THIN-FILM RESISTANCE THERMOMETERS ON SILICON WAFERS

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ABSTRACT

We have fabricated Pt thin-film resistors directly sputtered on silicon substrates to evaluate their use as resistance thermal detectors (RTDs). This technique was chosen to achieve more accurate temperature measurements of large silicon wafers during semiconductor processing. High purity (0.999968 mass fraction) platinum was sputter deposited on silicon test coupons using titanium and zirconium bond coats. These test coupons were annealed and four-point resistance specimens were prepared for thermal evaluation. Their response was compared with calibrated platinum-palladium thermocouples in a tube furnace. We evaluated the effects of furnace atmosphere, thin-film thickness, bond coats, annealing temperature, and peak thermal excursion of the Pt thin films. Secondary ion mass spectrometry (SIMS) was performed to evaluate the effect of impurities on the thermal resistance coefficient, α.

We present typical resistance versus temperature curves, hysteresis plots versus temperature and an analysis of the causes of uncertainties in the measurement of seven test coupons. We conclude that sputtered thin-film platinum resistors on silicon wafers can yield temperature measurements with uncertainties of less than 1 °C, k=1 up to 600 °C. This is comparable or better than commercially available techniques.

INTRODUCTION

Thermal processing of semiconductors is a critical, capital intensive step in achieving high yields and profitability in the manufacturing of electronic chips such as ASICs (application specific integrated circuits) and DRAMs (dynamic random access memory). Many techniques have been developed to control the temperature of the silicon wafer during thermal processing. One way is to monitor the wafer surface temperature using thin-film sensors on instrumented test wafers. These sensors are also used to calibrate the lightpipe infrared thermometers used to monitor the wafer temperatures during processing. Members of the semiconductor fabrication industries have encouraged NIST to investigate the use of thin-film Pt resistance thermometers directly on the wafer to monitor temperatures during thermal processing.

Thin-film platinum resistance thermometers (PRT) are commercially available on aluminum oxide substrates. Diehl reported on highly accurate thin-film Pt resistance thermometers on aluminum oxide (alumina) substrates within a tolerance band of ± .03 °C + 0.0005 t °C up to 600 °C [1]. Historically, the International Electrotechnical Commission Technical Committee 751 has developed standards on platinum resistance

thermometers (wire and film) [2]. These include standards for testing, resistance specifications, and tolerance bands. This standard is only useful for platinum thin-film resistors on aluminum oxide (alumina) substrates. The alumina substrates are particularly useful for platinum thin films because they do not react chemically with Pt at any temperature and the thermal expansion coefficient of Pt and aluminum oxide are very similar. This match in coefficients minimizes stress and strain between film and substrate.

The goal of this project was to develop a system using platinum thin-film resistance thermometers capable of calibrating lightpipe radiation thermometers (LPRTs) in industrial rapid thermal processing (RTP) for the temperature range 300 °C to 600 °C. This technique should be capable of measuring wafer temperatures during the processing cycle with a standard uncertainty of less than 1 °C (k=1).

We at NIST have investigated thin-film thermocouples for high temperature measurements during processing on silicon wafers. These studies have determined the uncertainty of temperature measurements on silicon wafers using compound thermocouples. The compound thermocouple has wire thermocouples bonded to the surface of the wafer and differential thin-film thermocouples reaching from the wire terminus to the point on the wafer where the measurement is required. Pt/Pd thin-film thermocouples were found useful up to 800 °C [3] and Rh/Pt were found best up to 900 °C [4] and up to 1000 °C [5] if covered with a thin sputtered film of aluminum oxide. The calibration of these wire/thin-film thermocouples requires a multistep procedure and has been documented [6]. Standard uncertainties in the 1 °C range were achieved for the thermocouples used for calibration of LPRTs in semiconductor rapid thermal processing equipment [7].

Some research on Pt thin films on low thermal expansion substrates has been reported in the literature. Bolker and Sidles [8] studied thin-film Pt resistance thermometers on 7059 glass [9] which has a low expansion coefficient of 4.5×10^{-6} °C^{-1} (0 °C to 300 °C). They did not determine the uncertainty of their measurements and were interested primarily in substrate temperature changes during sputtering.

FABRICATION AND TESTING PROCEDURES

We fabricated Pt thin-film resistors on silicon-wafer test coupons and evaluated their resistance as a function of temperature in a tube furnace. Calibrated Pt/Pd wire thermocouples were welded to each end of the resistor to determine the film's temperature. Calibrated Pt/Pd thermocouples were used because of their low measurement uncertainty (±1°C at 700 °C, k=1). The results of cycling these test coupons between room temperature and up to 700 °C were plotted for heating and cooling to determine the thermal coefficient of resistance and the hysteresis during the thermal cycle.

The test coupon for the 4 point Pt resistance test is a 50 mm × 10 mm × 0.75 mm thick silicon wafer with a 1.5 mm wide and 40 mm long strip of sputtered Pt for the resistor. We used the four point resistance test that places a known current through the resistor and

measures the voltage drop between two points within the current connections. This eliminates errors caused by the resistance of the leads to the resistor. The test coupons were magnetron sputtered from Pt targets (99.9968 % mass fraction) in (0.5 to 0.7) Pa of argon with less than 10^{-4} Pa of H_2O background gas. Also four test coupons were sputtered commercially to our specifications with the same masks on our Si substrates. All coupons were annealed at 850 °C to 900 °C for one hour in nitrogen or in air to eliminate the residual stresses from sputter deposition and develop a more stable grain structure. The Pt films were bonded to the thermally oxidized Si wafers and high purity alumina substrates with 3 nm to 10 nm of sputtered Ti or Zr. We investigated Pt films from 0.35 μm to 3.6 μm thick. For comparison, the first two Pt thin-film resistors were produced on high-purity, high-density alumina substrates with the same dimensions and fabrication procedures.

