In situ sample temperature measurement in plasma immersion ion implantation

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Plasma immersion ion implantation (PIII) is an excellent surface modification technique because it is not restricted by the line-of-sight limitation that plagues conventional beamline ion implantation. However, the lack of *in situ* monitoring has hampered wider acceptance of the technique in industry. It is known that the implantation temperature has a large influence on the surface properties of the treated specimens in addition to the more obvious parameters such as implantation voltage, pulse duration, pulsing frequency, and so on. Direct measurement of the target temperature is complicated by the sample high voltage as well as by interference from the electromagnetic field and plasma. In this article, we present a novel interference-free, *in situ* temperature measurement technique employing a shielded thermocouple directly attached to the sample stage. Our experiments show that the setup can monitor the target temperature in real time, even under severe arcing conditions. Our results also indicate that in a hot filament glow discharge radiation heating is quite small, and sample heating is primarily caused by ion bombardment during the PIII cycles. The new design will open up other possibilities such as *in situ* dose monitoring if, for example, the thermocouple is replaced by a Faraday cup. © *1999 American Institute of Physics.* [S0034-6748(99)03606-0]

I. INTRODUCTION

In recent years, a myriad of studies has proven that plasma immersion ion implantation (PIII) is a suitable method for surface modification of materials.[1–4] One of the major advantages of PIII is its ability to treat nonplanar samples with an acceptable dose uniformity. In order to achieve a thicker modified layer and optimal phase structure, elevated temperature PIII is widely used.[5–7] The conclusion is that the process temperature is crucial to the final surface properties. For instance, in improving the tribological properties of stainless steel, the implantation temperature must be controlled so that the wear resistance is improved without sacrificing the corrosion resistance. Performing cross-sectional transmission electron microscopy (XTEM) on the samples treated at 150–520 °C, Li *et al.*[8] have concluded that the treatment temperature has a strong influence on the evolution of the microstructure. Blawert *et al.*[9] found that the surface properties were very sensitive to the temperature between 400 and 500 °C. Up to 400 °C, an expanded austenite, a favorable phase, was formed on AISI318. However, at 500 °C, a surface layer consisting of CrN was formed and it adversely affected the corrosion resistance. The same is true for semiconductor applications such as the synthesis of separation by plasma implantation of oxygen (SPIMOX) which calls for an implantation temperature of 600 °C or higher to enable proper oxide nucleation[10–15] and a PIII/ion cut that requires an implantation temperature of 200 °C or lower.[16–20]

In situ temperature monitoring and control are thus extremely important. However, the PIII environment (high voltage, vacuum, interference, etc.) renders common temperature measurement techniques impractical. The sample is immersed into the plasma and biased to a negative voltage ranging from 5 to 100 keV. To make things worse, there is frequent electrical arcing in a PIII experiment due to the high voltage operation, and the high electric and magnetic fields can damage the measurement hardware. Moreover, the sample stage is usually located at the center of the vacuum chamber thereby making direct contact more complicated. Hence, real-time *in situ* temperature measurement in PIII has seldom been reported in the literature in spite of its importance. Collins *et al.*[21] used an infrared pyrometer in the range of 2.0–2.6 μm to view a point on the target in the plasma through a quartz window. The pyrometer was calibrated by

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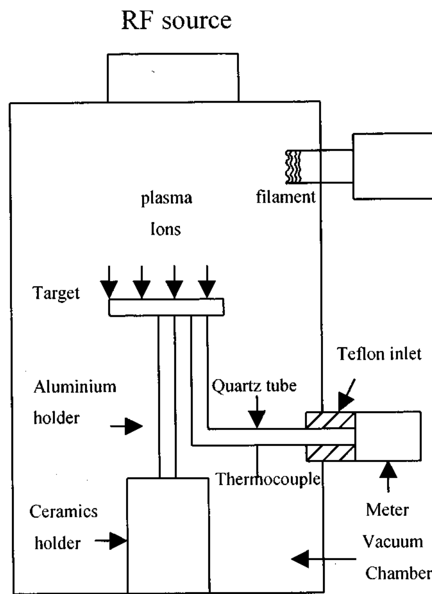

FIG. 1. Schematic of the temperature measurement system using a thermocouple in a plasma immersion ion implanter. Four sets of filaments are installed symmetrically around the chamber wall.

separately heating the target with thermocouple inserts. This method is very effective and the temperature can be maintained to within ±1 °C by regulating the duty cycle of the high voltage pulse. The temperature of the target was measured by Blanchard[22] using a Luxtron that is a phosphorescent probe. In both of these setups, the temperature sensor is far away from the biased target. Even though it is safer and easier to engineer, both methods are indirect.

