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THE PREPARATION OF THIN FILM
SOLAR CELLS UNDER VERY LOW
PRESSURE CONDITIONS

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FINAL REPORT

Ames Laboratory-DOE
Iowa State University
Ames, Iowa (50011)

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THE PREPARATION OF THIN FILM SOLAR CELLS UNDER VERY LOW PRESSURE CONDITIONS

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ABSTRACT

In this study the feasibility of fabricating backwall Schottky barrier polycrystalline solar cells under ultra-high vacuum conditions of 1×10^{-10} torr (N_2) was investigated. Thin films of electron beam vaporized silicon were deposited on cleaned metal substrates of tungsten, tantalum and hafnium. Mass spectra from the quadrupole residual gas analyzer were used to determine the partial pressure of peak heights of 13 residual gases during each processing step. During separate silicon depositions, the substrate temperature was varied between 400 and 750°C and deposition rates between 20 and 750 Å/min were used. Surface contamination and metal diffusion were monitored by in situ Auger electron spectrometry before and after cleaning, deposition and annealing. Auger depth profiling, X-ray analysis, and SEM in the topographic and channeling modes, were utilized to characterize the samples with respect to silicon-metal boundary layer, interdiffusion, silicide formation and grain size of silicon. The clean metal surface was found to enhance thin film silicide growth. Fine grain silicon films were obtained for all samples that were not completely converted to a metallic silicide. Tungsten, tantalum and hafnium were found to form silicides at temperatures as low as 600°C.

PREFACE

This study was a collaborative effort between the Space Systems Division, National Aeronautics and Space Administration (NASA), Langley Research Center, and the U.S. Department of Energy, Division of Basic Energy Sciences (BES). All of the equipment related to and including the space simulation chamber, the in situ analytical instrumentation, and the services of a post doctorate fellow for three fourths of a year were funded by NASA. Other operational costs were supported by the Materials Science Program (BES) of the Ames Laboratory. These costs included a one and a half scientific man year effort to assist in performing the various experiments and to carry out the surface analysis and depth profiling, the scanning electron microscopy, the X-ray and chemical analysis, and the determination of the electrical properties of the thin films.

This investigation represents our initial effort concerning the fundamental materials problems associated with photovoltaic materials. It has proven to be a convenient and highly effective way to move a substantial part of our program into the energy-technology realm; we are now deeply involved in the problems associated with the formation of efficient backwall Schottky barrier solar cells. We believe that, as a result of this work, our Laboratory has developed a unique capability for making such studies; the expansion in this effort demonstrates our confidence in and enthusiasm for this program.

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INTRODUCTION

Converting sunlight into electricity without using a thermodynamic cycle is a major driving force for the development of photovoltaic cells. The widespread use of solar cells is predicated on (1) the ability to prepare high efficiency devices, (2) their having a substantial lifetime and (3) their being economically produced. The present cost of single crystal cells still is excessive when compared with other energy sources and much work is currently in progress to find less costly methods of preparing efficient devices. These film polycrystalline cells avoid some of the costs of single crystal cells but they presently suffer from low output efficiency and a lack of a viable method of mass production.

Schottky barrier solar cells (SBSC) or metal-semiconductor solar cells have recently shown considerable promise. The basic frontwall device consists of a semiconductor substrate (e.g. n-type silicon) onto which a thin ($100 \text{ \AA}$) metal layer is evaporated forming a Schottky barrier. Photons enter the semiconductor through the metal layer side and if $h\nu > E_g$, electron-hole pairs are formed in the semiconductor and are separated in the depletion layer to generate a photovoltage. A possible major advantage of the SBSC is that polycrystalline semiconductors could be utilized to form the Schottky barrier. Further, for a SBSC a single thin film layer of polycrystalline semiconductor would be required. Pulfrey and McQuat⁽¹⁾ have shown that theoretical efficiencies of a SBSC are essentially was same as for p-n junction type cells and they stressed the simplicity of formation of the former over the latter as an advantage in terrestrial applications where economic considerations are dominant. It is a major conclusion of their work that the efficiency of a SBSC increases with increasing barrier height and reaches values of 25% for semiconductors with E_g near 1.5 eV.

The simple theory of metal-semiconductor interfaces due to Schottky,⁽²⁾ states that the electrostatic barrier, ϕ_B , is equal to the differences in the work function

of the metal, ϕ_M , and the electron affinity of the semiconductor, χ_S , i.e., $\phi_B = \phi_M - \chi_S$. However, actual measurements revealed that this relationship does not always hold.⁽³⁾ For ionic semiconductors the relationship does hold, but for covalent or predominately covalent semiconductors, ϕ_B is only weakly dependent on ϕ_M . Bardeen⁽⁴⁾ first proposed that surface states were the cause of the relative independence of ϕ_B on ϕ_M . However, little direct information was available about the nature of the states. In 1966, Heine⁽⁵⁾ suggested that instead of "dangling bond" surface states, the penetration of the metal electron wave function into the semiconducting gap provided the mechanism to explain the variation of ϕ_B with ϕ_M . This idea has been further developed by Pelligrini.⁽⁶⁾ Inkson⁽⁷⁾ stated yet another explanation of the Schottky barrier heights that involves modification of the electron-electron (exchange and correlation potential) interaction. This modification results in a new feature, the convergence of the semiconductor conduction and valence bands at the interface. In parallel with these theoretical developments, experimental progress has resulted in the formation of intimate, ideal Schottky barriers. Cleaving semiconductors in a metal vapor stream at pressure of 10^{-10} torr resulted in intimate contacts reasonably free of interfacial oxide layers. However, not until 1975 were in situ measurements of work function and barrier height made on intimate n-type silicon contacts to non-transition metals.⁽⁸⁾

The phenomenological behavior of Schottky barrier diodes is well known. The current-voltage (I-V) characteristics of a Schottky diode, where the current arises from thermionic emission, is described by

$$J = J_0 e^{-\phi_B/kT} [e^{qV/kT} - 1], \quad (1)$$

where J_0 is the saturation reverse bias current equal to approximately $120 \text{ T}^2 \text{ (A/cm}^2\text{)}$ and V is the applied voltage. This relationship holds except at large reverse

bias where image barrier reduction occurs leading to $J > J_0$ and at low temperature where recombination currents are important. When Eq. (1) is satisfied, ϕ_B can be determined either by measuring J versus V or J versus T . Differential capacitance measurements for a semiconductor under reverse bias conditions give

$$C = A \left[\frac{q \epsilon N_0}{2(V_d - V)} \right]^{1/2} \quad (2)$$

where A is area of sample, V_d is diffusion voltage, ϵ is the dielectric constant and N_0 is the net doping density. From V_d and the Fermi energy, E_F , one can extract ϕ_B and from Eq. (2), the doping level actually present at the interface can be determined. Eq. (2) requires an extrinsic semiconductor with uniform doping. A third method of measuring ϕ_B involves the measurement of photoresponse of the diode to less than band gap light which produces a photocurrent J_p proportional to $(qV - \phi_B)^2$. When carefully done on well characterized Schottky diodes, results obtained on the same device by these three methods generally agree within a few percent.

Since the barrier height determines the bias across the metal-semiconductor interface and hence the charge separation in the solar cell, the efficiency of the cell is critically dependent on the barrier height. Although series resistance, carrier lifetime and proper semiconductor doping level all affect the efficiency, the first step in the preparation of an efficient cell is the formation of a sharp interface and high Schottky barrier (1 eV).

In addition to the traditional metal on semiconductor Schottky barrier solar cells, it is also possible to prepare a cell by depositing the semiconductor on a metal substrate (backwall Schottky barrier) but this configuration requires a well defined and reproducible metal surface. Since the photovoltaic property originates at the interface between the two materials, the output of the cell can be greatly impaired by the various defects in the component materials. Some of these defects

can be related to elemental contaminants in both materials but the residual gases that comprise the environment in which the device is produced are also contributing sources. These gases can adhere to the surface of the substrate as an extremely thin layer of molecules thereby serving as heterogeneous nucleation sites for defects. Considerable improvement in the output efficiency may be possible if processing is performed under more controlled surface conditions and in a less contaminating environment. A strong correlation with interface quality would suggest the use of a non-contaminating space environment, such as the Molecular Shield Facility proposed by the Langley Research Center,^(9,10) for the preparation of efficient thin film solar cells.

