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Silicon Crystal Heating and Thermocouple Mounting Designs

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New mounting methods for a silicon crystal and a thermocouple are described. The sample mounted using this design exhibited a very uniform temperature distribution when resistively heated. Deviations between the silicon temperature and the mounted thermocouple temperature have been measured. Metal contamination on the surface of the sample mounted with a thermocouple by the new method was found to be below the detection limit of Auger electron spectroscopy. In addition, the emissivity data at 0.65 μm for silicon as a function of temperature has been fitted to a useful equation.

I. INTRODUCTION

Many methods have been used to connect a silicon sample to metal electrodes in order to provide uniform electrical and thermal contact during resistive heating in ultrahigh vacuum (UHV). A design which presses the sample to the electrodes by using metal springs[1,2] is often adopted. Another solution is to attach the sample to elastic metal foil which is connected to the electrodes either by welding or by tension.[3-5] The electrical contact between the sample and the electrode is not always uniform in these designs causing non-uniform sample heating. This is prominent at the beginning stage of heating, where the power delivered to the crystal is limited by its high resistance. Once the current starts to flow in some part of the sample, the temperature at that part increases, and consequently its resistance drops down further, increasing the current flow through that part. If the current flow is concentrated in a small area of the contact, the temperature in this area increases significantly due to the high current density. This may induce surface roughening and haziness or even melting of the sample as well silicide formation between the sample and the metal contact.

The attachment of a thermocouple to silicon presents a second problem, since contamination of the silicon by metal may result. A sample in the vicinity of a hot Chromel-Alumel thermocouple (nickel-chromium vs. nickel-aluminum) is easily contaminated by gas phase transport of nickel when it is heated at high temperature, because the vapor pressure of nickel is very high (10^{-5} - 10^{-4} Torr at

1450 - 1500K). It has been shown that only several atomic percent of nickel destroys the (2x1) structure on the Si(001) surface to form defect sites termed "split off dimers".[6] To inhibit this contamination, it is essential to use a thermocouple with a low vapor pressure. It is also important to prevent direct contact between the thermocouple and the sample, because most commercial thermocouples include materials which form silicides with silicon at high temperature.

An adhesive such as a high temperature ceramic cement is often employed to attach a thermocouple onto a sample indirectly. But even such an adhesive may contaminate silicon when it is heated at very high temperature. For example, we found that a zirconia base cement Ultratem 516 (Aremco Products, Inc.), which is a common adhesive used with silicon, was stable below 1100K, but Zr diffused easily through silicon upon annealing to 1400K .

Ho et al.[2] proposed a thermocouple which was welded to a tantalum tab; the tab was strongly bonded to a silicon sample by inserting a small germanium grain followed by heating to form a Si-Ge alloy. The adhesion was unfortunately lost when the sample was heated at high temperature.[8]

Bozack et al.[5] suggested a method to attach a thermocouple to a silicon sample without using an adhesive. They made slots along the edges of a silicon crystal by sawing. Then a thermocouple was welded to a tantalum tab and the tab was bent into an envelope and wedged into the slot. The bent tab was held in place by tension. As tantalum is relatively inert to silicon, the sample suffers little contamination by this attachment method. The problem in this method is that contamination may be introduced to the sample during slot manufacturing. After using the RCA cleaning procedure to remove metals, and heating the slotted silicon crystal in UHV at high temperature, nickel was sometimes detected on its surface in our experiments. The origin of the nickel is not known but it might come from the diamond impregnated stainless steel blade used to make the slots.

In this paper we suggest a new design for silicon crystal mounting as well as for thermocouple mounting. The results of sample heating and temperature measurement using the new design are shown and compared with those of the slotted sample design.

For optical pyrometry of silicon, two commonly used data sets[9,10] of emissivity at 0.65 μm are combined and a mathematical fit of the combined emissivity data from 540K - 1700K is presented.