II. TEMPERATURE MEASUREMENT HARDWARE

We used a direct *in situ* temperature measurement technique employing a commercial thermocouple attached directly to the sample stage shown schematically in Fig. 1. The *K*-type thermocouple is inserted into the target from below, and the thermocouple wire is insulated from the plasma and the chamber wall through a quartz glass tube 20 mm in diameter and a Teflon inlet at the chamber wall. The display meter is floated electrically together with the battery power supply. The glass tube including the thermocouple wire is installed very close to the target holder, so that there is negligible influence on the plasma sheath. It is necessary to keep the temperature of the cold end of the thermocouple constant in order that the thermal potential is proportional to the measured temperature. We use a long thermocouple to connect the display meter, which is outside the vacuum chamber, through two stainless steel rods of the same length and diameter. F-rubber rings are used to seal the rod to the chamber. In this way, the temperature of the cold end is not affected by the hot target or hot filaments. Meanwhile our cleanroom has a constant ambient temperature of 23 °C.

Strictly speaking, our method has three advantages over the traditional use of a pyrometer.

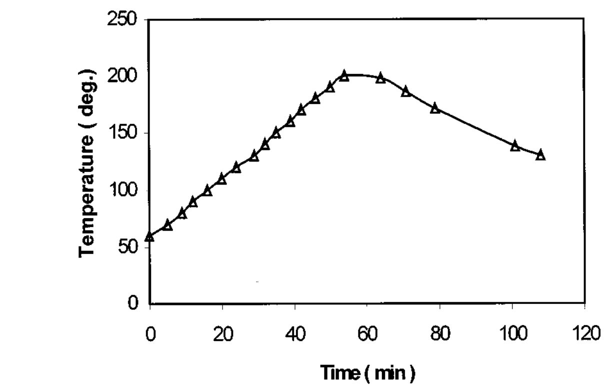

FIG. 2. Time-dependent target temperature during high voltage PIII. The experimental conditions are implantation voltage = −20 kV, pulse duration = 20 μs, and pulsing frequency = 120 Hz. The initial measurement starts at 60 °C and implantation stops after 54 min. The plasma is sustained by hot filaments and the chamber wall is water cooled.

(1) Pyrometers are calibrated for a certain temperature range, but our thermocouple approach can detect a wide range of temperatures.
(2) Pyrometers are usually set up outside of the vacuum chamber to aim at a certain point on the specimen. Our method is more flexible since the pyrometer can be placed at various locations on the sample stage and even on samples possessing an irregular shape.
(3) Our hardware is less costly.

III. RESULTS AND DISCUSSION

Figure 2 shows the measured temperature with time obtained from our PIII equipment.[23,24] The conditions are 20 kV implantation voltage, 20 μs pulse duration, and 120 Hz pulsing frequency. Figure 3 depicts the temperature changes during a high frequency, low voltage PIII process[25] using 2 kV implantation voltage, pulse duration of 20 μs, and frequency of 8 kHz. The plasma produced in those two experiments is activated by four sets of hot filaments installed symmetrically. That the initial temperature is not ambient temperature is due to pre-cleaning of the workpieces by Ar

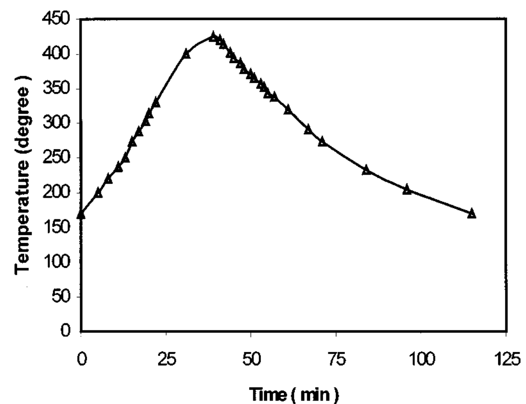

FIG. 3. Time-dependent target temperature during high frequency, low voltage PIII. The experimental conditions are implantation voltage = −2 kV, pulse duration = 20 μs, and pulsing frequency = 8 kHz. The initial measurement starts at 170 °C and implantation stops after 39 min. The plasma is sustained by hot filaments and the chamber wall is water cooled.

