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<sup>11</sup> Better than 99.98% <sup>3</sup>He vapor pressure standard grade, available from Monsanto Research Corporation, Miamisburg, Ohio 45342.

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## An Improved Thin-Film Gauge for Shock-Tube Thermal Studies\*

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An improved type of thin-film heat gauge has been developed and used to measure heat-transfer rates from a shock-heated plasma to the end wall of a shock tube. The gauge is a resistance thermometer consisting of a triple-layered sensor coated by a moderately thick film of SiO<sub>2</sub>. It was used to measure heat transfer from nonionized argon at temperatures of up to 16 000 K. A marked increase in heat transfer at the onset of ionization near the wall indicates that the gauge can be used to obtain the contribution of free electrons to the thermal transport.

### INTRODUCTION

Thin-film heat-transfer measurement techniques are widely used in experimental gas dynamics.<sup>1</sup> Heat flow from a hot gas through a thin film to a cold model or wall raises the resistance of the temperature-sensitive film, producing an observable voltage output signal. Development work on such devices has slowed down in recent years because of seemingly unsurmountable difficulties in extending their range of usefulness. Investigators found that in high-temperature shock-heated gases the gauge is shorted out by the electrons in the adjacent gas, and is sometimes destroyed by strong pressure loading. Attempts were made by some workers<sup>2</sup> to use thin silicon oxide-type dielectric coatings evaporated over the gauge to insulate the latter while essentially preserving most of its heat-transfer measurement capability. However, for reasons that were not usually clear at the time, the coatings were not too helpful. Our own microscope studies of similar coatings—typically 1000 Å thick—indicate that such dielectric covers usually contain tiny “pinholes” which provide a shorting path after all.

It was this experience which led to the use of coatings having a thickness of 8000 Å in the present studies.<sup>3</sup> Application of the latest vacuum-evaporation techniques produced a good dielectric shield whose quality has been

verified by microscopic observation and proved by the practically noise-free response of the gauge. Although the thickly-coated gauge is less perfect as a fast time-response device, the transient thermal behavior of shocked gases may be calculated from the experimental records by use of a transfer function determined through careful calibration techniques. The inconvenience of introducing the transfer function is justified by the extended temperature range over which the gauge provides heat-transfer data: e.g., measurements were made in nonionized argon to over 16 000 K, whereas previous investigators<sup>4</sup> were limited to about 5000 K with the resistance thermometer method. In addition, the gauge recorded the heat-transfer change during ionization equilibration, a phenomenon which had not been previously monitored by this type of instrument. The development of this heat gauge was motivated by the desire to complement interferometric measurements<sup>5</sup> of the argon thermal conductivity by an independent technique in the same facility.

### GAUGE DESIGN

The performance of a thin-film heat gauge is very dependent upon careful choice and preparation of the substrate. Quartz was used because its low thermal diffusivity gives a good film-temperature sensitivity for a given heat-

transfer rate and because the product of its thermal conductivity  $\kappa$ , density  $\rho$ , and specific heat  $c$ , which relates temperature rise to heat transfer, changes less with temperature than other commonly used dielectric substances such as Pyrex. Fused quartz of a good optical grade is virtually bubble-free and hence uniform, but microscopic observation of its polished surfaces revealed polishing rouge embedded in the surface. This caused weak spots in the films evaporated on it. Instead of depositing the film on the polished surface, as done by earlier investigators, we surface-melted the quartz prior to film deposition. The surface remained somewhat wavy, but smooth, after the rouge either burned off or sank into the quartz. Platinum was chosen as a sensor because of its good temperature sensitivity, corrosion resistance, high melting point, and linearity of its thermal coefficient of resistivity. Evaporation was performed under vacuum in order to obtain uniformity of coatings. The poor adhesion between platinum and quartz was overcome by evaporating a 40 Å thick chromium layer on the quartz first. At the time of deposition the chromium received oxygen from the quartz substrate and oxidized to a thickness of about 30 Å. The additional pure chromium formed a good bond for platinum. The platinum strip was evaporated immediately after the chromium in the same vacuum chamber in order to avoid oxidation from the atmosphere. The platinum film was made about 400 Å thick which corresponds approximately to the mean free path of electrons in the metal. An additional chromium layer (40 Å thick) was evaporated on the platinum sensor during the same operation and formed a thin dielectric layer because of oxidation with air after removal from the chamber. This raised the temperature limit for useful heat-transfer-rate experiments from 5000 to 8000 K in argon without noticeably affecting the response time of the gauge. In order to test at still higher temperatures, an additional heavy dielectric coating was evaporated on top of this triple-layered sensor.