II. Crystal mounting design and performance

Our sample mounting design, which is similar to that proposed by Olshanetsky[11], is shown in Figure 1. Part of a tantalum sheet (20 x 9 x 0.125 mm^3; purity 99.9%, Goodfellow) is bent to form a bracket shape (Figures 1(a),(b)) while the remainder is curved and spot-welded to a tungsten rod (diameter 2.0mm; 99.95%, Goodfellow) (Figure 1(c)). The rods receive electrical power through copper leads. Four spacers made from a silicon wafer (4.5 x 8.5 x 0.4 mm^3; Virginia Semiconductor) are prepared and used to sandwich a silicon slice (23 x 8 x 0.4 mm^3, p type, 10Ω·cm; floating zone grown, Virginia Semiconductor) (Figure 1(e)). This sandwich structure is fixed to the tantalum brackets by wedging a U-shaped spring made from a tantalum sheet (8 x 8.5 x 0.025 mm^3, 99.9%, Goodfellow) into the slot between the top of the bracket and the top silicon spacer (Figure 1(f),(g)).

The sample mounted by this method was resistively heated in ultrahigh vacuum and its temperature was measured by an optical pyrometer (The Pyrometer Instrument Co., Inc., Pyro Micro-Optical Model, wavelength 0.65μm). The temperature uniformity along the sample was typically ±15K at 1400K. This uniform heating was found to be reproducible during multiple annealing steps with this sample, and also in the heating of different samples mounted using the same design.

To compare with previous methods, a slotted silicon crystal was prepared following the procedure of Bozack et al.[5] and John et al.[12] Three sides of a single silicon crystal (5 x 10 x 1.5 mm^3, Virginia Semiconductor) were sawed to make 0.25-mm-wide slots, and 0.25-mm-thick tantalum sheets were pressed into two opposite slots. The sheets were then spot welded to the tantalum power leads. The third slot was used for measuring sample temperature by using an embedded thermocouple, which is depicted in a later section of this paper. The temperature uniformity of the resistively heated slotted crystal was found to be different from time to time, and from sample to sample. Furthermore, hot spots were often observed at the contacts between the sample and the tantalum sheets.

The uniform heating of the sample in the new design originates from the use of the silicon spacers. Even if hot spots are formed at the spacer/spring or the spacer/bracket interface, the non-uniformity of electron flow is smoothed in the silicon spacer and its distribution becomes more uniform at the spacer/sample contact. The heating uniformity can be also attributed to the stable force supplied by the U-shaped tantalum spring employed, and the good electrical and thermal contact between the Si spacers and the Si sample.

III. Thermocouple mounting and performance

Figure 2 shows the new mounting method for a thermocouple on a silicon sample. The thermocouple junction is made by welding a tungsten - 5% rhenium wire and a tungsten - 26% rhenium wire (diameter 0.08mm; Omega Engineering Inc.) . To prevent direct contact between the thermocouple and the sample, the thermocouple junction is inserted into a tantalum tube (tube inner diameter 0.19mm, wall thickness 0.06mm, length 5mm; tantalum purity 99.9%, Goodfellow) (Figure 2(a)) and the tube is squeezed and flattened to fix the thermocouple to it (Figure 2(b)). Then the tube is bent to form a rectangular bracket shape, and it is attached to the sample like a U-clip (Figure 2(c)). In this mounting method, it is important to cut one of the tube ends obliquely (Figure 2(b)) so as to protect the sample surface from touching the thermocouple as it exits the Ta tube (Figure 2(c)).

A cold junction compensator (model MCJ-C, Omega company) is used for the reference junction at 273K. The conversion from the thermocouple signal to the temperature is done in a computer-controlled acquisition board (model Lab-PC+, AT-M10-64E-3, National Instrument Company) using a calibration equation.[13] The true sample temperature is determined by using an optical pyrometer working at 0.65μm. The method employed to determine the silicon temperature using this pyrometer is described in the Appendix.

To compare the degree of the agreement between the true sample temperature and two different thermocouple attachment methods, a chromel vs. alumel thermocouple was welded to a tantalum sheet and the sheet was bent and wedged into the third slot of the slotted crystal, following the procedure of Bozack et al.[5]

Figure 3 shows the deviation of the thermocouple readings for the two thermocouple mounting methods from the true sample temperature measured with the pyrometer. The discrepancy becomes larger at higher temperature for both thermocouples. But the deviation is smaller for the thermocouple in the new U-clip design compared to that for the slotted sample. The problem of the new thermocouple mounting method is that the thermocouple signal fluctuates somewhat in time at high sample temperature, typically above 1100K (± 10K), probably due to thermal expansion effects in the U-shape clip.