The test coupon had 0.1 mm diameter Pt and Pd wires welded to the test film at each end of the thin film with a parallel gap welder. The 0.1 mm wires were welded to 0.25 mm Pt and Pd lead wires with a fine tube premix burner on a water welder. These lead wires were secured with alumina cement in alumina tubes (1 mm diameter) fixed in the Si wafer. Thus the 4-wire resistance test had Pt/Pd thermocouples at each end to determine its temperature.

The 4-wire dc resistance measurements were made with the test coupon in a fused quartz tube in a three zone furnace using both air and nitrogen atmospheres. Oxygen concentrations were measured using a ZrO_2 oxygen sensor in the flowing nitrogen atmosphere. The test coupon's Pt/Pd thermocouples follow the average temperature during heating and cooling as well as providing lead wires for the 4 wire resistance measurement of the Pt thin film. Thermocouple measurements were made with a distilled water/ice reference cell. Voltages were measured with an Hewlett Packard 3458A [9] 8½ digit multimeter. The measurements were automated with a scanner and a PC.

After the initial tests on alumina substrates, we have made over 80 test runs on Si coupons. Each test took about six hours for the heating and cooling and had two or more quasi-stable plateaus during heating. Each coupon was annealed in air before determining the resistance (R) as a function of temperature (t). Effectively all of the tests indicated that the R versus t data could be fitted with a quadratic function similar to the functions described in IEC 60751-1983 [1]. The residuals from these fits were quantified for each run and were very small if points were ignored that had heating or cooling rates greater than 5 °C per minute. This same function applied to both heating and cooling curves. No problems with adherence of the films were encountered.

RESULTS

A table of the results of testing seven wafer coupons with sputtered thin-film Pt resistors is presented in Table I.

Table I
Test results of Pt thin-film Resistors on Silicon

	R_0 ohms	α °C^{-1}	T_{max} °C	ΔT °C	ΔT_R °C
Pt 61.4	3.8	3.28	720	5	3*
n13		3.32	440	1.1	0.5
n7		3.29	670	3.3	2.2
n14		3.28	440	2.0	1.0
n2		3.29	720	5	3
Pt 62 n3	8.8	3.16	720	5	3
n15		3.20	720	8	5
Pt 69 n14	3.9	3.17	700	2.5	1.4
n15		3.18	700	2.5	1.5
n5		3.18	630	1.8	1.4
n4		3.28	700	1.6	1.5
Pt 75z.3	5.5	3.37	725	11	9*
.4		3.30	715	8	5
n6		3.29	715	2.5	1.8
n8		3.31	715	2.5	1.7
n10		3.31	715	2.3	1.5
n14		3.31	715	2.2	1.2
Pt81 n4	5.9	3.20	725	7	5*
16		3.30	690	3.5	1.9
Pt 93.1	6.2	3.06	710	4.0	2.6
.2		3.06	710	2.8	2.5
.4		3.07	710	2.5	2.4
Pt 85n3	4.4	3.18	715	4.2	3.5
.5	4.2	3.23	715	1.0	0.9
.7	4.3	3.35	715	1.0	0.9

* fast cycle

The resistance at room temperature (R_0) is inversely related to the resistor's thickness which ranges from 0.35 μm (Pt 62) to 0.77 μm (Pt 61). Pt 75z had a Zr bond coat; all the others had Ti bond coats. Alpha was calculated from the equation $\alpha = (R_{100}\text{-}R_0/100R_0)$. R_0 is the resistance at 0 °C and R_{100} is the resistance at 100 °C. T_{max} °C indicates the maximum temperature during the test run. It was determined that hysteresis problems were aggravated by cycling over 630 °C. We recommend limiting the test and its use to 630 °C to improve the measurement uncertainty. ΔT_R °C is the maximum deviation from the quadratic fit of the equation $R = xT^2 + yT + z$. ΔT °C is the maximum hysteresis at any given temperature.

All of the values of the temperature coefficient of resistance, α, fell between 0.00295 °C^{-1} and 0.00350 °C^{-1}. The largest values of α were found with the alumina substrates and the thicker films in air after the highest annealing temperature. A typical plot of R versus t is presented in Fig. 1. The sample Pt film was 0.77 μm thick and had one of the thinnest layers (≅5 nm) Ti bond coat. The coefficient α was 0.00320 °C^{-1} in this test. Other tests of the same sample in nitrogen yielded α's between 0.00317 °C^{-1} and 0.00320 °C^{-1}. The α's

are significantly lower than the standard coefficient for industrial PRTs of 0.00385 °C^{-1} The 0.00385 value corresponds to intentionally doped Pt. Reference grade Pt has α = 0.003926.

We also determined the residuals between the data and the quadratic curve fitting equations used to determine α. Figure 2 illustrates this information for sample Pt81. Notice the large deviation observed when the heating rate is nearly 12 °C per minute (from 506 °C to 563 °C in 5 min). This deviation is almost 4 % (0.232/5.90). Much better compliance is observed when we use the measurements of resistance at thermal arrests (see Fig. 3 for sample Pt69sn15a). Here most of the measurements fall within 0.3 % (0.012/3.89) or within 1 °C of the calculated curve. An interesting anomaly at 270 °C may indicate a real physical problem with the Pt film such as gas absorption or oxidation.

Two Pt thin-film resistors were fabricated to explore the effect of film thickness greater than 1.0 μm on α. Our earlier films ranged in thickness from 0.35 μm to 1.0 μm. Because the earlier measurements indicated higher α's with the thicker films (0.7 μm to 1.0 μm) we investigated 2.0 μm and 3.6 μm thick films. The average α after 20 test cycles on these Pt films was 0.00322 for the 2.0 μm thick film and only 0.00303 for the 3.6 μm films. The average α in air was slightly higher than that measured in nitrogen. These results indicate that we found no advantages for films thicker than 1.0 μm. The evaluation of the commercially produced films yielded lower α's but very similar results. SIMS measurements indicated a higher level of impurities was probably the cause of the higher α's.