During the past year a feasibility study of the preparation of backwall Schottky barriers has been made. This work was a collaborative effort between the Space Technology Branch of the National Aeronautics and Space Administration, (NASA), Langley Research Center and the Materials Science Program of the Department of Energy, (DOE); Ames Laboratory, Iowa State University. The object of the study was to deposit silicon on various metal substrates under very clean high vacuum conditions and to correlate Schottky barrier height with interface quality. Tungsten, tantalum and hafnium were chosen as substrate materials because their barrier heights, as calculated from work function data for p-type silicon,^(11,12) are high. Further, these metals also have high melting points, have relatively low coefficients of linear thermal expansion and are not expected to form compounds with silicon below 1200°C based on equilibrium phase diagrams.^(13,14,15)

EXPERIMENTAL PROCEDURE

In the following subsections the materials and apparatus used for the present study are described. The thin films of silicon were deposited on the various metal substrates inside a space simulation chamber previously used to develop a preprototype electrotransport apparatus⁽¹⁶⁾ for NASA. The first six-month period of this work consisted of revamping this high vacuum chamber and equipping it with the necessary instrumentation so that (1) silicon could be vaporized under low pressure, (2) the temperature of the substrate could be controlled and (3) the surfaces of interest could be analyzed in situ with Auger Electron Spectroscopy (AES).

A. Materials

(a) Silicon. The ingots used as the silicon vapor source were obtained from the Dow Corning Corporation, Hemlock, Michigan. They measured ~ 2.5 cm (1 inch) in diameter, were p-type and had an electrical resistivity of 13 to 15 ohm-cm. Sections were cut from each ingot using a diamond saw, and then surface cleaned and analyzed by spark source mass spectrometry. It was soon learned that the ingots contained large amounts of hydrogen that was emitted upon heating. For this reason the top portion of each ingot was electron beam melted for 1 to 2 hours under a pressure of 1×10^{-6} torr (N_2) prior to its use in the space simulation chamber. In this operation no crucible was used and melting was continued until the pressure in the furnace was 1×10^{-6} to 5×10^{-7} torr (N_2).

(b) Hafnium. The hafnium metal used as a substrate material was obtained from the Foote Mineral Company, Philadelphia, Pennsylvania. It had been refined by the van Arkel and de Boer crystal bar process and contained ~ 4 at.% zirconium as a major impurity⁽¹⁷⁾. The as received material was electron beam melted into

ingot form, jacketed in a stainless steel tube and hot rolled into sheet 0.20 cm (0.075 inch) thick. Final rolling to a thickness of 0.025 cm (0.01 inch) was done at ambient temperature in air. The hafnium substrates were sheared from this sheet and surface cleaned by electropolishing in a chilled methanol-perchloric bath⁽¹⁸⁾ before they were loaded in the space simulation chamber.

(c) Tantalum. The tantalum substrates and tantalum adapters for the sample assembly were sheared from 0.013 cm (0.005 inch) sheet. This material was obtained from the Kawecki Berylco Company, New York, New York. The substrates were electropolished in a methanol-sulfuric acid bath prior use in the experiments.

(d) Tungsten. The tungsten sheet from which the substrates were prepared, was obtained from the Rembar Company, Dobbs Ferry, New York. It also measured 0.013 cm (0.005 inch) thick and was surface cleaned before use.

A complete chemical analysis of the two silicon ingots and the hafnium, tantalum and tungsten sheets after surface cleaning is shown in Appendix A. Spark source mass spectrometry was used as the method of analysis except for the interstitial solutes in the hafnium, tantalum and tungsten. For these metals the carbon concentration was determined by combustion chromatographic analysis and the oxygen, nitrogen and hydrogen contents were determined by vacuum fusion analysis.

B. Apparatus

The experiments for this study were performed inside the space simulation chamber that was originally built and used to develop a preprototype electro-transport purification for NASA through the Langley Research Center (LRC) Hampton, Virginia. It is equipped with cryosorption, titanium sublimation and differential ion pumps. The chamber contains a large surface area (100 m^2)

honeycomb panel that, when filled with liquid nitrogen, serves as a high capacity cryogenic pump. The entire vacuum chamber is bakeable to 380°C and ultimate pressures of 5×10^{-11} to 5×10^{-12} torr (N_2) are routinely obtained. The residual gas pressure is measured using calibrated modulated and unmodulated ionization gages from LRC and a UTI Residual Gas Analyzer Model 100C. With this instrumentation and the aid of a digital computer, the partial pressure of 13 residual gases can be obtained and their actual weight concentrations determined.

(a) Modification in Space Simulation Chamber. The major modifications in the space simulation chamber prior to its use in this study were the addition of the appropriate pipe extensions containing high vacuum flanges to accommodate the various instruments and feedthroughs. The experimental system is shown schematically in Figure 1. Instrumentation includes a Varian Model 981-2607 cylindrical mirror analyzer for AES, a 3M minibeam ion gun for possible sputter cleaning and Auger depth profile analysis. A Physical Electronics Industries (PHI) 100 watt electron bombardment gun was available for electron stimulated desorption cleaning as was a Varian electron gun Model 981-2454 for relative work function measurements. A Veeco - 6 kW Pierce type electron beam gun, with electrostatic focusing, was used for heating the silicon and subsequent vapor deposition. The silicon ingots were placed on a stainless steel pedestal that was surrounded by a tantalum shield and chimney assembly (item J, Figure 1). During silicon vaporization only the top of the ingot was melted and this pool held to the solid portion of the ingot by surface tension. In this manner no mold assembly was required to contain the molten silicon and the entire ingot was heated which aided the initial degassing of the material.

(b) Rotating Substrate Assembly. A rotating substrate assembly, items C and H in Figure 1, was developed so that the metal substrates could be heated and positioned in the optical path of the selected instrument and the silicon vapor

Figure 1. Schematic drawing showing positions of components to surface clean and analyze specimens and to deposit thin films of silicon under ultra-high vacuum.

source. This assembly consisted of a specimen manipulator, PHI Model 10-503, and a sample holder designed and fabricated in our Laboratory. The sample holder or carrousel, held three metal substrates. A fourth position on the carrousel held a small metal plate that served as a cover for the silicon vapor source. A photograph of the carrousel is shown in Figure 2. Two carrousels were actually made; one fabricated from 0.10 cm (0.040 inch) thick 304 stainless steel and one from oxygen-free-high-conductivity (OFHC) copper sheet of the same thickness. Both units measured 15.3 cm (6.0 inch) in diameter and were so designed that each quadrant is electrically insulated from its adjacent section. To each quadrant a power cable was attached which passed through the chamber wall via a high amperage-low voltage electrical feedthrough. The metal substrates were mounted in a holder assembly that bridged between the quadrants. The substrate could then be resistively heated to a desired temperature.

The substrate holder assembly is shown in Figure 3. This assembly consists of two tantalum metal flats which are grooved and contain a tantalum adapter strap measuring 1.4 cm (0.55 inch) x 2.2 cm (0.87 inch) x 0.013 cm (0.005 inch) thick. The adapter straps are bent in a "S" shape and crimped into the slot of the tantalum flats. Two parallel cuts, approximately 0.30 cm (1/8 inch) long, are made in the other end of the tantalum straps forming a tab into which the metal substrate to be studied is inserted and secured by spot welding. A chromel-alumel thermocouple 0.015 cm (0.006 inch) in diameter was parallel-gap welded to the backside of the substrate and passed through the high vacuum feedthrough contained in the flange assembly of the specimen manipulator.

An overall view of the space simulation chamber and related equipment is shown in Figure 4.

Figure 2. Carrousel and substrate holder assemblies used in the deposition and characterization of thin films of silicon on metal substrates.

Figure 3. Schematic of substrate holder assembly used in the silicon deposition and characterization studies.

Figure 4. Overall view of apparatus used in the deposition and characterization of thin films of silicon on metal substrates.

(c) Metal Evaporator. In an effort to have the capability of adding ohmic contacts to the thin film deposits of silicon in situ, a single stage metal evaporator was designed and fabricated. The evaporator was bakeable to 400°C and consisted of a tungsten or tantalum resistively heated crucible, a mask containing a network of small holes and a cover. With this unit gold or aluminum could be vaporized and directed through the mask to the sample while it was in place on the carrousel thereby forming a network of ohmic contacts. Although this unit was not actually used in the space simulation chamber it was shown to work quite satisfactorily and was calibrated for rate of gold deposition.

C. Procedure

The carrousel, power leads, mold assembly and 6 kW electron beam gun were first cleaned with methanol and vacuum degassed for 24 hours at 400°C under a pressure of 5×10^{-7} torr (N_2). For this operation a prebakeout chamber was used that would accommodate any unit capable of fitting inside the space simulation chamber.

The metal substrates, onto which a thin film of silicon was to be deposited, measure 1.4 cm (0.55 inch) x 2.2 cm (0.87 inch). The tantalum and tungsten substrates were 0.013 cm (0.005 inch) thick and the hafnium was 0.025 cm (0.01 inch) thick. Prior to loading into the substrate holder assembly (Figure 3), the specimens were electropolished⁽¹⁸⁾ and vacuum degassed at 900°C for 3 to 6 hours under a pressure of 2×10^{-6} torr (N_2) in a separate vacuum furnace. They were then spot welded into the holder assembly and the chromel-alumel thermocouple attached to the back side. Each substrate holder assembly was attached to the degassed carrousel, the power leads attached and the entire assembly loaded inside the space simulation chamber. The mold assembly containing the silicon ingot,

tantalum shield and 6 kW electron beam gun, was then bolted to the base of the chamber and main door of the chamber secured.

