiation from the thimble. Since the only heat transfer from the rotor is radiative, it does not cool until the thimble drops below the rotor temperature, a few minutes after the drive is turned off, and then it gradually cools to ambient temperature. Due to the different time constants of heat transfer by radiation from the rotor to the thimble and by convection from the thimble, the maximum temperature difference between the rotor and the thimble, and the maximum rate of change of rotor temperature, occur ~20 min after the drive is turned off and the thimble has effectively cooled back to ambient temperature. The rotor cools towards ambient temperature with a time constant of ~1 h. Reliable values of the offset correction cannot be obtained until the rotor reaches an equilibrium temperature.

The effect of the heating caused by the drive circuit can be seen in Fig. 4, where the change in indicated pressure or change in offset correction is shown as a function of time after the drive circuit was turned off. During this time the pressure was maintained below 10^{-7} Pa so the effects are not due to pressure changes. The largest perturbation in Fig. 4 is labeled "steel ball, second generation." In this case a commercial second generation controller and suspension head were used to operate a steel ball as the rotor. During a start up from rest the second generation controller drives the ball to ~1000 Hz, and then inductively brakes it back to 415 Hz. A smaller perturbation is evident for the data labeled "Steel ball, first generation." The commercial controller and suspension head used were similar to that used for the first set of data except that the first generation controller drives the ball from rest directly to 400 Hz. This generates only $\frac{1}{4}$ to $\frac{1}{3}$ of the heat that the second generation unit does, with a significant reduction in the perturbation of the offset correction.

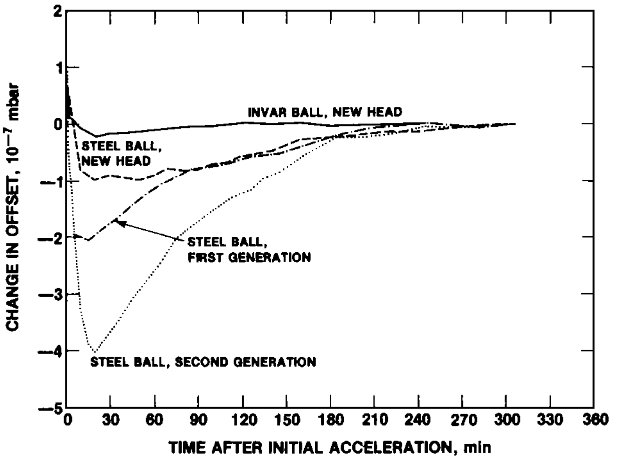

FIG. 4. Changes in the residual drags of four rotors observed as the rotors are cooled following acceleration from rest to ~415 Hz. The upper curve is for an Invar rotor, a new larger suspension head and reduced acceleration power. The next lower curve is for a steel rotor using a commercial suspension and the same controller. The third lower curve is for a steel rotor accelerated from 0 to 400 Hz. The lowest curve is for a steel rotor accelerated from 0 to 1000 to 415 Hz.

In an attempt to minimize these effects, two changes were tested: a larger suspension head with a more efficient drive circuit and better convective cooling, and a different type of rotor. The new rotor was made of Invar,[14] a high-nickel-content steel with a coefficient of thermal expansion $\sim\frac{1}{10}$ that of the steel normally used in rotors. The new suspension head has a diameter about two times larger than the commercial head. The internal structure of the head permitted more air circulation than the normal head and, because it had a 30%-40% weaker inductive drive mechanism and was better adjusted for the initial acceleration, less heat was produced during the acceleration from 0 to 415 Hz.

The performance of the new suspension head with a steel rotor can be seen in Fig. 4. The new head was operated with a second generation controller which had been modified by adjusting the efficiency of its drive circuit so that the ball was not accelerated above 415 Hz. As can be seen in Fig. 4, the perturbation of the drive circuit is further reduced when the new suspension head is used with an Invar ball. The effects of ambient temperature changes will also be reduced for an Invar rotor.