We measured the resistance versus temperature of ten NIST fabricated Pt thin-film resistors on Si wafers including a range of a factor of ten in Pt thickness (from 0.35 μm to 3.6 μm), a range of a factor of three in Ti bond coat thickness, and one with a Zr bond coat. We made the measurements in air and in nitrogen (from 40×10^{-6} to 350×10^{-6} volume fraction of oxygen). There was no debonding of the films even with thicker films. One of the samples in Table I and Fig. 4 (at 0.63 μm) was the Pt thin film bonded with Zr rather than Ti.

The sensitivity of α to film thickness was measured in two ways. First, we plotted the α coefficients of each sample using the average of three to seven tests both in air and in nitrogen versus the film thickness (Fig. 4). The average thickness of the film was determined from weight gain after sputtering. Two of these thickness measurements were confirmed with secondary ion mass spectrometry (SIMS) depth profiling (within 10 %). SIMS analysis also indicated 10^{-4} to 3×10^{-4} atomic fraction Cu and Fe in the Pt film (indicating a 2 % decrease in α) [10]. Although there is some scatter with the thinnest films it is also apparent that higher α's are measured in air compared to those measured in nitrogen. We were not able to correlate Ti thickness with α. In Figure 5, α is plotted versus the Pt film resistance at 0 °C. These results were very similar to those related to thickness with slightly lower α's in nitrogen (40×10^{-6} to 350×10^{-6} volume fraction of oxygen).

The temperature coefficient of resistance for our thin films is well below the α = 0.00385 °C^{-1} obtained with bulk wire resistors. As indicated above the thickness of the film is not the primary cause of this deviation. The two most likely causes are composition and crystalline defects such as those caused by the mismatch in thermal coefficients of expansion between film and substrate. To explore the effect of composition four samples of the NIST fabricated Pt and one commercially produced thin-film resistor were analyzed by secondary ion spectroscopy with depth profiling. We analyzed for Al, Ti, Fe, Mg, Zr, and Cu in the Pt thin films. Three NIST films were Ti bonded and one was Zr bonded. The commercially produced film was Ti bonded. Estimated uncertainties were three times that for Al. Ti, Cu, and Fe. A profilometer was used to calibrate the concentration as a function of depth. The samples that were annealed at 900 °C had their bond coats of Ti and Zr diffused throughout the Pt thin film. The bulk concentration of the Ti was near 0.2 % atomic fraction. Our target purities were measured by the supplier to be 99.9968 % Pt atomic fraction with Fe at 1 ppm (1 ppm = 10^{-6} weight fraction), Ni at 3 ppm, Pd at 10 ppm, and Ag at 15 ppm. The SIMS measurement indicated Fe concentrations of 0.1 % and 0.2 % atomic fraction in the bulk for the two NIST samples and much higher near the surface where O ions enhance the signal. Compared to Fe, the Ni and Cu concentrations have little effect one the α values, as predicted by Cochrane [10]. The iron impurity, however, might have a ≈ -10 % [10] effect on α on the NIST fabricated resistors. The commercially deposited Pt had approximately twice the Fe concentration. It seems likely that the Fe impurity may be responsible for much of the depressed value of α in the thin films.

One of the test uncertainties is related to the difference in temperature across the thin-film platinum resistor. To reduce this we upgraded our furnace temperature control and measurement for further experiments. We made major changes to reduce the thermal gradients in our three-zone tube furnace used for thin film testing. A new PID control system with quoted 0.1 % accuracy and 0.1 s sampling cycle was installed. All three heating coils were replaced and the thermal profile was reestablished. With these improvements we could ensure a temperature differential for the length of the platinum thin-film resistor of less than 1 °C. It was found that the residuals to the quadratic fit to the data did not correlate to the temperature difference between the ends of the resistor (up to 3 °C). This effect is displayed in Fig 6 for temperatures between 220 °C and 604 °C. In Fig. 7 it can be seen that the cooling rate $(t_{n+1}) - t_n$ did have a strong effect on the residuals with cooling rates above 2 °C/min for temperatures between 236 °C and 570 °C, where t_n is the temperature at one point and t_{n+1} is the temperature one minute later. Other tests confirmed this finding with heating rates of up to 25 °C/min.

Typical heating and cooling curves with the improved furnace are presented in Fig. 8 where α is 0.00323 °C^{-1} and the quadratic fit is $R = -2.300 \times 10^{-6} t^2 + 1.430 \times 10^{-3} t + 4.368$. The residuals from that plot (Figure 9) between 30 °C and 605 °C are all less than 1 °C.

These data prompt a discussion about the uncertainty of the measurements. The Pt/Pd wire thermocouples that are used to measure the temperatures at both ends of the Pt thin-film resistor have been calibrated with a NIST calibrated Au/Pt thermocouple to a 0.3 °C uncertainty ($k = 1$) when used with a distilled-water ice point. The uncertainty of the 4-

point resistance measurement leads to a standard uncertainty of 0.1 °C, and the standard uncertainty of the voltage measurement was 2 μV or 0.3 °C at 600 °C. By plotting the residual ohms versus the differential in temperature of the ends of the Pt film we found that a 1 °C limit on the difference between ends would lead to less than a 0.3 °C uncertainty of the measurement. A summary of the uncertainties for static measurements is presented in Table II. Achievement of the RMS 0.6 °C uncertainty requires a calibration of the thin-film Pt resistor with a comparison thermometer or a highly consistent fabrication process of the calibrated thin film.

CONCLUSIONS

Sputtered thin-film platinum resistors on silicon wafer can yield temperature measurements with uncertainties of <1 °C, k=1 up to 600 °C. This is comparable or better than any other method of on-wafer thermometry.

The temperature coefficient of resistivity (α) of the films up to 1 μm thick is approximately 15 % lower than the industrial specification for bulk Pt wire resistors and decreases with lower thickness. This is likely to be related to a higher film resistivity from impurities, point defects, and surface effects.