The vacuum chamber was evacuated to 1×10^{-2} torr (N_2) using the three cryo-sorption pumps which then were isolated from the system and one of the differential ionization pumps ("garbage" pump) started. The chamber and the second ion pump were baked at 380°C for seven days. At the beginning of the bakeout cycle, the pressure inside the chamber was $\sim 1 \times 10^{-7}$ torr (N_2). After seven days the pressure decreased to $\sim 1 \times 10^{-9}$ torr (N_2) while the chamber was still hot. All filaments in the system were degassed at temperature and again when the chamber was cooled to 250°C . When the chamber temperature reached 100°C the filaments were again degassed. The second ionization pump was then cooled to ambient temperature, its magnets attached and elements degassed. The entire system was cooled to room temperature, the garbage pump isolated, and the second ion pump started. The cryogenic panel was then filled with liquid nitrogen after which the pressure in the system was $< 5 \times 10^{-11}$ torr (N_2).

Prior to depositing silicon onto the metal substrates, the specimens were Auger analyzed and the surface contaminants noted. The substrate was resistively heated, usually to 1000 to 1100°C for 1 to 3 hours, after which the surface was again analyzed. The temperature gradient across the substrate during heating was less than $\pm 10^\circ\text{C}$ at 1000°C . This was confirmed using an optical pyrometer and correcting for the opaqueness of the sight glass and the emissivity of the specimen.

The peak heights in true amps of the 13 residual gases inside the space simulation chamber were monitored throughout each experiment. These gases were H_2 , He, CH_4 , H_2O , Ne, N_2 , CO, O_2 , Ar, CO_2 , C_2H_4 , C_2H_6 , and C_3H_8 . The procedure used to determine their partial pressure has been described in detail in a previous study⁽¹⁶⁾ and need not be repeated here. The only deviation from this procedure

was that a calibrated ionization gauge was not used in the present study. While this may introduce a systematic error in the partial pressure for a particular gas at a particular time, a comparison of the values for any two gases at a specific time or for a gas at any two times will still be accurate.

RESULTS AND DISCUSSION

In the following paragraphs the purposes and results of four silicon deposition experiments are given. The first experiment was a calibration experiment in which the evaporation rate of the silicon source was determined. In the other three experiments, thin films of silicon were deposited and characterized in situ and more extensively after the samples were removed from the space simulation chamber.

A. Experiment 47

The purpose of this experiment was to determine the vaporization rate of silicon as a function of the power delivered from the 6 kW electron beam gun. Three tungsten substrate assemblies were attached to the carousel and a tantalum strip 0.3 cm (1/8 inch) wide was attached across the face of each substrate. This strip served as a thin mask so that after the silicon was deposited, two sharp boundaries between the deposit and the tungsten would exist which could be used to measure the thickness of the coat after the

substrate was removed from the chamber. The chamber was evacuated to 3×10^{-6} torr (N_2) using only one of the ionization pumps. A small area on top of silicon Ingot I, was held molten for 15 minutes using 300 watts of electron beam power. This was done with the cover plate, substrate #4, in the 6 o'clock position over the silicon vapor source. After 15 minutes the carousel was rotated and silicon was vaporized onto substrate #1 for one hour. A similar deposit was placed on substrate #2 using 400 watts of beam power for ten minutes and on substrate #3 using 500 watts for five minutes. The substrates were cooled, removed from the chamber and their thickness measured using a Dektak thickness monitor manufactured by the Sloan Co. Figure 5 is a plot of the rate of silicon vaporization vs. the output power of the 6 kW electron beam gun. When 500 watts of power was applied to the ingot, approximately 75% of the surface was molten. In subsequent experiments, it was determined that 600 watts would melt the entire surface of the ingot and resulted in a vaporization rate of approximately $750 \text{ \AA}/\text{min}$. This was the maximum vaporization rate possible using this size ingot without using a retaining crucible.

B. Experiments 61, 87, and 107

The thin films of silicon were electron beam deposited on nine substrates, three each in three separate experiments, denoted by the numbers 61, 87 and 107. Series 61 and 87 each consisted of three fine grained tungsten substrates. The three substrates used in series 107 were tungsten, hafnium,

Figure 5. Plot of silicon vaporization rate as a function of output power from electron beam gun.

and tantalum. Silicon Ingot I was used in experiments 61 and 87 while Ingot II was used in experiment 107. The silicon deposition rates, R_D , were controlled by the power supplied to the electron beam gun and by adjustment of the distance between the substrate and the partially molten silicon ingot. The rate, R_D , varied from 20 to 750 $\text{\AA}/\text{min}$ and the effective silicon thicknesses ranged from 0.1 to 2.9 microns. The substrate temperatures during silicon deposition, denoted by T_D , were controlled by resistively heating with an alternating current and by adjustment of the substrate distance from the silicon ingot. These temperatures varied from 400 to 780°C and were monitored by thermocouples and sometimes by optical pyrometer readings.

The peak height of 13 residual gases contained in the space simulation chamber were measured at various stages of each experiment using the residual gas analyzer. These stages included 1) after chamber bakeout at 380°C, 2) after the cryogenic panel was cooled with liquid nitrogen, 3) during the resistive heating of the substrate, 4) during the silicon deposition and 5) after silicon deposition. The partial pressure for each gas was calculated and the results are listed in Appendix C for each specimen. As can be seen from these data the largest portion of the residual gas pressure inside the chamber during processing of the substrates was due to hydrogen. In some cases this concentration was as high as 98%. The

major source of hydrogen was the silicon ingots, and although extensive degassing procedures for this material were used, its partial pressure remained quite high. During the resistive heating step, used to surface clean the substrates, substantial increases in the partial pressure of the reactive gases H_2 , N_2 , CO , CO_2 and CH_4 were observed.

The major in situ diagnostic tool was the Varian Auger spectrometer which measured the surface contamination of the metal substrates and measured the presence of the substrate metal at the surface of the silicon deposit. If the metal substrate Auger signal appeared after silicon deposition, this implied that some interdiffusion had occurred between the substrate and the silicon. This was taken to mean that T_D was too high and/or R_D was too slow. This information was then used to determine T_D and R_D for the next experiment with the assumption that large grain silicon deposits required the highest possible T_D and the lowest possible R_D .⁽¹⁹⁾ In some experiments, the silicon films were annealed in situ while in others a second or third silicon layer was deposited on the same sample. A complete set of Auger data for each of the nine specimens at various stages of preparation are given in Tables IV, V, and VI in Appendix B. The entries in these Tables are to the nearest tenth of a percent and are meant to convey relative changes in surface concentration. The current state of quantitative Auger analysis would allow only a 20 to 50% confidence level in the absolute concentrations. Also, these values assume a homogeneous composition with depth.

A Varian work function apparatus was also incorporated into the vacuum chamber. Measurements revealed that precise alignment of the samples with respect to the work function electron gun was necessary to achieve reproducible results. An error of 0.1 mm produced a shift of 0.1 volt in the half current point of the usual I-V plot. Unfortunately, the present design of the carousel mounted on the manipulator did not give sufficiently reproducible results. Changes

in surface composition readily detected by the Auger spectrometer could not be readily confirmed with the work function apparatus. As a result, surface composition was monitored exclusively by the Auger spectrometer.

After the substrates were removed from the vacuum chamber, a variety of techniques were used for characterization. Optical microscope and scanning electron microscope (SEM) photographs were usually taken normal to the substrate plane. Wherever possible, the apparent grain structure of the silicon films were compared directly to that of the substrate near the edge of the silicon film. The purpose was to determine if the grain structure was truly that of the silicon layer or a mere replication of the substrate grain structure. Debye-Scherrer X-ray analysis was performed to determine whether the films were amorphous and to detect the presence of various silicide phases. Sharp X-ray lines implied crystallite size in excess of $100 \text{ \AA}$. Electron channeling measurements were used to determine if the grain size was larger than one micron. Diffuse X-ray lines implied amorphous films. Sharp X-ray lines and the absence of a distinct electron channeling pattern meant the grain size was between 100 and $10,000 \text{ \AA}$. This structure was denoted as "cryptocrystalline". Distinct electron channeling patterns implied grain sizes larger than one micron which we call large-grained polycrystalline. All nine deposited films were found to be cryptocrystalline. Because of this, electrical measurements on those samples with a well defined silicon layer were difficult to interpret. This was due to the dominance of the electrical properties by the large number of grain boundaries which are known to produce electrical discontinuities. Auger depth profiles were performed in our Surface Analysis Laboratory to investigate the extent of the metal-silicon interdiffusion and their reaction to form silicides. These data were taken using 2.5 KeV Argon ions to sputter etch the surface. Removal rates

varied from 10 to 50 Å/min. We attained a measured 2% depth resolution by rastering the ion beam over a 4 mm² square area.