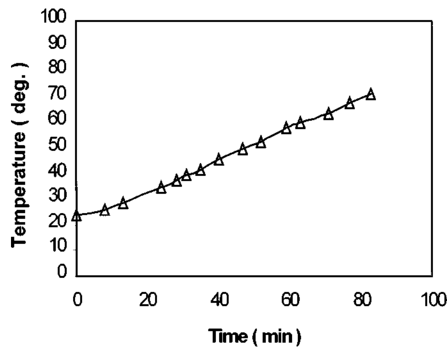

FIG. 4. Time-dependent temperature of the target when the hot filaments are on but without implantation. There are four sets of filaments consisting of 16 wires. The electrical parameters are 26 V and 58 A.

ion sputtering. Our results unambiguously indicate that the target temperature rises linearly with the implantation time, similar to the experimental results of Wei *et al.*[26] We have also found that our setup functions reliably and safely for a prolonged period of time. Careful insulation enables the hardware to work even when sparks and arcing occur during the PIII process. It means the whole measurement system is not affected by the high voltage, high frequency, electrical and magnetic field change, and so on during implantation, although there is direct contact between the target and the thermocouple.

The sample temperature is affected by four factors: (1) heating by the implanted ions, P_{ion}, (2) hot filament radiation heating during a glow discharge, P_{filament}, (3) radiation loss to the wall, P_{wall}, and (4) heat conduction loss through the target holder, P_{con}. Thus, the net heating power $P = P_{\mathrm{ion}} + P_{\mathrm{filament}} - P_{\mathrm{wall}} - P_{\mathrm{con}}$, and the sample temperature rise can be described by the following equation:

$$\begin{aligned}\Delta T &= (\Delta t/mc)(P_{\mathrm{ion}} + P_{\mathrm{filament}} - P_{\mathrm{wall}} - P_{\mathrm{con}}) \\ &= (\Delta t/mc)[P_{\mathrm{ion}} + \alpha(T_f^4 - T_t^4) \\ &\quad - \beta(T_t^4 - T_w^4) - H(T_t - T_h)], \end{aligned} \tag{1}$$

where m is the mass of the sample target, c is the specific heat of target material, T_f is the temperature of the hot filament, T_t is the temperature of the target, T_w is the temperature of the chamber wall cooled by flowing water, T_h is the temperature of the target holder, H is the heat conduction from the target to the holder, $\alpha = \sigma A \epsilon_f \epsilon_t / [\epsilon_t + \epsilon_f(1-\epsilon_t)]$, and $\beta = \sigma A \epsilon_w \epsilon_t / [\epsilon_w + \epsilon_t(1-\epsilon_w)]$,[27,28] σ is the Stephan–Boltzmann constant, A is the implantation area of the target, ϵ_f is the emissivity of the filaments, ϵ_t is the emissivity of the target, and ϵ_w is the emissivity of the chamber wall.

The power loss by conduction through the aluminum support is negligible since we use a rod of very smaller diameter. Thus, from the linear relationship between the target temperature and the implantation time, it can be inferred that the radiation heat exchange effect contributes very little to the target temperature rise, including that between the hot filaments and the target as well as that from the target to the water-cooled chamber wall. The radiation heating power density is proportional to T^4, and so at a lower temperature, the radiation loss of the target is very small.[21] However, the four hot filament sets in our equipment, consisting of 16 wires, get very hot when they emit electrons to ignite the plasma. To assess the filament radiation heating effect, the target temperature is monitored without implantation. A comparison of the results exhibited in Figs. 3 and 4 indicates that the magnitude of ion heating is nearly 14 times larger than that of the hot filament radiation heating. For example, the temperature increase by radiation heating of the hot filaments after 31 min is only 16 °C. On the other hand, ion implantation heating leads to a temperature rise of 220 °C. This increase has already been compensated for by heat loss to the cooled chamber wall as shown in Fig. 3 (after 39 min). Therefore, sample heating stems mainly from ion bombardment. Without ion bombardment, radiation cooling is evident and the target temperature drops precipitously.

To recapitulate, we have successfully fabricated a device to measure the sample temperature *in situ* in a PIII chamber. It provides a convenient and inexpensive way to monitor the treatment temperature and to optimize PIII processes. Our temperature measurement setup has a high interference resistance and can tolerate sparks and arcing under high voltage conditions. Using the new device, we observe that the sample temperature rises mainly because of ion bombardment during a hot filament glow discharge. The next design will be to incorporate the input/output lines of the thermocouple inside the sample stage extension rod and feedthrough in order to minimize the influence on the plasma sheath. Our design can be extended to monitor other important parameters of a PIII process in real time, for example, ion density or implantation current, if the thermocouple is changed to a Faraday cup.

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