Significant hysteresis is present with fast (> 5 °C/min.) heating or cooling of the Pt film on the Si wafer. It is assumed that a major part of this hysteresis is related to plastic deformation forced on the Pt film by the Si wafer because of the mismatch in thermal coefficients of expansion (approximately 5×10^{-6} °C^{-1}). Slower rates of heating or cooling (< 2 °C / min) probably permit recovery and lower stress levels in the Pt thin films. Calibration of the LPRTs in RTP tools can be achieved without rapid heating and cooling rates. The LPRTs have very fast response rates during thermal processing and are often chosen for rapid thermal processing (RTP) tools in semiconductor manufacturing.

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Table II. Standard uncertainties $u(k = 1)$ for R versus t measurements, at 600 °C.

Pt/Pd thermocouple	0.3 °C
Four point resistance reading	0.1 °C
Voltmeter reading	0.3 °C
Thermal uniformity of resistor	0.3 °C
Route mean square value	0.6 °C

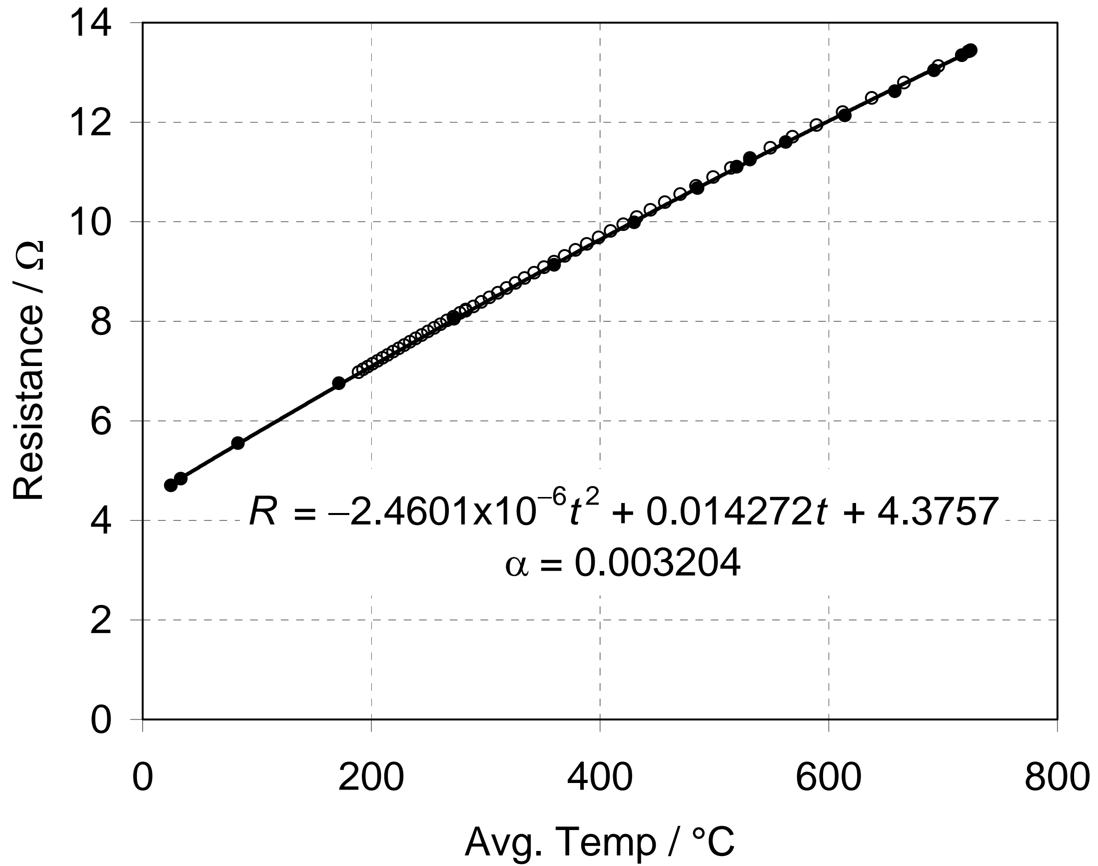

Figure 1. Resistance versus temperature for Pt thin film on Si wafer. Measurements made at 5 minute intervals for both heating (solid dots) and cooling (circles). Pt = 0.77 μm thick, 900 °C anneal, tested in N_2 atmosphere.