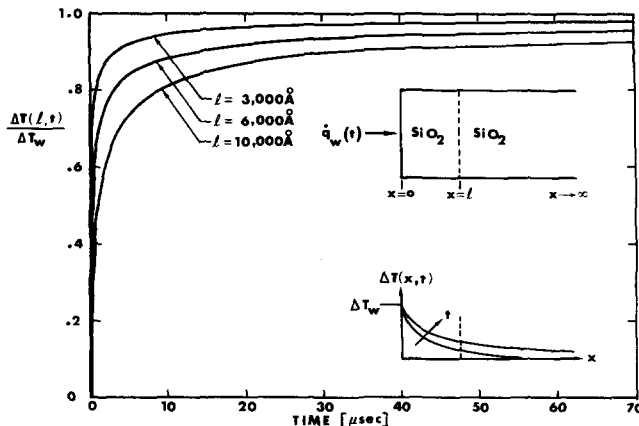


FIG. 1. The temperature history at the point  $x=l$  inside a homogeneous semi-infinite quartz slab due to a temperature step at  $x=0$ .

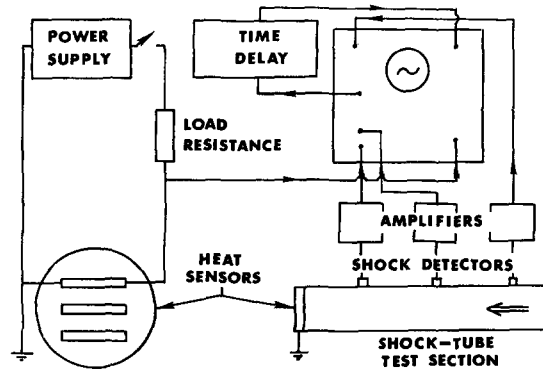


FIG. 2. Schematic diagram of the basic circuitry for heat-gauge measurements.

Silicon monoxide<sup>6</sup> is the most widely used dielectric compound in thin-film evaporation work. SiO was heated above its sublimation temperature in an oxygen-rich vacuum chamber and deposited as SiO<sub>2</sub> with small traces of SiO and Si<sub>2</sub>O<sub>3</sub>. Chemical matching of the coating to the quartz substrate (SiO<sub>2</sub>) simplified the calculation of the temperature distribution in the multilayered gauge and therefore encouraged the use of a heavy coating. The temperature distribution  $\Delta T(x,t)$  in a homogeneous semi-infinite slab due to a temperature step on the surface  $\Delta T_w$  is given by<sup>7</sup> a complementary error function,

$$\Delta T(x,t) = \Delta T_w \operatorname{erfc}(\tau/t)^{1/2}, \quad (1)$$

where

$$\tau = x^2 \rho c / 4\kappa = x^2 / 4a, \quad (2)$$

and  $t$  is the time after raising the surface temperature;  $\tau$  is the thermal transit time which depends on the thermal diffusivity  $a$  and the distance from the surface  $x$  (see Fig. 1).

The thermal transit time for the Cr-Pt-Cr sensor is so small that any temperature gradient in the sensor itself may be assumed negligible at any instant during a typical shock-tube experiment of 70  $\mu\text{sec}$  duration. However, the thermal diffusivity of SiO<sub>2</sub> is so much smaller than that of the sensor that a relatively thick dielectric coating on top of the sensor retards its temperature response during typical shock-tube times. Since the chemical composition of the coating is essentially that of the substrate, the sensor may be treated as an interface at distance  $l$  from the surface of a homogeneous semi-infinite quartz slab; and the time response of the gauge may be calculated as a function of coat thickness. Figure 1 illustrates that in spite of a heavy dielectric coating on top of the sensor, a temperature step on the wall surface raises the temperature quite adequately at point  $x=l$ . The wall surface temperature may then be calculated from the sensor response by use of Eq. (1).

