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PLASMA-EDGE DIAGNOSTICS BASED ON Pd-MOS DIODES

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Pd metal-oxide-semiconductor (MOS) devices can be used to detect energetic hydrogen atoms. H isotopes implanted into a Pd-MOS diode quickly diffuse through the Pd layer and are accommodated at available Pd–SiO_2 interface sites, causing an increase in the leakage current through the device. We find that a diode's response to energetic hydrogen is rapid, sensitive, dosimetric, and reproducible. Pd-MOS diodes can be regenerated when saturated with hydrogen by heating to 100–200 ° C for a few minutes. These properties make Pd-MOS diodes useful for plasma-edge diagnosis of hydrogen particle fluence when the energy distribution of the incident hydrogen is known. Pd-MOS diode sensors have been used in the laboratory and in the ZT-40M reversed-field pinch to measure energetic hydrogen fluxes. Their small size allows placement in locations inaccessible to conventional diagnostics and should provide a means for remote monitoring of hydrogen fluxes to plasma-facing surfaces.

1. Introduction

Solid-state hydrogen sensors have developed rapidly over the past fifteen years. In 1975, Lundström fabricated the first Pd metal-oxide-semiconductor (MOS) Si transistor that was reported to detect $H_2(g)$ [1]. During the following years, several groups made hydrogen sensing Pd-gated devices on various substrates [2,3]. In 1981, Ruths and co-workers studied a simple Pd/Si Schottky diode that exhibited the same high sensitivity to hydrogen shown in the earlier transistor devices [4]. As gas sensor technology matured, a shift in emphasis from device development to using the devices as tools in scientific studies occurred. Petersson et al. in 1984 used a Pd-MOS device to study surface reactions on Pd [5]. Recently, it was shown that Pd-MOS Schottky diodes respond to energetic hydrogen and that direct implantation of energetic hydrogen into the Pd layer of these devices bypasses surface processes that limit response time [6]. This finding has led to considering the use of Pd-MOS devices as hydrogen flux monitors in fusion experiments.

A Pd-MOS device placed in line of sight to a plasma can measure hydrogen emanating from the plasma-edge. Their small size and low cost make them well suited for placement at multiple locations inside a fusion experiment for detailed monitoring of hydrogen particle emission. In this paper, we describe laboratory measurements of the device's sensitivity and response to energetic hydrogen and report on tests of a hydrogen particle diagnostic using a Pd-MOS detector that has been placed on the ZT-40M reversed field pinch [7].

2. Pd-MOS diode design and operation

A diagram and circuit of a Pd-MOS Schottky diode for sensing hydrogen are shown in fig. 1. A thin SiO_2 layer separates the Pd metal contact from the n-Si substrate. When reverse biased with 0.5–2 V, a leakage current, I_r, of typically 0.1–200 μA flows through the diode and is easily measured using an ammeter. At room temperature, hydrogen that enters the Pd layer quickly diffuses to the Pd/SiO_2 interface where it tends to lower the metal work function and device barrier height. This causes an increase in the leakage current which, according to a thermionic emission model, is given by

$$I_r = AT^2 \exp[-\phi_b/kT], \qquad (1)$$

where A is a factor determined by the device size and applied bias voltage, T is temperature, ϕ_b is the barrier

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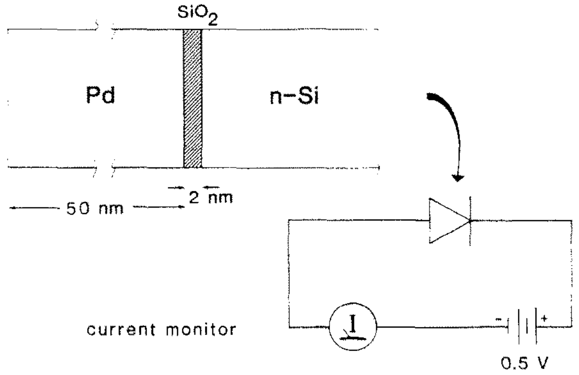