(a) Tungsten Substrates. Seven tungsten substrates labelled 61-1, 61-2, 61-3, 87-1, 87-2, 87-3, and 107-3, were investigated and some of the experimental parameters for each experiment are shown in Table I.

The three substrates used in the 61 series were prepared in the time sequence, 61-3, 61-1, and 61-2. The in situ Auger results during the various stages of sample preparation for experiment 61 are given in Table IV in Appendix B. All three substrates were mostly covered with carbon after initial loading. During the bakeout of the vacuum chamber some of the carbon was removed while the oxygen coverage increased. Before any of the substrates were annealed at high temperatures, the silicon ingot was outgassed for several days using the 6 kW electron beam gun. Even though the open space above the silicon was covered at all times with substrate #4, Auger spectra taken after the first high temperature anneal of each substrate showed the presence of silicon on the surface ranging from 14.6 at% for 61-3 to 24.5 at% for 61-2. After the first anneal, which was for one hour at 1000°C, all three substrates were found to be relatively free of carbon and oxygen. The major contaminant was silicon which was found at the 15 to 25% level.

Substrate 61-3 was held at 750°C and silicon was deposited for six minutes at a rate of 420 Å/min and at a pressure of $\sim 3 \times 10^{-10}$ torr (N₂). The Auger spectra after this deposition showed a significant concentration of tungsten on the surface (27 at%). This indicated significant interdiffusion of the silicon and tungsten. Three subsequent anneals for ten minutes each at 750°C produced an increased surface concentration of tungsten indicating further silicon-tungsten

TABLE I. Experimental Conditions for the Seven Tungsten Substrates.

The silicon deposition temperature is T_D , R_D is the silicon deposition rate, t_D is the deposition time, and D is the thickness of the silicon deposit.

SAMPLE NUMBER	T_D (°C)	R_D (Å/min)	t_D (hours)	D (microns)	SILICIDE* FORMATION
61-1 ⁺	625	100	0.5	0.3	W_5Si_3
61-2	640-670	40	8	2.0	WSi_2
61-3 ⁺	750	420	0.1	0.25	WSi_2
87-1	600	80	3.0	1.5	WSi_2 (weak)
87-2	780	420	1.0	2.5	WSi_2
87-3	* 615-640	630	0.25	1.0	n.d. **
107-3	400	20	24	2.9	n.d.

* By X-Ray Analysis.

⁺ These samples were annealed three times for ten minutes at T_D after silicon was deposited on the substrate.

** None detected.

interdiffusion. On the basis of the results found for 61-3, the T_D for the next substrate, 61-1, was lowered to 625°C. Silicon was deposited on this substrate for 30 minutes at a rate of 100 Å/min at 5×10^{-10} torr (N_2). No tungsten was found at the surface of the silicon layer, even after three successive ten minute anneals at 625°C. For the third sample, 61-2, the T_D was raised slightly to 640-670°C and the R_D was lowered to 40 Å/min to encourage grain growth in the silicon. The pressure during this deposition was 6×10^{-9} torr (N_2). The silicon deposition continued for eight hours to provide a two micron silicon layer for possible electrical characterization. However, subsequent Auger analysis indicated 21 at.%tungsten on the surface of the deposit indicating either T_D was too high or R_D was too low.

After removal from the vacuum chamber, SEM photographs were taken and Auger depth profiles were measured. These results for samples 61-1, 61-2 and 61-3 are shown in Figures 6, 7 and 8 respectively. The number of hillocks observed in the SEM pictures increases as the surface tungsten to silicon ratio increases. These features probably represent the formation of a tungsten silicide phase. The X-ray analysis of 61-2 and 61-3 revealed the presence of WSi_2 while sample 61-1 showed the presence of W_5Si_3 . The AES depth profile of 61-1 (Fig. 6b) shows several features. First, there is a small but significant diffusion of tungsten into the silicon layer. Second, the interface width is much larger than that expected from our depth resolution of 2%, indicating that the interface width is approximately 1000 Å. It is assumed that the interface is ~ 3000 Å below the silicon surface, since a deposition rate of 100 Å/min for 30 minutes was used on this sample. Third, oxygen is present at the interface at a level of approximately 1%. Fourth, the increase in the carbon signal is due to the higher absorption rate of carbon bearing gases (CH_4 and CO) on the sputter cleaned metal substrate

61-1

a)

Figure 6. SEM and Auger profile of sample 61-1. a) SEM picture of the surface taken at 4350 X showing the presence of hillocks covering approximately ten percent of the surface. b) Auger depth profile showing the relatively broadened interface region containing ~ 1 at.% oxygen.

Figure 7. SEM and Auger profile of sample 61-2. a) SEM picture showing $\sim 70\%$ of the surface covered by the so-called hillock structure. b) The Auger depth profile of sample 61-2 shows that extensive reaction and inter-diffusion of the silicon and the tungsten substrate had occurred.

Figure 8. SEM and Auger profile of sample 61-3. a) SEM picture showing the surface covered with structural features several microns wide. b) The Auger depth profile of sample 61-3 shows severe silicon-tungsten reaction and interdiffusion as was found in sample 61-2.

and does not represent a actual increase in carbon below the interface. This effect was easily verified. When the ion beam current was increased the carbon signal was decreased. The AES profiles for 61-2 and 61-3 are in Figures 7b and 8b respectively. These samples exhibit somewhat similar behavior in that the tungsten to silicon ratio is low during the first few hundred angstroms and then rises to a nearly constant level until the tungsten substrate is reached. No clearly defined interface exists for these two samples confirming the strong silicon-tungsten interaction. The X-ray and electron channeling measurements indicated that the film structure was cryptocrystalline. Therefore, the hillock structure cannot be ascribed to single crystal silicon grains.

Samples in Experiment 87 were prepared in the time sequence, 87-2, 87-1, and 87-3. The in situ AES results are given in Table V of Appendix B. After bakeout of the vacuum chamber, the surface of these substrates contained more oxygen but less carbon than was found in the 61 series substrates. Potassium, sodium, and nitrogen were found as minor contaminants. An Auger spectra typical of the 87 series substrates after bakeout but before any high temperature anneals is shown in Figure 9 for substrate 87-1. The same substrate after the final anneal, but before silicon deposition, is illustrated in Figure 10 and shows no carbon (<1 at.%) but does show much higher oxygen (7 to 14 at.%) than the 61 series substrates. This level of oxygen corresponds to less than one-half a monolayer even if it is all concentrated in the top monolayer. It is evident from Tables IV and V of Appendix B that another major difference between the 61 and 87 series is the total absence of silicon in the 87 series of substrates just before silicon deposition.

Sample 87-2 was an attempt to verify the results found for sample 61-3. The values for T_D and R_D were nearly identical for 87-2 and 61-3 but the time of deposition was ten times longer for 87-2. It is known that silicide formation is a diffusion limited process so that the rate of formation is proportional to the square root of time.⁽²⁰⁾ Figure 11 shows that after the silicon layer was deposited

Figure 9. Auger spectrum of sample 87-1 after the bakeout of the vacuum chamber but before any high temperature annealing. From the relative peak-to-peak heights of carbon, oxygen, and tungsten it is estimated that several monolayer of carbon and oxygen cover the tungsten substrate.

Figure 10. Two Auger spectra of sample 87-1. Spectrum (a) shows the surface after an anneal at 800°C for ten minutes. Compared to Figure 9 most of the carbon, sodium, potassium, nitrogen and oxygen have been removed but the calcium level increased. The total coverage is less than a monolayer. Spectrum (b) after a subsequent 1000°C anneal for two hours and was identical to spectrum (a) except that no carbon could be detected.

Figure 11. Auger trace of sample 87-2 after silicon deposition at 780°C for one hour showing the presence of tungsten at approximately 3.5 at.%.

only 3.5 at.% tungsten was found on the surface compared to 36 at.% for 61-3. A silicon deposit was then placed on sample 87-1 using T_D equal to 600°C for 3 hours, with R_D at 80 Å/min and a pressure of $\sim 3 \times 10^{-8}$ torr (N_2). As shown in Figure 12, no tungsten was detected by Auger analysis even at the highest gain. It is estimated that less than 0.25 at.% tungsten was present on the surface. Sample 87-3 was to have been an attempt to produce a 10 μ m thick layer of silicon for device characterization. A zirconium shield was used around the silicon ingot in experiment 87 only, and when a higher electron beam power was used a hole was melted in the zirconium shield after about 15 minutes, forcing the termination of the experiment. The pressure in the chamber during this deposition was $\sim 6 \times 10^{-8}$ torr (N_2) and the hydrogen partial pressure accounted for 98% of the residual gases present. Auger analysis revealed a large concentration of zirconium, oxygen, manganese, and silicon on the surface.