It is important to note the surface cleanness of the sample mounted with a thermocouple by our new technique. When the mounting parts for the sample and those for the thermocouple were prepared, we were very careful about not contaminating them with metal from tools. We cleaned all parts by boiling in concentrated HCl solution, which effectively removes metal contamination such as nickel.[6] The sample mounted by this method showed no metal contamination by AES even after multiple annealing (detection limit is about 0.1 percent atomic concentration in the depth of sampling).

IV. Summary

New sample and thermocouple mounting methods for silicon single crystals are proposed. The sample mounted with the new design was found to heat very uniformly. The readings of the thermocouple attached to the sample by our new method are slightly lower compared to those of optical pyrometer. The methods developed here avoid metal contamination of Si.

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V. Appendix

Measurement of Silicon Temperature using an Optical Pyrometer

The temperature of a hot object can be estimated by measuring its radiant power in a given solid angle at a specific wavelength λ. When an object is a perfect blackbody, its radiant power per unit area, $W_b(\lambda,T)$, is described by Planck's law:

$$W_b(\lambda,T)=\frac{2\pi hc^2}{\lambda^5}\frac{1}{\left[\exp\left(\frac{hc}{\lambda kT}\right)-1\right]} \tag{1}$$

where λ is the wavelength, T is a temperature, c is the speed of light, k is Boltzmann's constant, and h is Planck's constant. When $hc/\lambda kT >> 1$, this equation can be simplified:

$$W_b(\lambda,T)\cong\frac{2\pi hc^2}{\lambda^5}\exp\left(-\frac{hc}{\lambda kT}\right). \tag{2}$$

The radiant power from a non-blackbody object is smaller than that from a blackbody. The ratio of the radiant power of the non-blackbody, $W_n(\lambda,T)$ to that of a blackbody, $W_b(\lambda,T)$, is termed the emissivity, $\varepsilon(\lambda,T)$:

$$W_n(\lambda,T) = W_b(\lambda,T) \cdot \varepsilon(\lambda,T). \tag{3}$$

For a pyrometer of the disappearing filament type, the brightness of a hot tungsten filament at a particular wavelength (which is calibrated to correspond to that of blackbody) is adjusted to that of the hot sample by observing the superposition of the filament on the sample through a filter and changing the filament current until the filament becomes invisible against the sample image. The temperature of the hot filament can be determined from its calibration by measuring the current flowing through it. When the brightness of the tungsten filament and that of the non-blackbody sample are same, their radiant power is also equal:

$$W_b(\lambda,T_{pyro}) = W_n(\lambda,T_{sample}) = W_b(\lambda,T_{sample}) \cdot \varepsilon(\lambda,T_{sample}), \tag{4}$$

where $W_b(\lambda,T_{pyro})$ and $W_n(\lambda,T_{sample})$ are the radiant powers for the filament in the pyrometer and for the sample. Their temperatures are T_{pyro} and T_{sample}, λ is the wavelength of the light that the filter transmits, and $\varepsilon(\lambda, T_{sample})$ is the emissivity of the sample.

When the sample brightness is measured through a window, the brightness reduction caused by the reflection of the window surfaces must be taken into account. In this case, an extra factor $(1-R)^2$ should be included:

$$W_b(\lambda, T_{pyro}) = W_b(\lambda, T_{sample}) \cdot \varepsilon(\lambda, T_{sample}) \cdot (1-R)^2, \tag{5}$$

where R is the reflectivity of the window material. R can be estimated by:

$$R \cong (n-1)^2/(n+1)^2, \tag{6}$$

where n is the refractive index of the window material at the wavelength. For example, $R \cong 0.035$ for a glass window at room temperature with $n = 1.46$ for type 7056 glass at 0.65μm. The correction does not consider either absorption by the window material or by inadvertent thin film deposits on the window.

Using equations (2) and (5) and solving it for T_{pyro}, one obtains:

$$T_{pyro} = T_{sample}/\{1 - \lambda k T_{sample} \ln[\varepsilon(\lambda, T_{sample}) \cdot (1-R)^2]/hc\}. \tag{7}$$

The emissivity for silicon at 0.65μm was measured by Allen[9] and Sato.[10] Equation (8) below fits the combined emissivity data in the temperature range of 540K - 1700K:

$$\varepsilon(0.65\mu m, T) = 0.4826 - 2.09 \cdot 10^{-5}\, T + \frac{0.1583}{1 + \exp\left[{(T-1410)}/{112.9} \right]}. \qquad (8)$$

The experimental data and this fit are shown in Figure 4, and the maximum deviation between the experimental emissivity points and the empirical curve is ≤ 0.004.