Fig. 1. Schematic of a Pd-MOS diode and and current measuring circuit. Hydrogen that enters the Pd layer and accumulates at the Pd–SiO_2 interface causes a change in the measured leakage current.

height, and k is Boltzmann's constant. The barrier height is approximately given by

$$\phi_b = \phi_m - \chi, \qquad (2)$$

where ϕ_m is the metal work function and χ is the electron affinity of the Si substrate. Because of the exponential relation between I_r and ϕ_b, a small change in the metal work function caused by hydrogen gives a large change in the measured leakage current through the device.

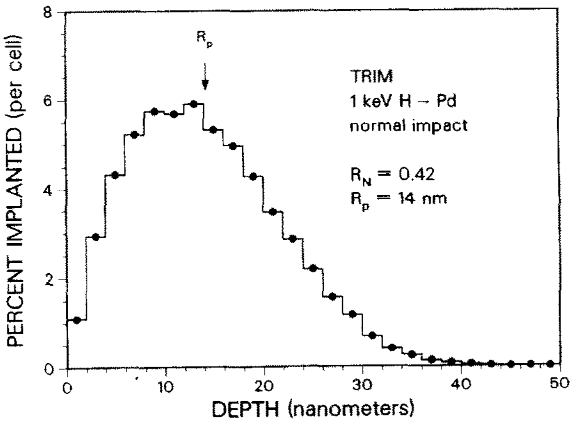

Fig. 2. Calculated range distribution of 1 keV H implanted in Pd at normal incidence. The stopping point of 5×10^4 incident H atoms were tallied into cells 2 nm wide. Resulting values for the projected range, R_p, and the reflection coefficient, R_N, are listed. The calculation was made using the TRIM model (ref. J.P. Biersack and L.G. Haggmark, Nucl. Instr. and Meth. 174 (1980) 257).

The thickness of the Pd layer is not critical except that, for energetic particle detection, it must be greater than the range of the particles striking the detector to prevent damage to the Pd/SiO_2/Si interfaces. Typical plasma-edge particle energies are in the neighborhood of 10–1000 eV, so a Pd layer thickness of 50–100 nm is usually sufficient. For example, 1 keV H at normal incidence has an average projected range in Pd of 14 nm and a distribution extending to less than 50 nm beneath the surface, as shown in fig. 2.

The particular devices used in this study were Pd-SiO_2–Si Schottky diodes having a 50 nm Pd layer and a 2 nm SiO_2 layer on top of a n-Si substrate 2.5 mm square and 1 mm thick. Except as noted, the Pd layer was deposited in a circular area of 2.4 mm diameter centered on the substrate. The devices were prepared at the Sandia Center for Radiation Hardened Microelectronics using conventional microelectronic fabrication techniques [8]. Each diode was mounted in a standard header which provided connection leads and physical protection.

3. Experimental

A Pd-MOS diode as described above was attached to a small resistance heater and a type K thermocouple. This assembly was inserted into a vacuum chamber equipped with a Colutron ion source, which produced a mass analyzed, hydrogen ion beam (H_1^+, H_2^+, or H_3^+) with an energy adjustable in the range of 50–1000 eV [9]. A Faraday cup could be positioned in the beam to independently measure the ion flux to the target. At a given energy, a series of successive hydrogen ion doses in the range 0.1 to 5 μC was delivered to the diode, which was kept unbiased and connected to an electrometer to monitor the ion current during the exposures. After each ion dose, the diode was reverse biased at 0.5 V and the leakage current recorded. The power consumption of the diode was 200 μW or less. The heater was used to control the temperature of the diode so that the temperature dependence of the leakage current could also be determined.

