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Photon shield for atomic hydrogen plasma sources

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Atomic hydrogen sources are usually strong photon emitters. To produce an atomic hydrogen beam and suppress the undesired photon flux, a small but effective light blocking device has been developed which fits into the quartz tube of a hydrogen plasma source. The device is made of stainless steel and uses angled passages and offset throughholes to absorb plasma generated photons while permitting hydrogen atoms and molecules to pass. The photon flux was reduced by a factor of at least 10^4 , whereas an attenuation of the H atom flux was not observed. By measuring the average velocity of the H atoms passing through the light blocker it has been shown that this device in the microwave plasma tube produces a room temperature thermalized atomic hydrogen beam. © 1999 American Vacuum Society. [S0734-2101(99)02302-7]

The interaction of hydrogen atoms with surfaces has been studied intensively for decades in various fields of surface physics and material science. Surfaces may be reconstructed or modified and surface reactions may be initiated by exposure to atomic hydrogen. For semiconductors, atomic hydrogen is used to etch, clean or passivate surfaces. For these reasons, various methods have been developed to dissociate H_2 molecules in an ultrahigh vacuum (UHV) environment using either thermal or plasma dissociation.^{1–8}

In the case of thermal dissociation, the UHV chamber is typically backfilled with H₂ ($p \approx 10^{-4}$ Pa) while a tungsten filament is resistively heated to above 2000 K.⁵ Higher fluxes and lower H₂ background pressures are provided by recently described H atom sources using hot tungsten capillaries or surfaces^{1,4-6} (1700–2200 K). Alternatively, plasma sources based on a microwave or radio frequency discharge produce H atoms from H₂ flowing through a quartz tube within a resonant cavity.^{2,3,7} To thermalize the atoms a cryogenic reflector may be used.⁵ The dissociation rate as well as contamination in the H atom beam have been investigated carefully for the various methods and different conditions.^{5–8}

Both thermal and plasma sources of atomic hydrogen are strong emitters of photons. Thermal sources give rise to visible and infrared radiation, while the more energetic plasma emits strongly in the near ultraviolet (UV). Little attention has been focused on blocking the photons created by the sources. The strongest UV line of atomic hydrogen is found at approximately 10.2 eV (Ref. 9) which may significantly effect surface reactions under study or induce undesirable surface changes. The separation of photons from the atomic or molecular hydrogen is essential for many experiments; either hydrogen atoms may be extracted from the beam or the photons may be selectively absorbed. Selective H atom extraction has the advantage that molecules are also suppressed. One method uses strong inhomogeneous magnetic fields which deflect H atoms from the beam based on their magnetic moment.¹ Synchronized double choppers may also be used as velocity filters; however, with an average thermal velocity of 2500 m/s at room temperature and chopper frequencies of 2500 Hz the distance between the choppers must be at least 0.5 m. Therefore, long travel tubes or large deflection magnets are necessary.

We have developed a small but effective light blocking fixture which selectively absorbs photons in a standard microwave discharge source. Details of the source are shown in Fig. 1. In the side view of Fig. 1(a), two quartz tubes (B) with 13 mm outside diameter are connected by Cu-sealed UHV flanges which are welded on stainless steel-quartz transitions (C). The metal connect between the tubes blocks any light waveguided in the quartz. Molecular hydrogen is introduced into the left tube which is surrounded by a 2.45 GHz McCarrol microwave cavity (A). The microwave radiation is supplied by a 100 W power supply (KIVA Instruments). The pressure in the tube typically varies between 20 and 40 Pa to sustain the plasma. The right quartz tube enters the first vacuum chamber through an O-ring compression fitting (D) sealed to a stainless steel nozzle fixture which also mounts to the first skimmer cone for beam shaping. A crosssectional view of the fixture is shown in Fig. 1(b). It shows the quartz tube (B) with the inserted light blocker device (E).