The SEM photographs of the 87-1 and 87-2 samples are shown in Figure 13a and Figure 14a, respectively. Sample 87-1 has a relatively smooth silicon deposit with features of ~ 3000 Å. Sample 87-2, deposited at a much higher temperature, has features five microns in size. The Auger depth profiles of the three 87 samples are given in Figures 13b, 14b, and 15. The interface region for sample 87-1, shown in Figure 13b, is twice the width expected from a 2% depth resolution indicating some interdiffusion. Weak WSi_2 lines were found by X-ray analysis. Figure 14b shows the gradual increase in tungsten with depth for sample 87-2. The sharp interface for 87-3 in Figure 15 is essentially depth resolution limited. Although prepared at essentially the same temperature as sample 61-1, sample 87-3 has a narrower interface probably due to the shorter time at the deposition temperature. It is important to note that all three 87 samples were cryptocrystalline although the morphology of sample 87-2 is considerably different.

Figure 12. Auger spectra of sample 87-1 following silicon deposition at 600°C for three hours. Only silicon transitions at 92, 105, and 145 eV are evident even at very high gain.

Figure 13. SEM and Auger profile of sample 87-1. a) SEM picture after silicon deposition at 600°C for three hours. The small features are ~ 3000 Å across and may represent silicon grains. b) Auger depth profile near the silicon-tungsten interface. The measured interface width is larger than the depth resolution indicating some interdiffusion of the silicon and tungsten phases.

Figure 14. SEM and Auger profile of sample 87-2. a) SEM picture after silicon deposition at 780°C for one hour. The 3 to 5 μm features are not single grains of silicon as indicated by X-ray and electron channeling. b) AES depth profile of sample 87-2 through the first micron showing the interdiffusion of tungsten and silicon.

Figure 15. Auger depth profile of sample 87-3. Silicon deposition at 615 to 640°C for 15 minutes at 630 Å/min. Most of the interface width arises from the depth resolution and little or no interdiffusion is evident. No carbon or oxygen were detected at the interface.

The final tungsten substrate 107-3 was studied in Experiment 107. On this substrate, a 2.9 μm thick silicon layer was deposited at 400°C at a deposition rate of only 20 $\text{\AA}/\text{min}$. An Auger spectrum taken just before silicon deposition and after annealing at 1050°C for three hours gave a surface oxygen concentration of 12 at.%. This concentration is comparable to that found for the 87 series substrates. An SEM picture at 23,000 X is shown in Figure 16a, and the silicon-tungsten interface is shown in Figure 16b. The dark lines in Figure 16a corresponds exactly to the grain boundary structure of the tungsten substrate. The interface width is depth resolution limited and is probably much smaller than the apparent width of 600 $\text{\AA}$. X-ray and electron channeling results indicate that the silicon layer is not amorphous but cryptocrystalline. It is therefore possible that the 600 $\text{\AA}$ features visible in Figure 16a represent the silicon grains.

(b) Tantalum Substrate. The tantalum substrate was numbered 107-2. Taking advantage of our experience with tungsten, silicon was deposited at several temperatures and the surface concentration of tantalum was monitored after each deposition. Table VI of Appendix B contains the in situ Auger results. A R_D of 750 $\text{\AA}/\text{min}$ for each deposition was used. Just before the initial silicon deposition, the tantalum surface contained 0.4 at.% carbon, 11.1 at.% oxygen, and 11.3 at.% sulphur and no silicon as shown in Figure 17a. The sulphur probably diffused from the bulk since no sulphur was found on the substrate before the high temperature anneal. After a ten minute silicon deposition at 750°C, the surface showed a tantalum concentration of 29.4 at.% as illustrated in Figure 17b. This level of surface concentration of tantalum is probably due to the formation of TaSi_2 , since this

Figure 16. SEM and Auger profile of sample 107-3. a) SEM picture after silicon was deposited at 400°C for 24 hours at a rate of only 20 Å/min. The ~ 600 Å features may represent the silicon grains. b) Auger depth profile of sample 107-3 near the interface region which lies 2.9 μm below the top of the silicon layer. The observed width is depth resolution limited.

Figure 17. Auger spectra of sample 107-2 during various stages of sample preparation. a) Trace taken after annealing the substrate at 1080°C for three hours showing oxygen and sulphur contaminants. b) Trace after ten minute silicon deposition at 750°C. The presence of 30 at.% tantalum indicates that at the surface complete reaction to TaSi_2 had occurred. c) Trace taken after final two hour silicon deposition at 550°C. No tantalum was observed on the surface.

phase was detected by X-ray analysis. A ten minute anneal of the sample at 750°C produced a slight increase in tantalum at the surface (32.9 at.%). Successive ten minute depositions of silicon were made at 700, 650, 600 and 550°C. Between each deposition the sample surface was returned to TaSi₂ by annealing at 750°C for ten minutes. The tantalum and silicon concentrations after each deposition are given in Table II. From these data it is clear that to avoid significant interdiffusion of tantalum and silicon the T_D must be below 600°C. A one micron thick silicon layer was deposited over the TaSi₂ at 550°C and no tantalum was found on the sample surface. This result is shown in Figure 17c. Our detectable limit for tantalum was 0.15 at.%.

After removal from the vacuum chamber a SEM photograph of sample 107-2 was taken and is shown in Figure 18a. The surface has features of $\sim 3000 \text{ \AA}$. The AES depth profile (Fig. 18b) had two distinct interface regions corresponding to the silicon-TaSi₂ and the TaSi₂-tantalum phase boundaries. At both interfaces the measured width is somewhat larger than that expected assuming a 2% depth resolution. The concentration gradient at both phase boundaries existed over a distance of several hundred Angstroms. An edge view of the sample, Figure 18c, clearly shows the three phases present. The silicon was determined to be cryptocrystalline.

(c) Hafnium Substrate. Several layers of silicon were also deposited on the hafnium substrate 107-1 at various temperatures. Table VI of Appendix B has the relevant in situ Auger results. It is interesting to note that after the first anneal and just before the first silicon deposit, a 13.5 at% zirconium concentration was found on the surface. As can be seen from Table III of Appendix A, the hafnium starting material contained a bulk concentration of 3.9 at.%

TABLE II. Surface Concentrations of Tantalum and Silicon for Sample 107-2.

These concentrations are shown as a function of deposition temperature and were calculated from the Auger peak-to-peak heights.

T_D (°C)	750	700	650	600	550	550
Ta (at.%)	29.4	27.8	12.3	4.1	1.6	<0.15
Si (at.%)	70.6	72.2	87.7	96.9	98.4	>99.85

Figure 18. SEM, Auger depth profile and optical micrograph of sample 107-2. a) SEM picture taken after two hour silicon deposition at 550°C . The small features are $\sim 1000\ \text{\AA}$ and may represent the grain size of the silicon. b) Auger depth profile near the silicon-TaSi₂ and TaSi₂-tantalum interfaces. c) Edge view showing the silicon and TaSi₂ layers and the tantalum substrate (2000 X).

zirconium. Apparently, the anneal at 1050°C for three hours produced a surface enhancement of the zirconium. All silicon depositions were made at a R_D of 750 Å/min and a pressure of $\sim 5 \times 10^{-9}$ torr (N_2). After the first silicon layer was deposited at 600°C, 39 at.% hafnium was found at the surface of the sample. Following a ten minute anneal at 600°C, the hafnium concentration increased to 53 at.% indicating that hafnium-silicon reactions were occurring at 600°C. The second silicon deposit at 500°C yielded a clean silicon surface (< 0.3 at.% hafnium). Subsequent annealing for ten minutes at 600°C still did not produce any measureable hafnium at the surface. However, a second anneal at 600°C for 30 minutes, produced a hafnium concentration of 6 at.%. A one micron silicon layer was deposited over a two hour period at 600°C and no hafnium was detected.

When the sample was removed from the vacuum chamber, the surface exhibited severe buckling with parts of the top layer detached from the hafnium substrate as shown in Figure 19a. Unfortunately, it could not be determined exactly when the buckling occurred. An Auger depth profile of the raised portion of the sample indicated some hafnium metal in the raised film. This indicates that the large stresses responsible for the effect may have come from the hafnium-silicon interdiffused layer. Areas measuring as large as 50 μm were observed by SEM (Fig. 19b) that were in perfect registry with the partially recrystallized hafnium substrate. At first observation these appeared to be grains of silicon. However, the silicon layer was determined to be cryptocrystalline by X-ray and electron channeling analysis so that the 50 μm structure does not represent the size of the silicon grains. This result shows very clearly that SEM photographs alone can not be used to accurately reveal the grain structure of silicon layers. In Figure 19c, the high magnification SEM picture has some structural features whose size is ~ 3000 Å. We believe that this structure represents the actual grain size of the silicon layer.