A similar diode assembly was installed in the ZT-40M reversed field pinch at Los Alamos, where it was exposed to a series of deuterium plasma discharges. It was placed in the diagnostic section one octant away from the flux gap at a radial position 40 cm behind the liner and had a 0.013 sr solid viewing angle. This location allowed only charge-exchange neutral particles to strike the detector due to a strongly varying magnetic field that swept ions out of the path line to the sensor. A gate

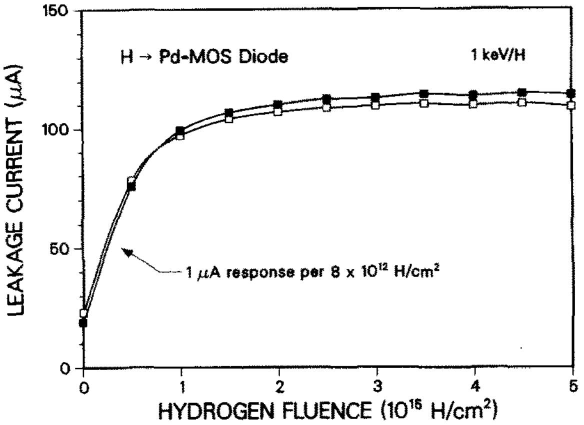

Fig. 3. Signal response of a Pd-MOS diode exposed to a 0.02 cm^2 hydrogen ion beam at an energy of 1 keV/H. At this energy, the device response saturates at a fluence of $(1-2) \times 10^{15}$ H/cm^2. The leakage current was measured while the diode was biased at 0.5 V. The two curves were obtained on the same device after an intervening annealing treatment to 110 ° C.

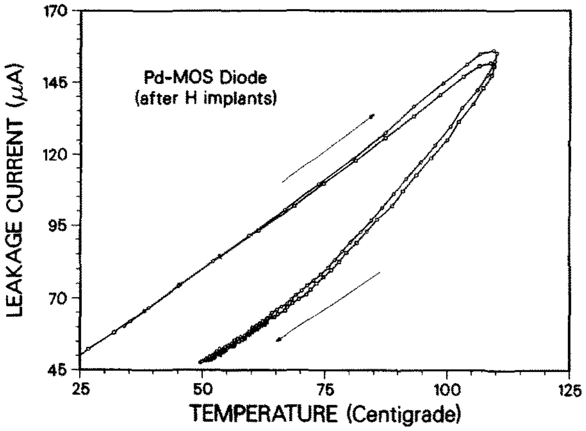

Fig. 4. Signal response of a Pd-MOS diode versus temperature. The data were obtained during two heating and cooling cycles following hydrogen implantation of the diode to saturation. The sampling interval was 10 s.

valve positioned in front of the diode assembly allowed it to be isolated from the plasma chamber. During pulse discharge cleaning cycles, this valve was kept closed. For these measurements, the diode was reverse biased at 1.5 V using a battery and the leakage current was measured continuously using a electrometer. A fiber optic link supplied an analog signal to the ZT-40M data acquisition system which recorded the leakage current through the diode immediately before and for a 100-s period after each plasma discharge monitored.

4. Results

The diode response at 25 ° C to a 1 keV/H ion beam is shown in fig. 3, which illustrates the sensitivity and saturation behavior of the device. Initially, the diode's leakage current increases by 1 μA, an easily measurable quantity, for each 8×10^{12} H/cm^2 that strike the diode. The response time is less than 0.1 s, the limiting factor being the time required to connect the diode to the measuring circuit, apply the bias, and make the reading. The device's response is dosimetric, that is, the diode signal persists after the ion beam is turned off and indicates the total H fluence seen by the diode. After a fluence of $(1-2) \times 10^{15}$ H/cm^2, the diode response saturates and no signal increase occurs with further exposure to the ion beam.

The leakage current decays very slowly at 25 ° C with no further exposure to hydrogen. At saturation, the leakage current drops about 10% after six hours in vacuum. The decay time is influenced by the surface cleanliness of the diode and the presence of oxygen-containing gases. Cleaning the surface by Ar$^+$ bombardment and/or exposing the diode to O_2(g) causes a rapid drop in the leakage current.