The deviation between the true silicon temperature $T_{silicon}$ and the temperature measured with a pyrometer, T_{pyro}, through a type 7056 glass window at 0.65μm can be calculated using equations (7) and (8). The results are shown in Figure 5.

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[13] $T = 286.88 + 62.715\times V - 0.97944\times V^2 + 0.02732\times V^3 - 13.433\times \exp(-1.2334\times V)$, where T is the temperature in Kelvin and V is the thermoelectric voltage of the W26%Re/W5%Re thermocouple in mV. The deviations of this fitting from the real temperature data are slightly less than those of the polynomial equation fitting proposed by V. S. Smentkowski and J. T. Yates, Jr. (J. Vac. Sci. Technol. A 14, 260 (1996).

Figure Captions

Figure 1. Schematic diagram of mounting design for heating a silicon sample.

Figure 2. Design of thermocouple mounting on a silicon sample.

Figure 3. Deviation of temperature measured by pyrometer from that measured by thermocouple mounted in the slotted silicon sample design and in the new design.

Figure 4. Experimental silicon emissivity and its fit to an equation.

Figure 5. Deviation between measured temperature by pyrometer and the true temperature, corrected for emissivity and type 7056 window reflectivity.

Mounting Design for a Silicon Sample

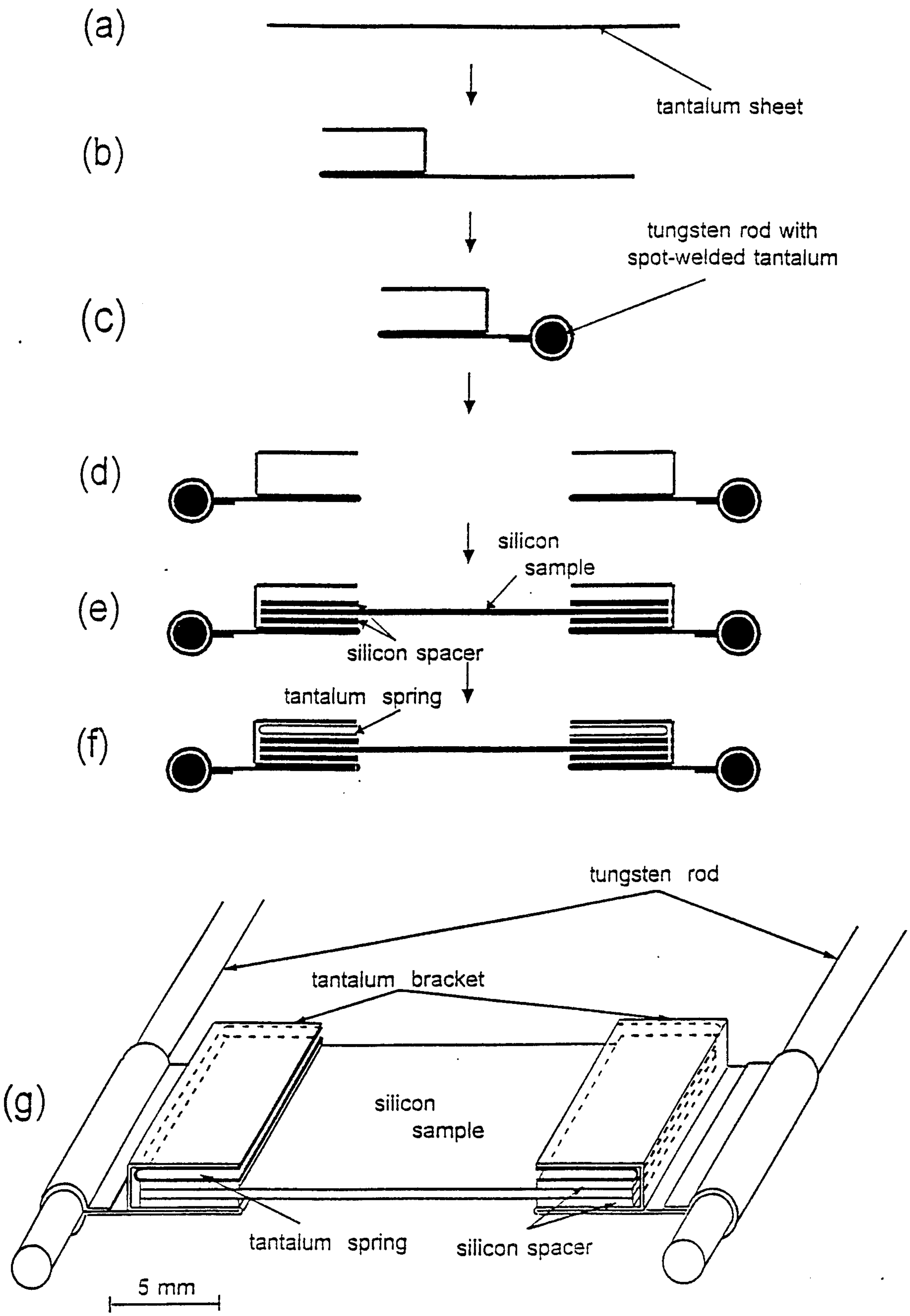

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Figure 1

Thermocouple Mounting Design for a Silicon Sample

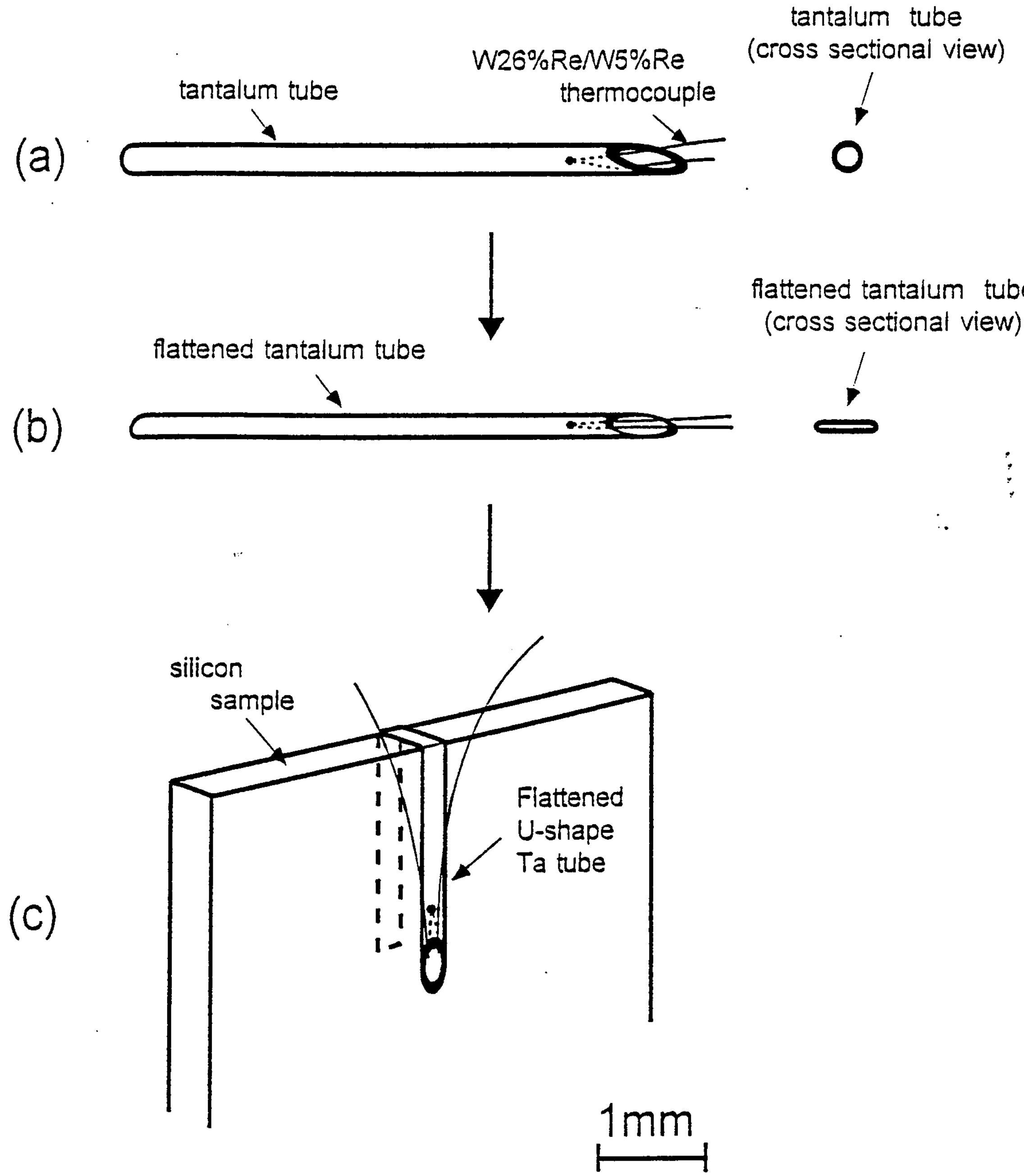

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Figure 2

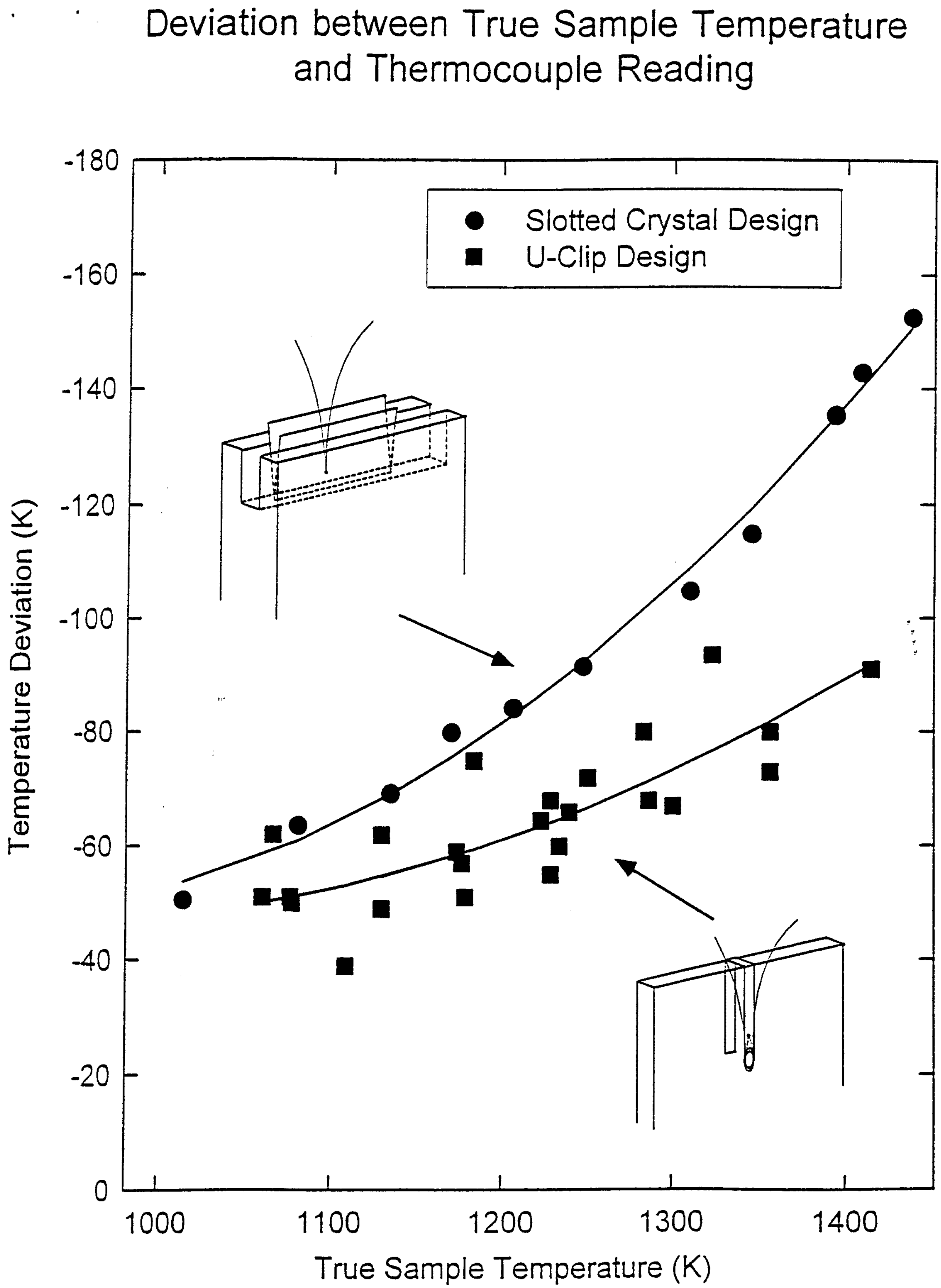