The light blocker is a two-piece stainless steel cylinder. The first piece has eight inclined passages which meet in a coaxial straight through hole. The second piece is behind the

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FIG. 1. (a) Side view of the hydrogen plasma source: (A) microwave cavity, (B) 13 mm quartz tubes, (C) steel–quartz transitions, and (D) O-ring compression fitting. (b) Magnified cross-sectional view through light blocker (E), stainless steel nozzle fixture with pinhole (F), and skimmer cone (G). (c) Front and back view of the light blocker.

first and has eight offset through holes. The front and back view of the light blocker are the same and shown in Fig. 1(c). There is no straight path through the device and hence photons are absorbed after one or multiple reflections. Hydrogen atoms and molecules pass through the channels and leave the quartz tube through a pinhole (F) 1 mm in diameter. The distance between the pinhole and the 0.7 mm orifice of the skimmer cone (G) is approximately 1 mm. After the skimmer cone the beam passes two further differential pumping stages before entering the analysis chamber. When the beam shutter is opened the pressure in the analysis chamber increases typically from 10^{-8} to 10^{-7} Pa.

The atomic hydrogen content of the beam $c_{\rm H}$, which is the number of H atoms divided by the total number of beam particles, is measured with a line-of-sight mass spectrometer from the intensity of the mass 2 peak. With the mass spectrometric intensities of the mass 2 channel with the plasma on ($I_{\rm on}$) and off ($I_{\rm off}$) the percentage α of dissociated H₂ molecules in the beam may be written as

$$\alpha = \frac{I_{\text{off}} - I_{\text{on}}}{I_{\text{off}} - I_b},\tag{1}$$

where I_b denotes the H₂ background signal when the beam shutter is closed. From α the atomic hydrogen content may be calculated by

$$c_{\rm H} = \frac{2\,\alpha}{\alpha+1}.\tag{2}$$

After inserting new quartz tubes and a new light blocker, the hydrogen source must run for more than 6 h until $c_{\rm H}$ rises above the limit of detection, i.e., 2%. After this induction period, the source delivers atomic hydrogen with values of $c_{\rm H}$ between 10% and 30%, depending on the plasma conditions. This same hydrogen content is also found if the light blocker is removed, demonstrating that the atomic hydrogen flux is not significantly reduced by the device. The physical processes on the stainless steel surfaces during the induction period are not clear. Most likely, the contamination and oxide layers are removed by atomic hydrogen until the surfaces are saturated with hydrogen. Apparently, further adsorption of atomic hydrogen on the stainless steel parts is strongly inhibited after the initial induction period.

To estimate the photon suppression, we fabricated a largearea Schottky diode with an ultrathin Ag film of 60 Å on Si(111) for photodetection. This sensor is sensitive to photons with energies greater than the Schottky barrier height, which for our *n*-type diode had a measured value of 0.55 eV. The photodiode was kept at 135 K during measurements to reduce the noise level which was typically below 0.5 pA. The photocurrent was measured using conventional lock-in techniques while modulating the beam at 471 Hz with a mechanical chopper. Both hydrogen and neon plasmas were used and the photoresponse was measured with and without the light blocker in the beam. The hydrogen plasma is a strong UV emitter above 10 eV, and the neon plasma is a bright source in the visible. The photocurrents without the light blocker were 4 and 3 nA for the hydrogen and neon plasmas, respectively. No photocurrent above the noise floor was detected if the light blocker was inserted. Hence, the device reduces the total photon flux by at least a factor of 2×10^4 for the wavelength ranges of these two plasmas.

A similar thin film Schottky diode was used to detect atomic hydrogen. We have described this new atomic hydrogen sensor elsewhere,¹⁰ and we have found it to be insensitive to H₂ molecules. Using the H atom sensor and the mechanical beam chopper, we could determine the average velocity $\bar{\nu}$ of the hydrogen atoms by measuring the phase difference φ between reference and signal channel of the lock-in amplifier as a function of the chopper frequency. If the average travel time of the particles between chopper and



FIG. 2. Difference of the signal phases as measured by the lock-in amplifier for an atomic hydrogen and deuterium beam as a function of the chopper frequency. The solid line is a least-squares fit through the data. The reciprocal of the slope is proportional to the average atom velocity.