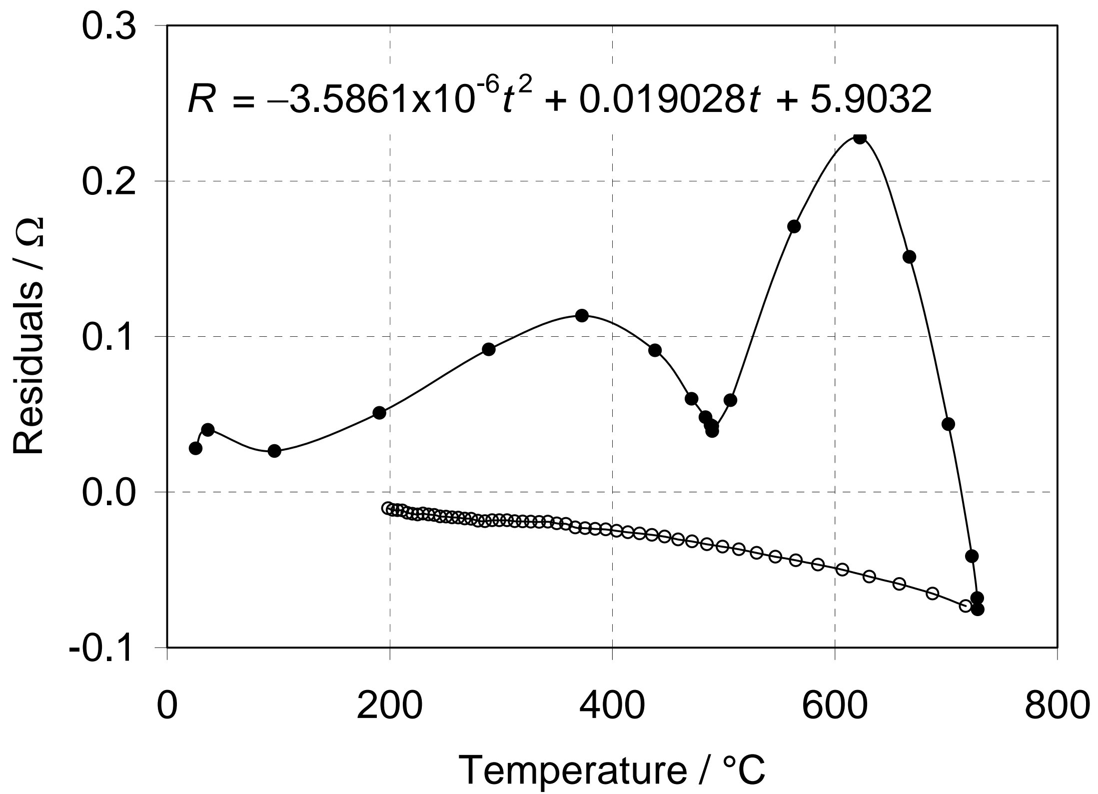

Figure 2. The deviation of the data from Pt81 from its equation on heating (solid dots) and cooling (circles) in ohms. 0.1 Ω at 0 °C is equal to 5 °C.

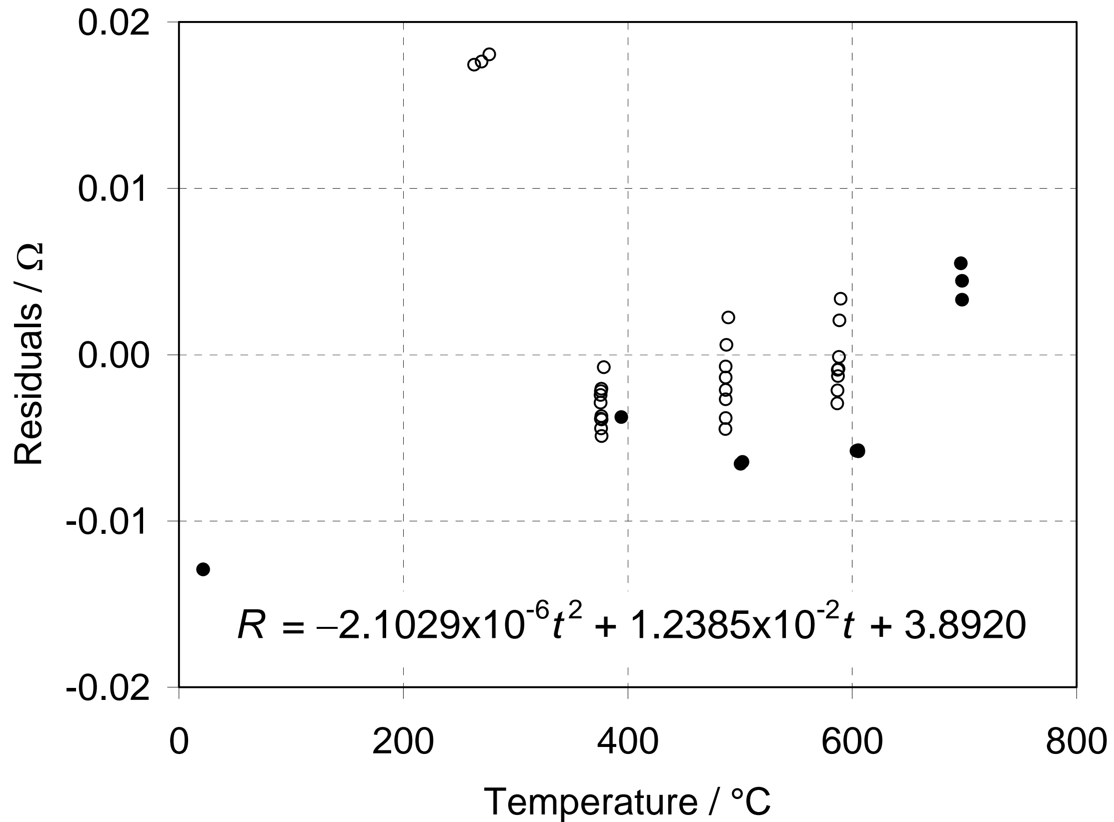

Figure 3. The residuals or deviation from the equation for sample Pt69 resistance versus temperature on heating (solid dots) and cooling (circles). Data points were recorded only for the thermal arrests and not during rapid temperature changes. 0.01 Ω is equivalent to 0.8 °C at 0 °C.