Nearly complete recovery of the diode's sensitivity to hydrogen can be obtained by heating the diode to 100–200 ° C for a few minutes. This heating process evidently purges hydrogen from the diode, although the amount released was below the detection limit of a

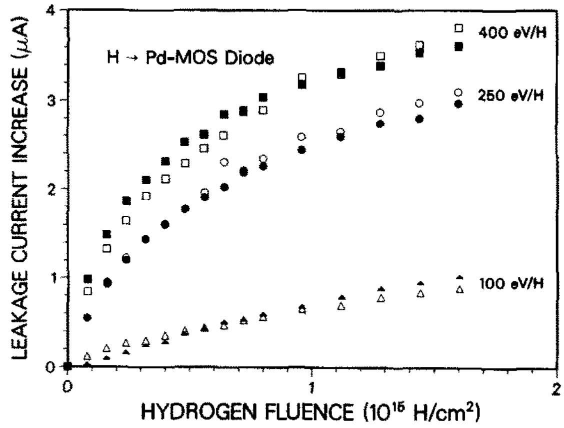

Fig. 5. Signal response of a Pd-MOS diode to hydrogen ion beams of various energies. The diode was annealed before beginning the exposure at each energy. The diode used for these measurements has a diameter of 400 μm.

residual gas analyzer connected to the vacuum chamber. When the diode returns to 25 ° C after annealing, its leakage current drops back to a characteristic background value. It should be noted that the characteristic background value obtained after such a procedure (e.g., 20 μA) is higher than the leakage current through a diode that has never been exposed to hydrogen (e.g., < 1 μA). However, one this characteristic background level is established, it remains constant for many cycles of hydrogen exposure and purging.

The response of a conditioned Pd-MOS diode to energetic hydrogen is repeatable, as demonstrated by the two curve in fig 3. Between recording the response curves, the diode was annealed to 120 ° C for 3 min and then allowed to cool back to 25 ° C. The reproducibility of the curve is approximately 10%. The main factor affecting the variation appears to be the temperature of the diode, which in these experiments was controlled only to ±3 ° C.

The temperature dependence of the diode leakage current is shown in fig. 4. As before, the curves were obtained for a diode that had been previously saturated with H and then annealed. The hydrogen induced contribution to the leakage current is evident from the difference in current seen at each temperature during heating and cooling. Heating releases hydrogen so lower leakage currents are seen during cooling. The effect is reproducible as demonstrated by the two curves in fig. 4 which show the temperature dependence of successive annealing cycles following diode saturation. The temperature dependence is not as strong as predicted by Eq. (1). Evidently, a thermionic emission model is not completely adequate to describe the behavior of Pd-MOS diodes in the presence of hydrogen.

It was found that the response of the diode was energy dependent: the increase in leakage current per unit dose was smaller at lower energies. This is illustrated in fig. 5, which shows response curves for three different incident H energies. The signal response per unit dose diminishes as the incident hydrogen energy decreases. Also, saturation does not occur as readily at the lower H energies and the diode can tolerate higher H fluences before its response saturates.