107-1

a)

b)

c)

Figure 19. Optical and SEM micrographs of sample 107-1 after final silicon deposition. a) Optical picture showing the buckling of the silicon rich overlayer. b) SEM picture showing 50 μm features which correspond to the hafnium substrate grains. c) SEM photograph of a raised portion of sample. The ~ 3000 Å features may be actual grains of silicon.

Recently, extensive studies have been performed on metallization processes for silicon technology utilizing Rutherford backscattering spectroscopy (RBS).⁽²¹⁾ These studies often involved the determination of the kinetics of metal-silicon reactions to form various silicide phases. Nearly all of these investigations involved thin metal films sputtered onto single crystal silicon having a native oxide layer. This layer, which is typically $20 \text{ \AA}$ thick and cannot be detected by RBS, has been suspected as the source of irreproducible growth in the initial stages of the metal-silicon interaction. After the oxide layer has been penetrated, diffusion controlled kinetics are almost always observed during the growth of the silicide layer. The samples in the present study were metal substrates with less than one-half a monolayer of contaminants onto which, silicon was vapor deposited. It is therefore of interest to compare the rates of silicide reaction for the two fabrication techniques.

Examination of Auger depth profiles of the seven tungsten substrates show that none have a well defined silicide layer except sample 61-3 which has both a silicide and an interdiffusion region. This means that it was very difficult to compare silicide reaction rates of our samples to those previously reported for the tungsten-silicon system.⁽²²⁾

We could find no data on the kinetics of tantalum silicide formation but our results indicate that the first phase to form is TaSi_2 . Christou and Day⁽²³⁾ have investigated interdiffusion in silicon-tantalum thin film structures. They found by Auger depth profiles, that after 24 hours at 650°C no TaSi_2 was formed but interdiffusion over $\sim 1000 \text{ \AA}$ had occurred. Our data for sample 107-2 showed that, after silicon deposition at 650°C for ten minutes at a rate of $750 \text{ \AA}/\text{min}$, 12.3 at.% tantalum was present at the surface. Thus for the same temperature, our results indicate interdiffusion rates nearly 1000 times that found by Christou and Day.

Ziegler et al⁽²⁴⁾ have investigated the kinetics of hafnium-silicon reactions and found that HfSi_2 forms first followed by HfSi . At 600°C , they found that a 500 Å layer of HfSi_2 forms in only ten minutes. Our sample 107-1, after deposition of an equivalent 7500 Å silicon layer at 600°C for ten minutes, gave a surface composition very close to HfSi_2 . Our reaction rate is about an order of magnitude faster than that observed by Ziegler et al. when a native oxide layer is present.

Our data for tantalum and hafnium strongly suggest that cleaned metal-silicon interfaces give rise to much faster silicon-metal reaction rates than those found when a native SiO_2 layer is thought to be present between the metal and silicon phases.

SUMMARY AND CONCLUSIONS

Thin films of high purity silicon were electron beam vaporized onto cleaned substrates of tungsten, tantalum and hafnium. Deposition was done at pressures of $\sim 1 \times 10^{-10}$ torr (N_2). Most of the residual gas pressure was due to hydrogen that was emitted from the silicon ingots, even though they were degassed by prolonged heating in vacuum at elevated temperatures. The partial pressures of 12 other gases were quite low as determined by residual gas analysis. The substrates were resistively heated prior to the deposition of silicon to clean their surfaces and to complete chemical reactions. During this step, significant increases in the partial pressure of H_2 , N_2 , CO , CO_2 and CH_4 were noted.

In our deposition experiments, the substrate temperature was varied between 400 and 750°C and deposition rates between 20 and 750 Å/min were used. In situ Auger electron spectrometry was used as the major diagnostic tool for determining surface contamination and metal diffusion. Subsequent characterization studies

were made of each specimen using Auger depth profiling, X-ray analysis and scanning electron microscopy to determine silicon-metal boundary layers, interdiffusion profiles, silicide formation and the grain size of the silicon deposits.

At the beginning of this study, it was believed that the grain size of the silicon as deposited on cleaned metal substrates could be enhanced by performing the deposition in a near non-contaminating environment. Using tungsten substrates with a grain size of three microns, we were unable to grow silicon films with large grain structure. The grain size obtained is believed to be cryptocrystalline in nature and measures 600 to 3000 Å. At higher temperatures, where larger grain structure might be expected, silicide reactions occur and diffuse interfaces are formed at rates faster than the silicon could be deposited (750 Å/min). Similar results were obtained for tantalum substrates having a grain size of 30 to 50 microns and for hafnium substrates having grains measuring 40 to 60 microns. It is interesting to note that all of the deposits gave sharp X-ray lines indicating crystallinity.

No silicide was formed on the tungsten substrate that was heated to only 400°C during deposition. A sharp interface between the silicon layer and the tungsten was observed by depth profiling. The silicon was cryptocrystalline, measuring ~ 600 Å, and clear diode behavior was not observed because of the large number of grain boundaries present.

Comparison of our data for tantalum and hafnium strongly suggest that metal surfaces cleaned in a near non-contaminating environment give rise to much faster silicon-metal reaction rates than those formed when an interface layer such as SiO_2 is present.

RECOMMENDATIONS

Further work on backwall Schottky barrier solar cells depends on the existence of a temperature "window" in which large grain silicon growth can occur and in which metal-silicon interdiffusion is minimized. In our experiments, we did not find a window for the metals: tungsten, tantalum or hafnium. However, there are other experimental approaches that should be made in an effort to enhance the grain growth and to suppress the metal-silicon interaction. First, to enhance the grain growth of silicon the substrate should have large grain size and a good match should exist between the lattice parameters and the thermal expansion of the silicon and the metal substrate. This could be accomplished by first forming a large grain metal silicide at high temperature followed by silicon deposition at a lower temperature in an effort to enhance epitaxial growth. Second, to suppress the metal-silicon interaction, substrates other than tungsten, tantalum and hafnium should be studied with respect to their silicide formation rate.

Another approach to the suppression of the metal-silicon interaction could be to use a diffusion barrier. One technique would be to take advantage of the decreasing rate of silicide growth with thickness until the growth rate of the reaction front is slower than the deposition rate. This configuration would consist of a metal-silicide-silicon sandwich with the Schottky barrier at the silicide-silicon interface. A second approach would be to use oxide or nitride layers that are stable with respect to the silicide reaction. This latter configuration would form the basis for a metal-insulator-silicon Schottky barrier solar cell.

We believe these experiments should be performed under a near non-contaminating environment using as many in situ diagnostic tools as practical. In this manner, a true evaluation of the feasibility of fabricating Schottky barrier solar cells under a space environment would be accomplished.

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APPENDIX A

TABLE III. Chemical Analysis of Silicon Ingots
and Metal Substrates,

Element	Concentration, ppm atomic				
	Silicon Ingot I	Silicon Ingot II	Hafnium	Tantalum	Tungsten
Al	0.04	<0.3	<0.5	<0.6	10
Ag	<0.005	<0.1	<0.006	<0.06	<0.006
Ar	<0.03	<0.3	<3	<0.6	<100
As	<0.04	<0.1	<0.02	<0.04	2
Au	<0.05	0.36*	0.2	<1	0.3
B	0.02	<0.05	0.02	0.8	0.06
Ba	<0.04	<0.2	<0.04	<0.2	<0.04
Be	<0.04	<0.08	<0.1	<0.01	<0.1
Bi	<0.03	<0.09	<0.04	<0.09	0.1
Br	<0.08	<0.2	<0.01	<0.1	<0.01
C	6 *	1 *	195(1)*	660(1)*	15 (1)*
Ca	0.50*	0.4 *	2	<0.8	10
Cd	<0.04	<0.4	<0.1	<0.1	<0.01
Ce	<0.6	<0.4	<0.1	<0.3	<0.2
Cl	2 *	0.4 *	3 *	5 *	1
Co	<0.4	<1	<0.3	<0.1	3
Cr	<0.3	<0.07	<0.1	7 *	12
Cs	<0.003	<0.009	<0.004	<0.007	0.005
Cu	2 *	0.6 *	0.2	2	0.2
Dy	<0.4	<1	<0.3	<0.9	<0.4
Er	<0.5	<1	<0.5	<1	<0.6
Eu	<0.2	<0.4	<0.1	<0.3	<0.1
Fe	<0.6	<1	31 *	70 *	100 *
Ga	<0.03	<0.09	<0.03	<0.07	<0.03
Gd	<0.6	<1	<1	<2	<1
Ge	<0.1	<0.2	<0.04	<0.1	<0.04
H	-	-	<180(2)	<180(2)	<180(2)