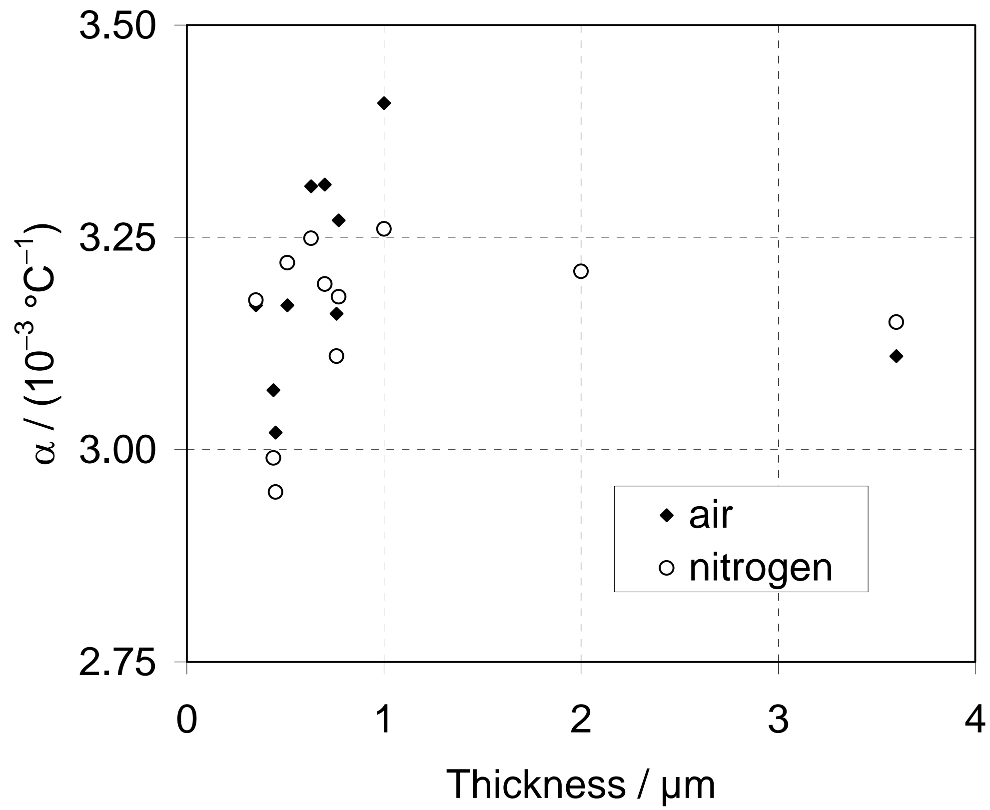

Figure 4. Thermal coefficient of resistivity, α, versus thickness of sputtered Pt film on Si. α is higher in air and with thicker Pt film.

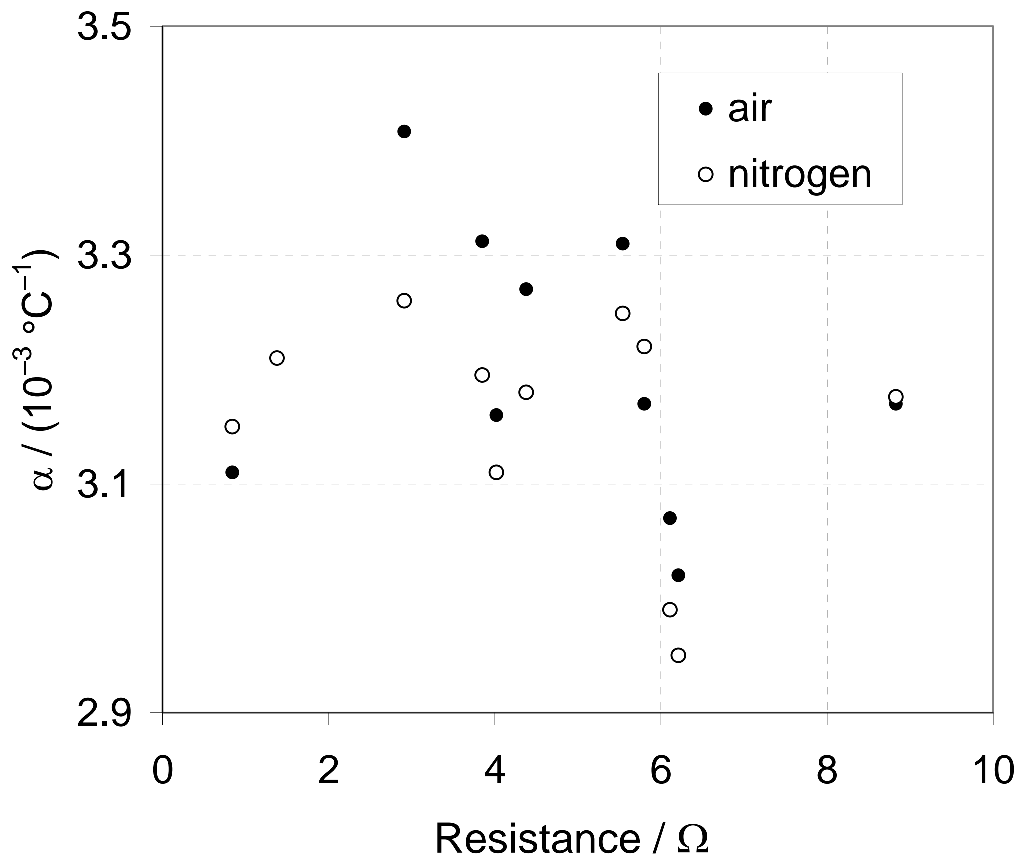

Figure 5. Thermal coefficient of resistance, α, versus resistance of Pt film on Si. α is higher when measured in air and with less resistance of film.