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Figure 3

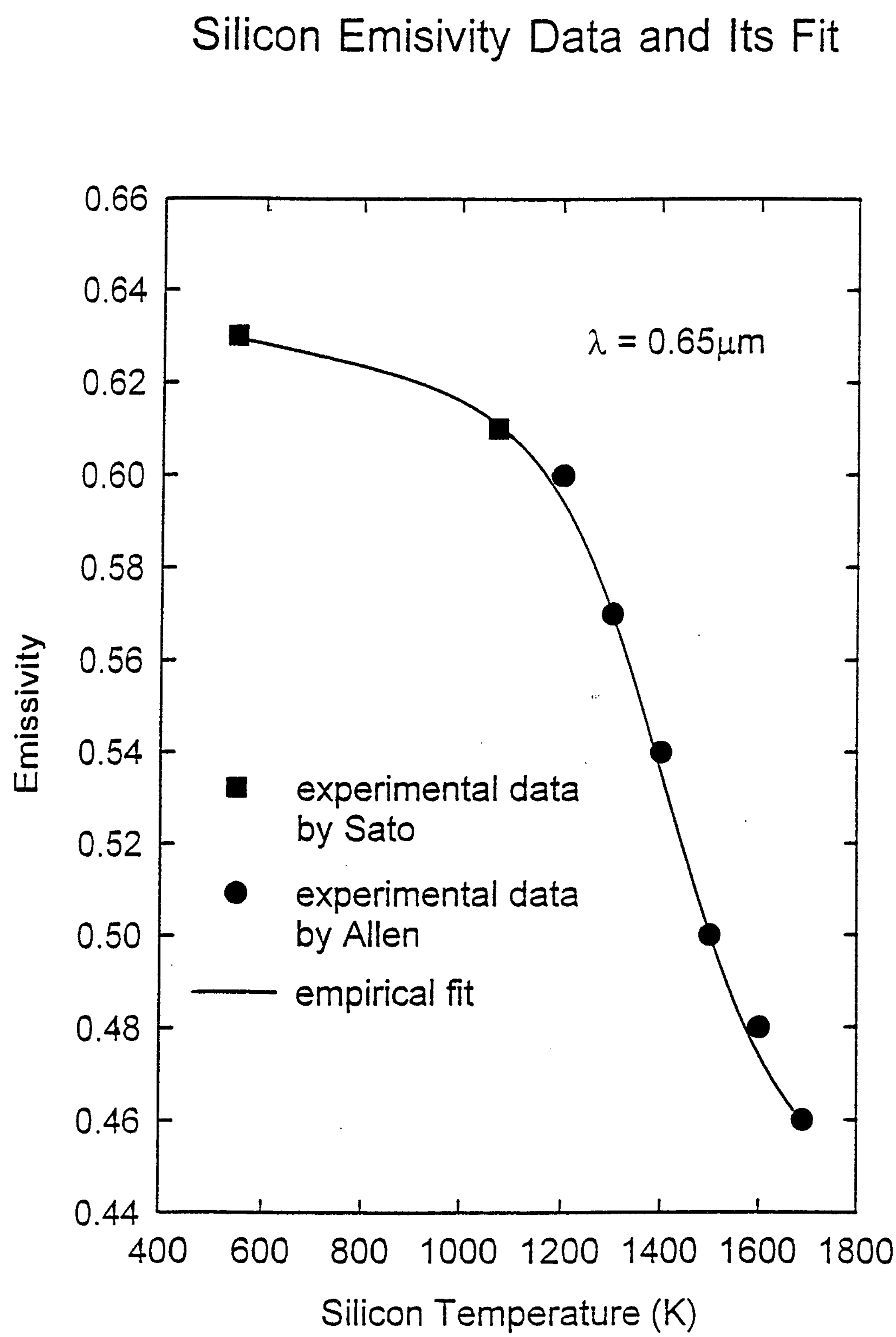

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Figure 4

Deviation between True Silicon Temperature and Temperature Measured by Pyrometer

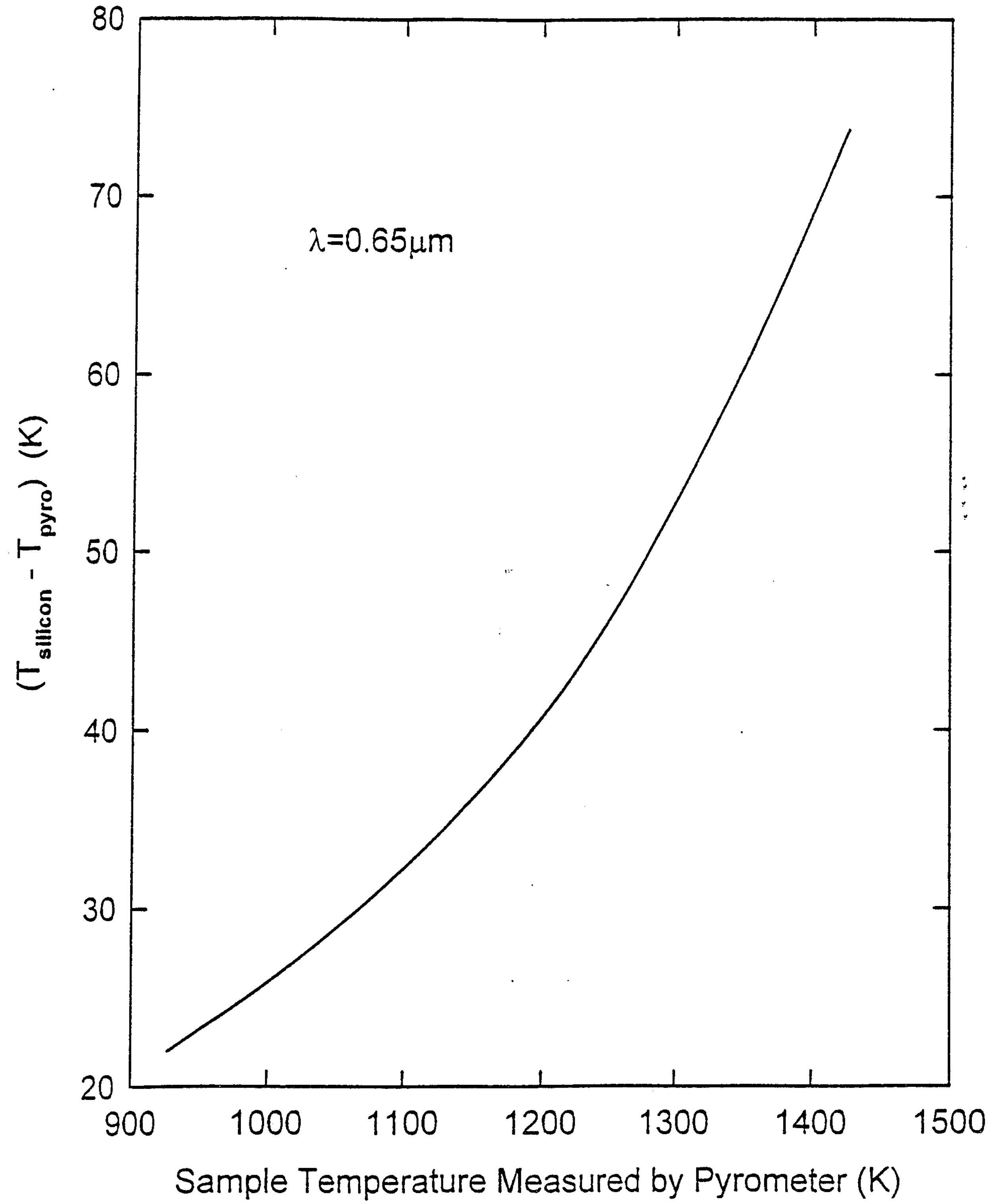

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Figure 5

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