sensor is equal to or smaller than the reciprocal of the chopper frequency *f*, the phase difference may be written as

$$\varphi = -\frac{2\pi f d}{\bar{\nu}} + \varphi_e(f) + \varphi_0, \qquad (3)$$

where *d* is the distance between sensor and chopper wheel, $\varphi_e(f)$ is the frequency dependent phase shift due to electrical connections and filters in the amplifiers, and φ_0 is a constant phase shift. The first term on the right-hand side of Eq. (3) represents the phase shift due to the travel time of the atoms. Since φ_e and φ_0 are unknown, the experiment was repeated with atomic deuterium, and the phase difference $\Delta \varphi = \varphi_H$ $-\varphi_D$ was measured as a function of *f*. Using the relation $\overline{\nu}_H = \sqrt{2} \, \overline{\nu}_D$, the average velocity of the hydrogen atoms may then be determined from

$$\bar{\nu}_{\rm H} = \frac{2\pi f d}{\Delta \varphi} (\sqrt{2} - 1). \tag{4}$$

In Fig. 2, $\Delta \varphi$ is shown as a function of the chopper frequency *f*. The two quantities show a linear dependence in excellent agreement with Eq. (4). Using d=0.28 m and the slope of the linear fit from the data in Fig. 2, one obtains an

average hydrogen velocity of approximately 2650 m/s. Assuming a Maxwellian velocity distribution,¹¹ this value corresponds to a temperature of $T = \pi m_{\rm H} \bar{\nu}_{\rm H}^2/8k_B = 336$ K which is close to room temperature. Thus, the plasma tube with light blocking device produces a thermalized H atom beam due to the multiple reflections in the stainless steel passages. Cooling this device may result in an atomic hydrogen beam of lower temperature.

In summary, we have introduced a small light blocking device which fits into a conventional plasma tube for atomic beam production. The device suppresses photons in the beam while allowing passage of plasma-produced hydrogen atoms. The temperature of the H atoms was estimated by measuring the average velocity and was found to be close to room temperature. Therefore, the light blocker offers a simple but effective method of producing a dark, thermal source of atomic and molecular hydrogen.

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- ¹M. A. D. Fluendy, R. M. Martin, E. E. Muschlitz, and D. R. Herschbach, J. Chem. Phys. **46**, 2172 (1967).
- ²A. Ding, J. Karlau, and J. Weise, Rev. Sci. Instrum. **48**, 1002 (1977).
- ³J. T. M. Walraven and I. F. Silvera, Rev. Sci. Instrum. 53, 1167 (1982).
- ⁴U. Bischler and E. Bertel, J. Vac. Sci. Technol. A 11, 458 (1993).
- ⁵K. H. Bornscheuer, S. R. Lucas, W. J. Choyke, W. D. Partlow, and J. T. Yates, Jr., J. Vac. Sci. Technol. A **11**, 2822 (1993).
- ⁶V. M. Bermudez, J. Vac. Sci. Technol. A 14, 2671 (1996), and references therein.
- ⁷S. M. Rossnagel, in *Thin Film Processes II*, edited by J. L. Vossen and W. Kern (Academic, San Diego, CA, 1991), p. 11.
- ⁸J. Kikuchi, S. Fujimura, M. Suzuki, and H. Yano, Jpn. J. Appl. Phys., Part 1 **32**, 3120 (1993).
- ⁹J. Reader and C. H. Carliss, in *CRC Handbook of Chemistry and Physics*, 77th ed., edited by D. R. Lide (Chemical Rubber Corp., Boca Raton, FL, 1996), pp. 10–41.
- ¹⁰H. Nienhaus, H. S. Bergh, B. Gergen, A. Majumdar, W. H. Weinberg, and E. W. McFarland, Phys. Rev. Lett. **82**, 446 (1999).
- ¹¹U. G. F. Weston, *Ultrahigh Vacuum Practice* (Butterworths, London, 1985), p. 277.