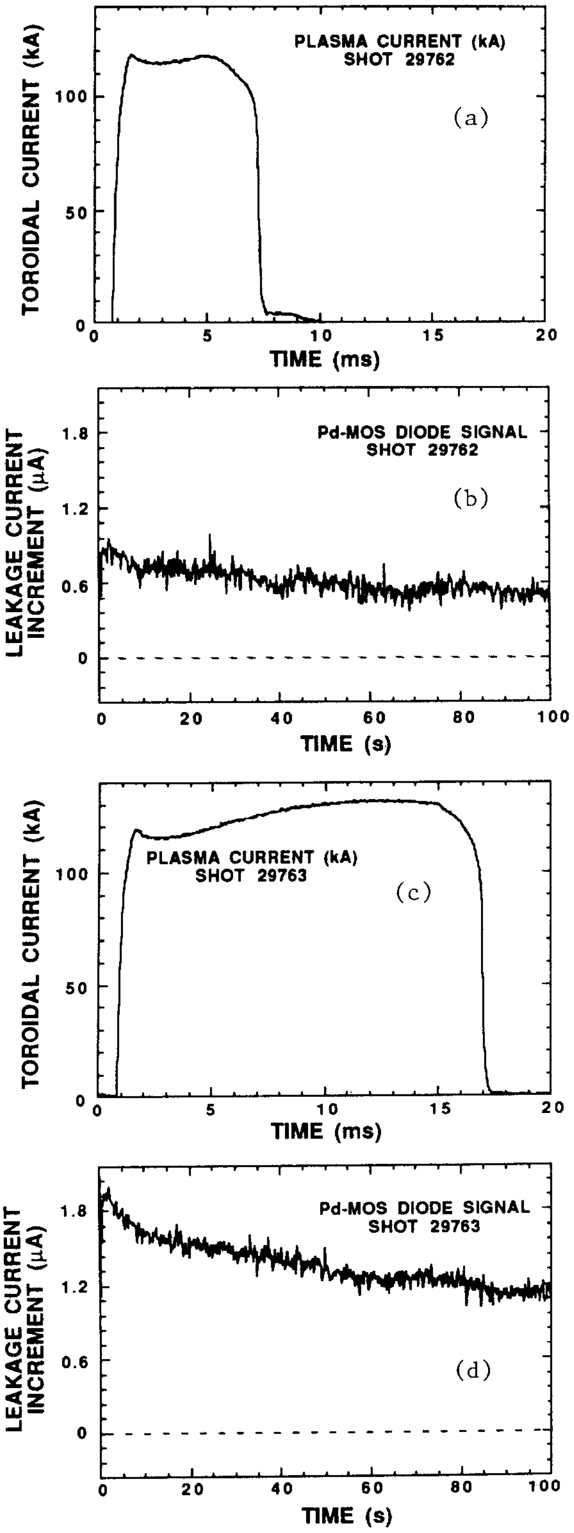

Fig. 6. Signal response from diode exposed to deuterium plasmas in ZT-40M. Curves (a) and (c) indicate the toroidal plasma current during two exposures, and curves (b) and (d) show the corresponding changes in the diode signal following each discharge. The measured hydrogen fluence per shot scales with discharge length.

The response of a diode placed in the ZT-40M reversed field pinch to deuterium plasma discharges of 7 and 16 ms duration is shown in fig. 6. During these discharges, the plasma conditions were kept constant, except for the discharge length, as indicated by the steady toroidal current. The diode signal scaled with the discharge length, indicating that the hydrogen flux to the sensor was nearly constant during the predominant flat top portion of the discharge. A noticeable decay in the diode signal was seen with time. This can be attributed to the relatively high partial pressure of oxygen-containing residual gases in the vicinity of the detector, which promotes hydrogen release from the device.

The maximum increments of the diode leakage current shown in fig. 6, together with the discharge lengths, diode calibration, and solid viewing angle imply an average flux of $\bar{\Gamma} = (6.8 \pm 0.3) \times 10^{15}$ H^0/vm^2-s-sr. This flux represents approximately 50% of the total steady-state charge-exchange neutral efflux computed using the code NEUCG2 [10] for this type of ZT-40M discharge. The inferred flux, $\bar{\Gamma}$, is bracketed by setting low-energy cutoffs in the numerical evaluation of the total efflux and assumes a response threshold of 30–40 eV. The difference between the measured and calculated values is within the estimated uncertainty in the calculation.

5. Discussion

Pd-MOS diodes provide a sensitive means for detecting energetic hydrogen. Like the carbon-film resistance probe [11], which has also been used to monitor charge exchange particle fluxes in fusion devices [12], a Pd-MOS diode is small in size and provides an electrical readout of the accumulated hydrogen fluence incident on its surface. The maximum fluence that can be measured in both types of devices is limited to about 2×10^{15} atoms/cm^2, but Pd-MOS diodes can be regenerated by heating when this limit is reached.