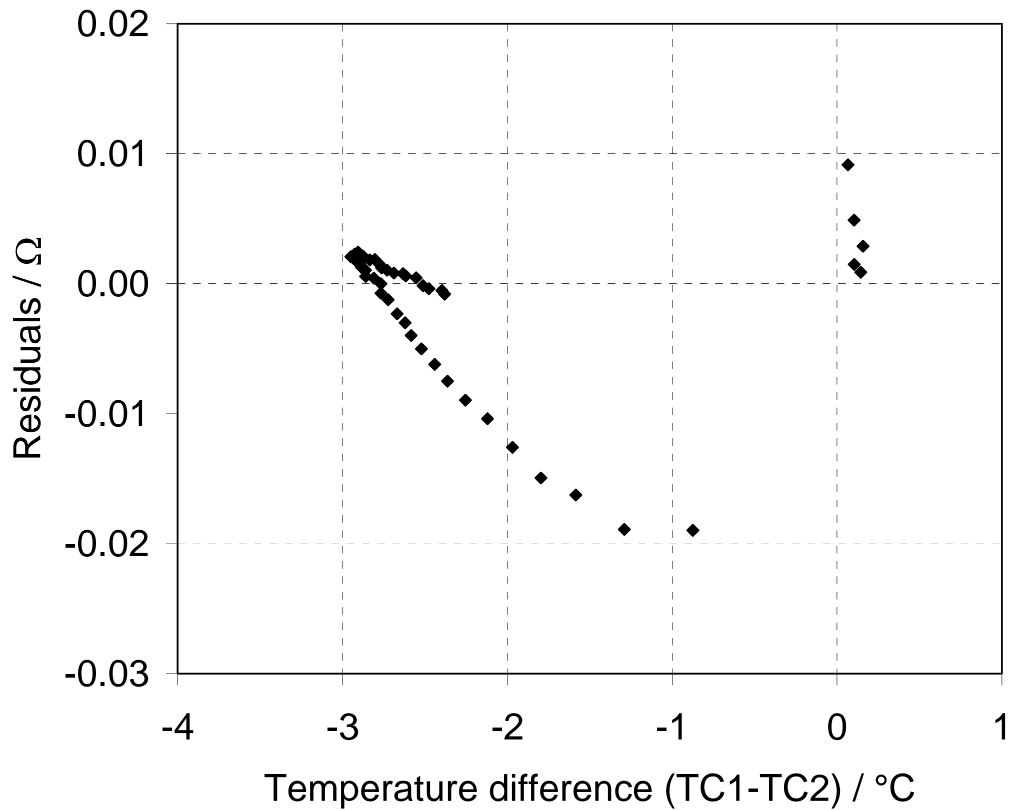

Figure 6. The effect of temperature differentials along the length of the Pt resistor during test on the residual misfit to the equation for the resistance of the Pt film as a function of its temperature. Positive residuals are for temperatures near 600 °C. The values for cooling start near –1 °C at 587 °C, drop to near –3 °C at 302 °C, and then rise to –2.4 °C at 220 °C. Thermal gradients along the thin-film resistor do not correlate with residuals from the fitted equation.

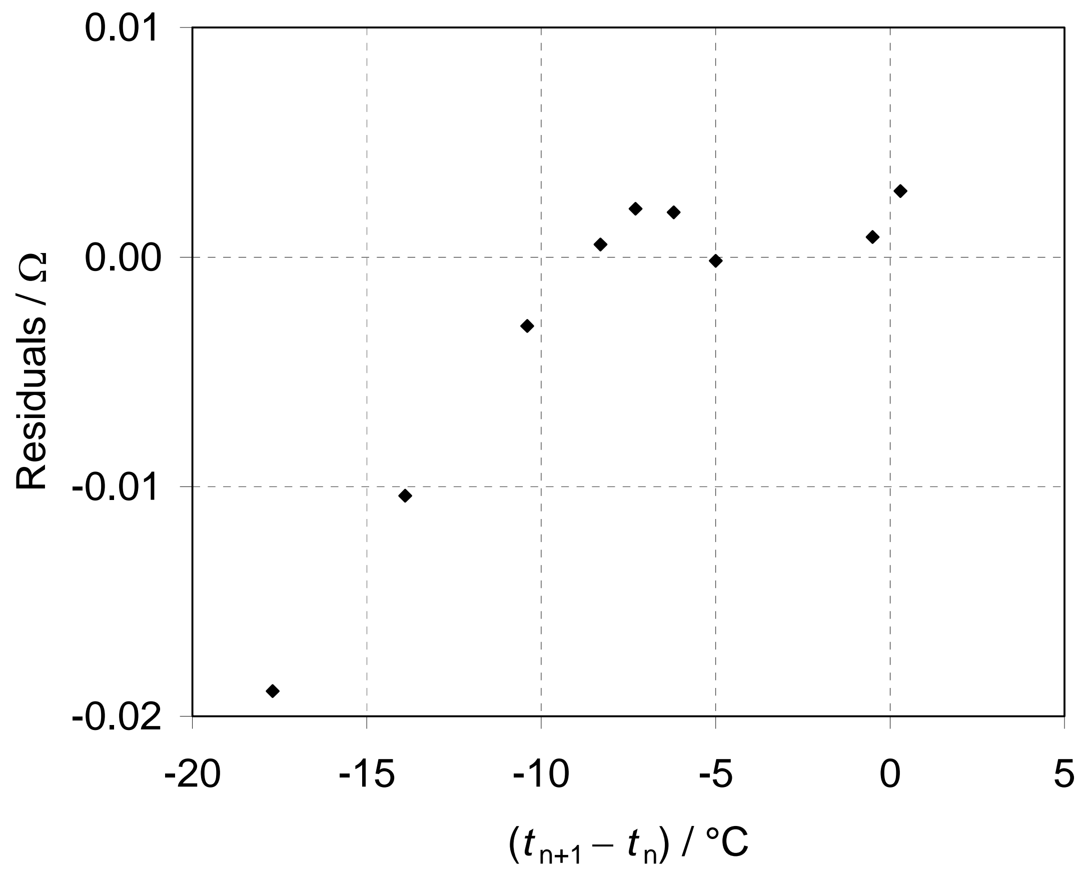

Figure 7. Residuals versus the rate of cooling of the thin film. Faster cooling leads to higher deviation (residuals) from the equation for the coefficient of resistance.