Unfortunately, the Invar rotor was more susceptible to a different type of instability. The residual drag of some rotors has a significant dependence on frequency. It is believed that this may be due to a changing alignment between the magnetic and inertial moments of the rotor as it slows down. Typically, this causes the rotating component of the magnetic moment to decrease with frequency, inducing eddy currents in the thimble and suspension head with a corresponding reduction in the residual drag. This effect is apparent in many balls, but the Invar rotors had a large dependence of the residual drag on the frequency. We believe this was due to a large asphericity of the Invar rotors. In addition, each time the inductive drive was activated, the signal strength decreased indicating that the rotor magnetization had changed. This effect saturated with repeated accelerations but could be a significant problem for work at low pressures. Further work is planned with other Invar rotors.

VII. CONCLUSIONS

Our experience at NIST in using the MDG as a transfer standard has led to increasing confidence in its stability, precision, and robustness. We have seen short-term (one to two months) random errors of a few tenths percent and long-term changes of 3% or less over several years time. Repeated calibrations of eight rotors used in industrial calibration laboratories have shown changes of − 2.3% to 2.7% over periods of ~2 yr. The largest observed rate of change was 1.6%/yr. However, because the effective accommodation coefficient depends directly on the surface characteristics of the rotor, care must be taken to ensure that the surface is neither damaged nor changed by mechanical or chemical action. With great care, the effective accommodation coefficient or calibration constant can be expected to change by no more than 1% or 2% in a year's time. Periodic comparisons of different rotors can increase the confidence in their stability. Smooth bearing balls can be assumed to have an effective accommodation coefficient of 1.00 within a range of − 3%, + 6%.

A different design of the suspension head can significantly reduce the perturbations caused by the inductive drive. Reductions of this effect and that due to ambient temperature changes can be achieved with Invar rotors. However, additional work will be required to determine if the large frequency dependence of the Invar rotor offset corrections can be reduced, and there is some concern about the stability of the soft Invar surface. We intend to investigate the properties of Invar rotors with a hard surface coating (titanium nitride).

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[1] K. E. McCulloh, J. Vac. Sci. Technol. A **1**, 168 (1983).

[2] K. E. McCulloh, C. R. Tilford, S. D. Wood, and D. F. Martin, J. Vac. Sci. Technol. A **4**(3), 362 (1986).

[3] K. E. McCulloh, C. R. Tilford, C. D. Ehrlich, and F. G. Long, J. Vac. Sci. Technol. A **5**, 376 (1987).

[4] George Comsa, J. K. Fremerey, and Bernd Lindenau, in Proceedings of the Eighth International Vacuum Congress, Cannes, France, 1980 (Supplement to Le Vide, les Couches Minces, no. 201), Vol. III, p. 218.

[5] J. K. Fremerey, Vacuum **32**, 685 (1982).

[6] J. K. Fremerey, J. Vac. Sci. Technol. A **3**, 1715 (1985).

[7] G. Messer, P. Rohl, G. Grosse, and W. Jitschin, J. Vac. Sci. Technol. A **5**, 2440 (1987).

[8] George Comsa, J. K. Fremerey, and Bernd Lindenau, in Proceedings of the Seventh International Vacuum Congress and Third International Conference on Solid Surfaces, Vienna 1977 (R. Dobrozemsky *et al.*, Vienna, 1977), Vol. I, p. 157.

[9] George Comsa, J. K. Fremerey, B. Lindenau, G. Messer, and P. Rohl, J. Vac. Sci. Technol. **17**, 642 (1980).

[10] J. K. Fremerey, Rev. Sci. Instrum. **43**, 1413 (1972).

[11] J. K. Fremerey, Phys. Rev. Lett. **30**, 753 (1973).

[12] K. E. McCulloh, S. D. Wood, and C. R. Tilford, J. Vac. Sci. Technol. A **3**, 1738 (1985).

[13] M. Hirata, J. Isogai, and M. Ono, J. Vac. Sci. Technol. A **4**, 1724 (1986).

[14] Citation of a commercial name conveys neither NIST endorsement of the manufacturer nor belief that the product is necessarily the best available.