The SiO<sub>2</sub> coating was approximately 8000 Å thick and covered a 40 Å Cr-400 Å Pt-40 Å Cr sensor of 1.23 cm length and 0.11 cm width. Three such strips were deposited

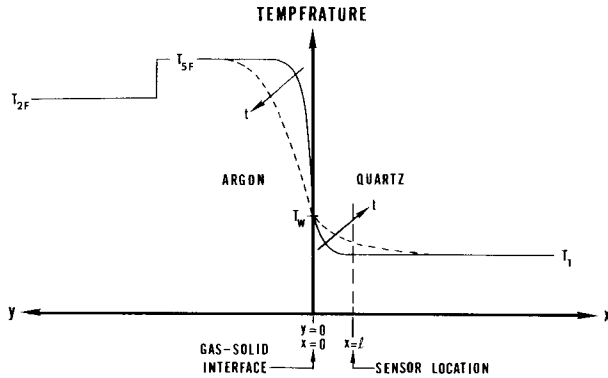


FIG. 3. Temperature distribution in gas and solid during heat-transfer-rate measurements on shock-tube end wall. For high temperature experiments,  $l=8000 \text{ \AA}$ .

on a 2.5 cm diam quartz disk which was embedded in the end wall of a shock tube of 5 cm $\times$ 5 cm square interior cross section. The voltage response in the gauge was related to its temperature rise through its thermal coefficient of resistance  $\alpha$  which was found from measurements of gauge resistance vs temperature in a heated oven,

$$\alpha = (1.731 \pm 0.009) \times 10^{-3} [\text{K}^{-1}].$$

The coefficient  $\alpha$  is smaller than that of pure bulk platinum by a factor of about 2, showing that thin-film properties have to be individually determined each time a new gauge is used for experiments. This provides further motivation for wanting a relatively nondestructible gauge.

The basic circuitry, shown in Fig. 2, consisted of a constant current source for the gauge and an oscilloscope to record the shock speed and the heat-transfer signal. The gauge current of 20 mA was supplied by a 500 V power supply with a 25 k $\Omega$  load resistor: Initial gauge resistance was 62.65  $\Omega$  at 25°C.

### CALIBRATIONS AND RESULTS

The temperature distribution in the gas and the solid is shown in Fig. 3 for a typical experiment on the shock-tube end wall. The stationary gas behind the reflected shock is initially at uniform temperature  $T_{2F}$  and the solid at uniform temperature  $T_1$ . Contact between the two media raises the wall-surface temperature to an intermediate  $T_w$

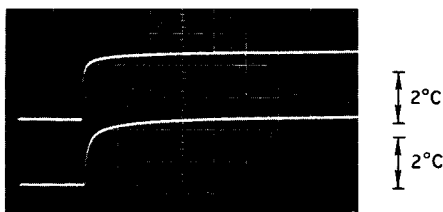


FIG. 4. Typical heat-gauge calibration run in Ar.  $M_s=2.33$ ,  $P_1=50$  Torr,  $T_1=297 \text{ K}$ ,  $P_5=1177$  Torr,  $T_5=1,334 \text{ K}$  (2.8 mV, 10  $\mu\text{sec/div}$ ). Upper trace—Obtained from gauge with 4000  $\text{\AA}$  dielectric coating; lower trace—from gauge with 8000  $\text{\AA}$  dielectric coating.

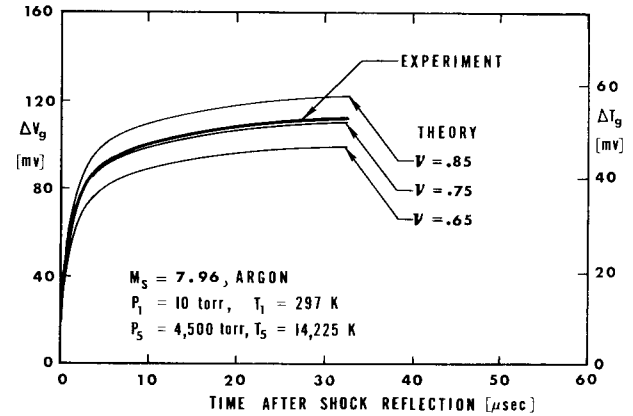


FIG. 5. Experimental and theoretical response of heat gauge embedded in shock-tube end wall. Parameter  $\nu$  represents thermal conductivity-temperature exponent. Experiment corresponds to Fig. 6(b).

and creates thermal transition regions in gas and solid. While the surface temperature  $T_w$  remains essentially constant, the transition regions grow with time such that the sensor located at distance  $l$  within the end wall measures a temperature rise which slowly approaches that of the wall surface.

The gauge response was calibrated in dense, low-temperature argon where gas conditions and heat-transfer rate are well known. Figure 4 shows a typical calibration run. The two traces recorded the response of two different elements on the same substrate which differ in the thickness of their coatings. The lower trace was obtained with a sensor coated with 8000  $\text{\AA}$  of  $\text{SiO}_2$ , as described above, whereas the upper trace displays a faster response corresponding to its thinner  $\text{SiO}_2$  coating of 4000  $\text{\AA}$ . Only thermal conductivity data from the more heavily coated gauge were considered valid, since some records obtained in the high Mach number range with the less heavily coated gauges displayed large and irregular fluctuations.