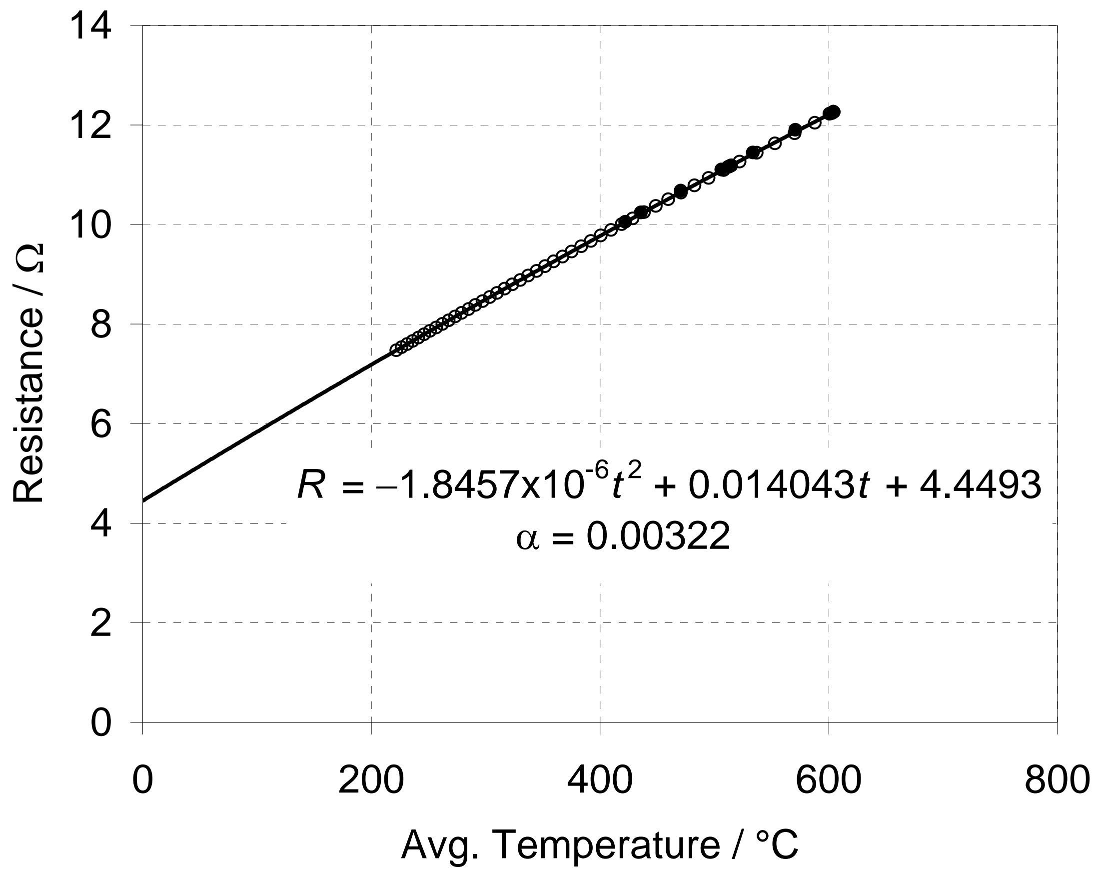

Figure 8. Resistance versus test temperature for heating (solid dots) and cooling (circles) of the Pt85 thin film, 0.77 μm thick, tested in air, $\alpha = 0.00322$ °C^{-1}

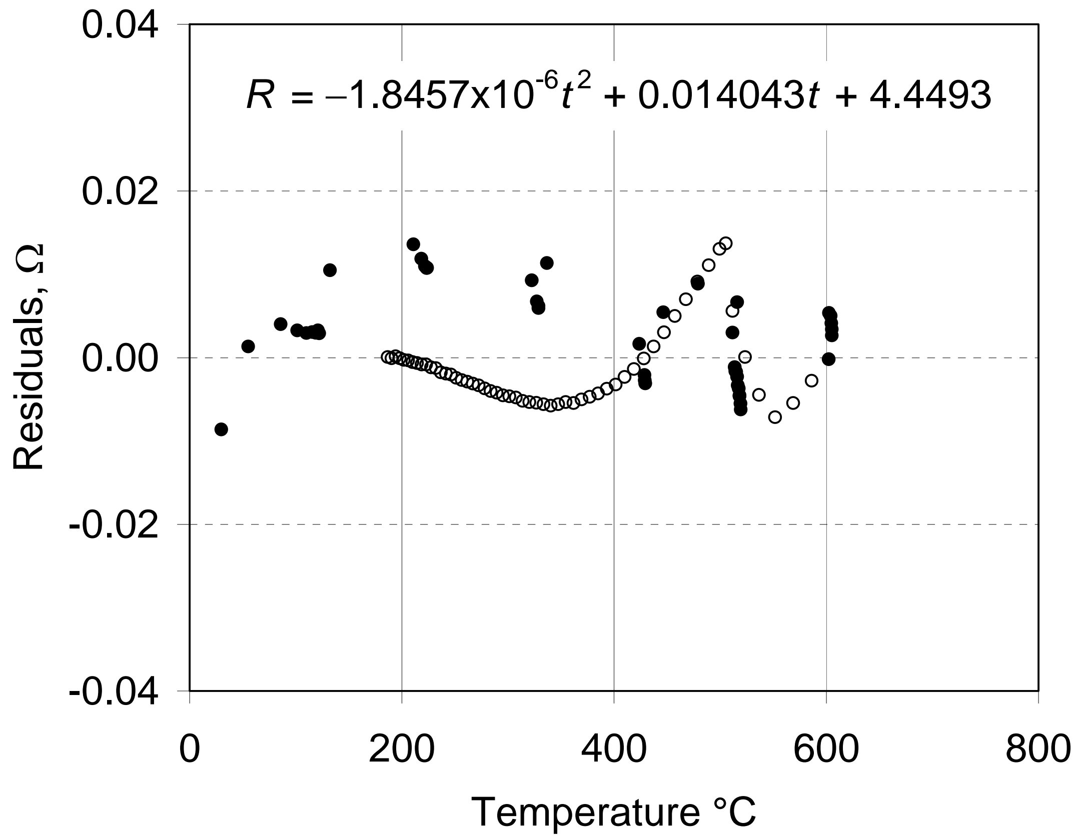

Figure 9. The residuals from the plot in Fig. 8 between the resistance measurements and the fitted quadratic formula in the figure for temperature rates of change less than 2 °C per minute. Almost all measurements (solid dots for heating, circles for cooling) lie less than 1 °C (0.0143 Ω) from the curve.