	Silicon Ingot I	Silicon Ingot II	Hafnium	Tantalum	Tungsten
Hf	<0.6	<2	-	<4	6
Hg	<0.05	<0.2	<0.01	<0.1	<0.02
Ho	<0.2	<0.5	<0.1	<0.4	<0.3
I	<0.03	<0.09	<0.05	<0.07	0.07
In	<0.08	<0.1	<0.3	<0.06	<1
Ir	<0.2	<0.7	<1	<0.8	<0.4
K	4 *	<0.1	<0.02	10 *	20 *
La	<0.1	<0.4	<0.2	<0.3	<0.2
Li	<0.005	<0.01	<0.1	<0.01	0.06
Lu	<0.2	<0.8	<3	<1	<0.4
Mg	0.60 *	<0.05	<0.06	<0.4	<1
Mn	<0.07	<0.04	<0.04	<0.2	1
Mo	<1	<1	<1	<1	170 *
N	-	-	25(2)*	130(2)*	13(2)
Na	3 *	<1	100 *	9 *	<5
Nb	<3	<0.2	<4	<20	70 *
Nd	<0.6	<1	<0.4	<1	<0.5
Ni	<1	<0.4	<5	31 *	60 *
U	9 *	20 *	550(2)*	1075(2)*	185(2)*
Os	<0.4	<1	<3	<1	<0.2
P	<0.02	<0.3	0.8	1	300 *
Pb	<0.1	<0.4	<0.09	<0.3	<0.1
Pd	<0.09	<0.6	<0.2	<0.5	<0.3
Pr	<0.1	<0.4	<0.1	<0.2	<0.2
Pt	<0.3	<0.9	<1	<1	<0.3
Rb	<0.002	<1	<0.002	<0.2	<0.1
Re	<0.3	<1	<0.5	<0.8	<0.7
Rh	<0.07	<0.2	<0.09	<0.2	<0.08
Ru	<0.3	<0.6	<0.6	<0.6	<0.4
S	1 *	<0.03	6 *	1	<0.5

	Silicon Ingot I	Silicon Ingot II	Hafnium	Tantalum	Tungsten
Sb	<0.04	<0.1	<0.008	<0.08	0.1
Sc	<0.6	<0.1	200 *	<2	<0.4
Se	<0.05	<0.2	0.04	<0.1	<0.03
Si	-	-	90 *	110 *	30 *
Sm	<0.3	<0.8	<0.2	<0.7	<0.4
Sn	<0.03	<0.3	<0.03	<0.2	0.1
Sr	<0.2	<0.3	<0.1	<0.07	<0.1
Ta	<0.2	<0.8	<1	-	<2
Tb	<0.2	<0.5	<2	<0.3	<5
Te	<0.01	<0.2	<0.01	<0.1	<0.01
Ti	0.2	<0.04	<0.3	1	60 *
Tl	<0.04	<0.1	<0.1	<0.2	<0.01
Tm	<0.1	<0.5	<0.2	<0.3	<0.2
U	<0.1	<0.5	<0.2	<0.4	<0.2
V	<0.01	<0.05	0.3	<0.3	0.7
W	<1	<3	<1	<2	-
Y	<0.09	<0.2	<500	<0.5	<0.3
Yb	<0.2	<0.6	<0.4	<2	<0.3
Zn	1 *	<0.08	<0.1	0.8	0.1
Zr	<0.5	<0.6	39000 *	<1	<80

(1) Combustion chromatographic analysis.

(2) Vacuum fusion analysis.

* Ten highest detectable impurities.

APPENDIX B

All of the entries in Tables IV, V, and VI were calculated from the measured Auger peak-to-peak heights using the relative sensitivity factors derived from data in the Physical Electronics Handbook of Auger Electron Spectroscopy.⁺ These values were calculated assuming a homogeneous distribution of all elements within the top several monolayers. If I_i is the measured Auger peak-to-peak height of the i th element, then A_i , the unnormalized elemental concentration, is given by $A_i = \alpha_i I_i$, where the α_i are the relative sensitivity factors. We have used $\alpha_{Si} = 1.00$, $\alpha_W = 4.3$, $\alpha_{Ta} = 4.3$, $\alpha_{Hf} = 7.2$, $\alpha_O = 0.86$, $\alpha_C = 2.6$, $\alpha_S = 0.53$, $\alpha_P = 0.9$, $\alpha_K = 0.56$, $\alpha_{Ca} = 1.00$, $\alpha_N = 1.4$, and $\alpha_{Na} = 0.60$. The concentrations in atomic percent, denoted by C_i , are then obtained from

$$C_i = \frac{A_i}{\sum_j A_j} \times 100.$$

The Auger lines used were Si (92), W (155-179), Ta (155-183), Hf (153-180), O (510), C (272), S (152), P (112), K (242), Ca (280), N (385), and Na (990), where the number in parentheses is the energy in eV of the Auger transition. The values in Tables IV, V, and VI are to the nearest tenth of a percent and should only be used to determine changes in elemental compositions from one treatment to another. In no sense can these values be assumed to represent the absolute concentrations for a given sample, since they are probably only accurate to within twenty-five percent.

⁺ Handbook of Auger Electron Spectroscopy, 2nd Edition, Physical Electronics Industries, Inc., 1976, Eden Prairie, Minnesota.

TABLE IV. Auger Surface Concentrations for the Three 61 Series Samples after Each Step of the Sample Preparation.

SAMPLE NUMBER	TREATMENT - TEMP./TIME (°C/hours)	SILICON	TUNGSTEN	CARBON	OXYGEN	OTHER
61-1	Initial	-	-	99.9 ¹	0.1	-
	ABO ²	-	0.5	91.4	8.1	-
	AN ³ 1000/1.0	19.4	76.0	1.8	0.05	P=2.7
	AN 1050/1.0	15.5	79.3	1.0	0.1	P=4.1
	SD ⁴ 650/0.5	>99.6	<0.2	<0.2	<0.1	-
	AN 600/0.16	>99.6	<0.2	<0.1	<0.1	-
	AN 600/0.16	>99.6	<0.2	<0.1	<0.1	-
	AN 600/0.16	>99.6	<0.2	<0.1	<0.1	-
61-2	Initial	-	-	99.2	0.8	-
	ABO	-	24.5	61.2	14.3	-
	AN 1000/1.0	24.5	69.8	<0.8	<0.3	P=4.6
	AN 925/4.0	23.1	74.7	<0.6	-	P=1.6
	AN 1000/1.5	20.3	72.2	<1.9	<0.7	P=4.7
	SD 640-670/8.0	79.0	21.0	-	-	-
61-3	Initial	-	4.0	87.7	6.8	N=1.5
	ABO	-	45.9	33.2	18.1	N=2.8
	AN 650/2.0	14.6	66.3	12.2	5.0	N=1.9
	AN 1000/1.0	23.3	73.8	0.7	0.2	P=2.0
	AN 1020/1.0	26.1	68.4	0.7	<0.1	P=4.8
	SD 750/0.1	73.0	27.0	-	-	-
	AN 750/0.1	64.6	35.4	-	-	-
	AN 750/0.1	60.3	39.7	-	-	-
	AN 750/0.1	59.7	40.3	-	-	-

1. Concentrations are in atomic percent.
2. ABO = After bakeout of vacuum chamber.
3. AN = Anneal.
4. SD = Silicon deposition.

TABLE V. Auger Surface Concentrations for the Three 87 Series Samples after Each Step of the Sample Preparation

SAMPLE NUMBER	TREATMENT - TEMP./TIME (°C/hours)	SILICON	TUNGSTEN	CARBON	OXYGEN	OTHER
87-1	ABO ²	-	23.5 ¹	40.9	28.2	K=7.4
	AN ³ - 800/0.1	<0.4	84.9	2.0	9.8	Ca=1.0
	AN - 1000/2.0	<0.2	84.6	<1.0	14.2	-
	SD ⁴ - 600/3.0	>99.6	<0.4	-	-	-
87-2	ABO	-	23.9	38.7	31.8	K=2, Na=1.5, N=2
	AN - 400/0.16	-	30.9	33.0	32.0	K=1.1, Na=1.5, N=1.4
	AN - 1000/2.0	<0.1	75.7	<0.8	13.9	Ca=3.7, P=5.8
	SD - 780/1.0	96.6	3.4	-	-	-
87-3	ABO	-	18.6	40.1	36.7	K=1, Na=1, Ca=2
	AN - 800/0.16	-	79.1	1.5	17.6	Ca=1.8
	AN - 980/2.0	10.3	78.8	<0.6	5.6	P=4.3, Ca=0.4
	AN - 980/2.0	2.0	84.4	<0.9	7.0	P=4.8, Ca=0.9
	SD - 615-640/0.2					

1. Concentrations are in atomic percent.
2. ABO = After bakeout of vacuum chamber.
3. AN = Anneal.
4. SD = Silicon deposition.

TABLE VI. Auger Surface Concentrations for the Three 107 Series Samples after Each Step of the Sample Preparation.