The gauge was used to generate data in argon, nitrogen, and carbon dioxide. It did not change its cold resistance after a total of 90 shock-tube experiments, 25 of which were combustion driven, and consistent data were obtained on duplicate runs. Linearity between gauge response and gauge current was verified by conducting an experiment at half the normal current.

Figure 5 shows how experimental temperature signals obtained with nonionized argon compared to theoretical gauge response. The gauge was assumed to consist of a semi-infinite quartz slab with the sensor at interface  $x=l$  distant from the end of the solid. The thermal conductivity  $\kappa$  was assumed to be represented by the familiar temperature dependent power law

$$\kappa \propto T^\nu. \quad (3)$$

This simple expression permits relatively easy numerical

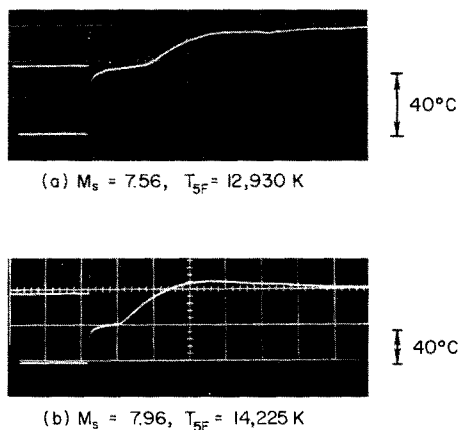


FIG. 6. Response of end-wall heat-transfer gauge in argon;  $P_1 = 10$  Torr,  $T_1 = 297$  K, sweep speed =  $50 \mu\text{sec/div}$ . Second rise represents contribution of free electrons.

calculations of the entire thermal boundary layer profile and of the heat transfer to the wall for a given  $\nu$ . The exponent  $\nu$  was then determined by least-square fitting of theory to experiment.<sup>3</sup>

Tests in high-temperature argon ( $T > 10^4$  K) also showed the effect of gas ionization on heat transfer to the wall. This is the first time, to the authors' knowledge, that such

measurements have been recorded by this type of gauge. Figure 6 shows that the heat transfer to the end wall is increased significantly after gas ionization. The ionization relaxation time may also be measured from the separation of the two rises of the temperature-time curve. The time decreases with increase in Mach number (i.e., gas temperature) and agrees with relaxation time data obtained by optical interferometric techniques.<sup>5</sup> Finally, we note briefly that tests were conducted in molecular gases as well. The results show variations in heat transfer associated with kinetic rate processes and demonstrate the capability of the device for such studies.

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## A Simple Circuit for cw NMR Measurements

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A simple circuit for cw NMR measurements is discussed. The purpose of this circuit is similar to that of bridge arrangements: The rf at the input of the receiver is compensated by an additional branch within the circuit consisting of a potentiometer and a phase shifter. As shown by theoretical calculations, the sensitivity of the discussed arrangement is higher than similar uncompensated circuits and higher than bridge arrangements as well. The  $^{203}\text{Tl}$  and  $^{205}\text{Tl}$  resonances measured with this circuit show signal-to-noise ratios comparable to the signal-to-noise ratios of the resonances measured with a commercial wide-line NMR spectrometer.

### INTRODUCTION

Common setups for detecting nuclear magnetic resonance absorption or dispersion are the bridge,<sup>1-3</sup> the crossed coils,<sup>4</sup> and the oscillator<sup>5,6</sup> arrangements. For a review on NMR instrumentation see Refs. 7-9. The simplest circuits used for NMR measurements are probably those described by Rollin<sup>10</sup> (Fig. 1) and by Gabillard.<sup>11</sup> These circuits have one disadvantage: The total voltage<sup>12</sup>  $U_1$  (across the tuned circuit) enters the receiver. Since the NMR signal is only a very small change of  $U_1$ , the rf amplification is limited by the amplitude of  $U_1$ . In order to

compensate the voltage  $U_1$  at the input of the receiver, bridge arrangements as mentioned above have been developed.

By use of a phase shifter (Helidel, model 8811, Beckman Instr.), with a maximum delay time of  $0.1 \mu\text{sec}$  (phase shift of more than  $2\pi$  at 15 MHz) an arrangement with compensation has been developed (Fig. 2) almost as simple as the circuit described by Rollin. The location of the resonance circuit, which is not grounded but is in series with the input impedance  $Z_1$  of the amplifier, is one obvious difference with respect to the circuits described up to now.