In order to utilize Pd-MOS diodes in a plasma-edge diagnostic, several design and operating requirements must be considered. First, because of its high sensitivity to hydrogen and its saturation behavior, a diode must be positioned so that the expected incident flux and particle energy are matched to the diode's signal range. Considering that the diodes can detect $\ll 10^{13}$ H/cm^2 and that saturation occurs at $(1-2) \times 10^{15}$ H/cm^2, the high-energy incident flux should be about 10^{13}–10^{14} H/cm^2 s for plasma discharge lengths of 10 s or less if several measurements are to be made before reaching diode saturation. Since hydrogen fluxes at the wall in large fusion experiments can be rather high (e.g., 10^{14}–10^{16} H/cm^2 s), it may be necessary to locate the detector at a larger radial distance from the plasma than the wall.

Second, Pd-MOS diodes are best suited for use as shot-to-shot hydrogen flux monitors and cannot conveniently be used for obtaining time resolved hydrogren flux data during the course of a single discharge. This limitation results from interference during a plasma discharge that arises from photon absorption and charged particle collection. Pd-MOS diodes produce significant photocurrents which can add to a hydrogen-induced current. Also, ions and electrons striking or being emitted from the diode surface can affect current measurements. Consequently, the diode signal while being irradiated with photons and charged particles from a plasma may not be representative of the hydrogen fluence. Fortunately, the hydrogen induced diode signal persists while the transient photocurrents and particle currents cease soon after a discharge ends. Thus, it is possible to eliminate these interferences by reading the diode signal after the termination of a discharge. This ability to separate the hydrogen signal from interferences comes at the expense of not being able to make measurements during a plasma shot. It is of course possible to place a shutter in front of a diode or array of diodes in order to obtain such information, if the added complexity can be accommodated.

Another consideration in using Pd-MOS diodes to measure energetic hydrogen is the effect of the incident particle energy upon sensor response. The response per unit dose decreases at lower energies. This effect appears to result from the energy dependence of the reflection coefficient and enhanced release of hydrogen implanted close to the diode surface. If there is no information available about the energy distribution of the incident hydrogen flux, it is not possible to obtain an absolute measurement of the hydrogen fluence from the diode signal response. However, if the energy distribution of incident particles is known, then quantitative fluence measurements are possible. Similarly, if the incident particles are monoenergetic and the flux is known, then the diode response provides a measure of the particle energy. Ideally one would like to obtain both energy and fluence information. This can be done by using an array of diode sensors that have differing energy-response characteristics. One approach is to deposite Au layers of varying thicknesses on top of the diodes in an array to act as high-pass energy filters. The response of each diode in the array to energetic hydrogen then depends upon the properties of its filter, and the collective response of the diodes can be used to determine both the hydrogen energy and fluence. The fabrication and testing of such diode arrays is outside the scope of this paper and is described elsewhere [8,13].

For application as a plasma-diagnostic, it is important to monitor diode temperature, since a significant temperature effect on leakage current is observed. Surface heating of the diode during plasma exposure may occur and this must be taken into account if readings are made before the diode equilibrates with its surroundings. Excessive heating can also cause release of hydrogen and a diode's temperature should be kept below 100 ° C to ensure that retained hydrogen is not released. Because the diode's high sensitivity requires that it be well shielded from large particle fluxes, this requirement can likely be met by appropriate positioning.

Pd-MOS diodes have been tested using H and D sources and respond to both isotopes. It is possible that an isotope effect exists but none has yet been observed. The work function decrease at the $Pd-SiO_2$ interface per hydrogen depends upon the strength of the induced dipole, which should be different for the hydrogen isotopes. It is expected that Pd-MOS diodes will also respond to tritium. The β decay current of T at saturation is calculated to be < 1 nA, which is small in comparison to the leakage current induced by a hydrogen isotope. Consequently, there does not appear to be any intrinsic difficulty in detecting T with Pd-MOS diodes.

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