SAMPLE NUMBER	TREATMENT - TEMP./TIME (°C/hours)	SILICON	METAL SUBSTRATE	CARBON	OXYGEN	OTHER
107-1 (Hf)	Initial	-	30.9 ¹	35.4	31.5	S=0.4 N=1.8
	AN ² - 1050/3.0	-	69.5	-	0.3	S=16.8 Zr=13.4
	Just Before SD ³	-	64.8	3.6	1.8	S=16.3 Zr=13.5
	SD - 600/0.16	56.0	39.2	-	-	S=4.8
	AN - 600/0.16	40.9	52.9	0.5	0.4	S=5.3
	SD - 500/0.16	>99.7	<0.3	-	-	-
	AN - 600/0.16	>99.5	<0.5	-	-	-
	AN - 600/0.5	94.0	6.0	-	-	-
	SD - 600/2.0	>99.7	<0.3	-	-	-
107-2 (Ta)	Initial	-	29.0	30.1	39.2	S=0.3 N=1.4
	AN - 1080/3.0	-	77.2	0.4	11.1	S=11.3
	SD - 750/0.16	70.6	29.4	-	-	-
	AN - 750/0.16	67.1	32.9	-	-	-
	SD - 700/0.16	72.2	27.8	-	-	-
	SD - 650/0.16	87.7	12.3	-	-	-
	AN - 750/0.16	69.6	30.4	-	-	-
	SD - 600/0.16	97.0	3.0	-	-	-
	AN - 750/0.16	70.1	29.9	-	-	-
	SD - 550/0.16	98.4	1.6	-	-	-
	AN - 750/0.16	69.8	30.2	-	-	-
	SD - 550/2.0	>99.8	<0.2	-	-	-
107-3 (W)	Initial	<0.2	39.7	29.9	26.4	K=2.4 Ca=1.4
	AN - 1050/3.0	<0.3	83.5	0.4	12.0	K<0.2 Ca=3.6
	SD - 400/24.0	>99.3	<0.1	<0.2	<0.3	S<0.1

1. Concentrations are in atomic percent.
2. AN = Anneal.
3. SD = Silicon deposition.

APPENDIX C

During Experiments 61, 87 and 107, the peak height of 13 residual gases contained in the space simulation chamber were continually monitored. From these values, their partial pressures were calculated⁺ at the following stages of the experiment; 1) after chamber bakeout at 380°C, 2) after the liquid nitrogen was added to the cryogenic panel, 3) during the resistive heating of the substrate, 4) during the silicon deposition and 5) after silicon deposition and the ingot was cooled. The data obtained are presented in the following Tables.

⁺ F. A. Schmidt, B. K. Lunde and D. E. Williams, Report No. IS-4125, Ames Laboratory-DOE, Ames, IA, January, 1977, p. 135.

TABLE VII. Partial Pressure of Residual Gases During Processing of Tungsten Specimen 61-1.

Gas	Partial Pressure, torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake-out	Cryogenics In	Resistive Heating (typical)	Silicon Deposition	After Deposition
H ₂	1.4×10^{-8}	2.7×10^{-9}	4.5×10^{-9}	2.5×10^{-10}	4.0×10^{-10}
He	1.1×10^{-10}	3.2×10^{-11}	4.0×10^{-10}	9.0×10^{-11}	4.9×10^{-12}
CH ₄	7.7×10^{-10}	9.8×10^{-13}	1.9×10^{-9}	1.7×10^{-12}	9.2×10^{-13}
H ₂ O	1.3×10^{-11}	1.3×10^{-12}	6.3×10^{-11}	7.0×10^{-13}	3.9×10^{-13}
Ne	4.9×10^{-11}	4.8×10^{-12}	1.8×10^{-10}	2.5×10^{-12}	1.2×10^{-12}
N ₂	2.2×10^{-9}	5.7×10^{-12}	3.7×10^{-9}	1.5×10^{-11}	3.8×10^{-12}
CO	1.9×10^{-9}	1.1×10^{-11}	1.4×10^{-9}	2.2×10^{-11}	8.6×10^{-12}
O ₂	1.2×10^{-11}	4.1×10^{-13}	2.5×10^{-11}	1.9×10^{-13}	7.2×10^{-14}
Ar	6.7×10^{-12}	1.6×10^{-13}	1.1×10^{-11}	4.8×10^{-14}	4.0×10^{-14}
CO ₂	7.6×10^{-11}	2.9×10^{-12}	2.5×10^{-10}	4.3×10^{-12}	6.5×10^{-13}
C ₂ H ₄	3.2×10^{-11}	3.5×10^{-13}	6.0×10^{-11}	3.0×10^{-13}	1.1×10^{-13}
C ₂ H ₆	3.5×10^{-12}	2.5×10^{-14}	3.9×10^{-11}	1.9×10^{-13}	5.5×10^{-14}
C ₃ H ₈	5.0×10^{-12}	2.5×10^{-14}	2.3×10^{-11}	3.8×10^{-13}	9.0×10^{-15}

TABLE VIII. Partial Pressure of Residual Gases During Processing of Tungsten Specimen 61-2.

Gas	Partial Pressure, torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake-out	Cryogenics In	Resistive Heating (typical)	Silicon Deposition	After Deposition
H ₂	1.4×10^{-8}	2.7×10^{-9}	4.5×10^{-9}	2.5×10^{-9}	4.0×10^{-10}
He	1.1×10^{-10}	3.2×10^{-11}	4.0×10^{-10}	2.8×10^{-11}	4.9×10^{-12}
CH ₄	7.7×10^{-10}	9.8×10^{-13}	1.9×10^{-9}	3.8×10^{-12}	9.2×10^{-13}
H ₂ O	1.3×10^{-11}	1.3×10^{-12}	6.3×10^{-11}	1.2×10^{-12}	3.9×10^{-13}
Ne	4.9×10^{-11}	4.8×10^{-12}	1.8×10^{-10}	6.0×10^{-12}	1.2×10^{-12}
N ₂	2.2×10^{-9}	5.7×10^{-12}	3.7×10^{-9}	9.3×10^{-12}	3.8×10^{-12}
CO	1.9×10^{-9}	1.1×10^{-11}	1.4×10^{-9}	2.4×10^{-11}	8.6×10^{-12}
O ₂	1.2×10^{-11}	4.1×10^{-13}	2.5×10^{-11}	2.8×10^{-13}	7.2×10^{-14}
Ar	6.7×10^{-12}	1.6×10^{-13}	1.1×10^{-11}	1.8×10^{-13}	4.0×10^{-14}
CO ₂	7.6×10^{-11}	2.9×10^{-12}	2.5×10^{-10}	3.7×10^{-12}	6.5×10^{-13}
C ₂ H ₄	3.2×10^{-11}	3.5×10^{-13}	6.0×10^{-11}	3.9×10^{-13}	1.1×10^{-13}
C ₂ H ₆	3.5×10^{-12}	2.5×10^{-14}	3.9×10^{-11}	5.3×10^{-14}	5.5×10^{-14}
C ₃ H ₈	5.0×10^{-12}	2.5×10^{-14}	2.3×10^{-11}	5.1×10^{-14}	9.0×10^{-14}

TABLE IX. Partial Pressure of Residual Gases During Processing of Tungsten Specimen 61-3.

Gas	Partial Pressure, torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake-out	Cryogenics In	Resistive Heating (typical)	Silicon Deposition	After Deposition
H ₂	1.4×10^{-8}	2.7×10^{-9}	4.5×10^{-9}	1.4×10^{-10}	4.0×10^{-10}
He	1.1×10^{-10}	3.2×10^{-11}	4.0×10^{-10}	1.1×10^{-10}	4.9×10^{-12}
CH ₄	7.7×10^{-10}	9.8×10^{-13}	1.9×10^{-9}	1.6×10^{-12}	9.2×10^{-13}
H ₂ O	1.3×10^{-11}	1.3×10^{-12}	6.3×10^{-11}	1.0×10^{-12}	3.9×10^{-13}
Ne	4.9×10^{-11}	4.8×10^{-12}	1.8×10^{-10}	5.5×10^{-12}	1.2×10^{-12}
N ₂	2.2×10^{-9}	5.7×10^{-12}	3.7×10^{-9}	1.1×10^{-11}	3.8×10^{-12}
CO	1.9×10^{-9}	1.1×10^{-11}	1.4×10^{-9}	2.1×10^{-11}	8.6×10^{-12}
O ₂	1.2×10^{-11}	4.1×10^{-13}	2.5×10^{-11}	3.8×10^{-13}	7.2×10^{-14}
Ar	6.7×10^{-12}	1.6×10^{-13}	1.1×10^{-11}	9.6×10^{-14}	4.0×10^{-14}
CO ₂	7.6×10^{-11}	2.9×10^{-12}	2.5×10^{-10}	4.8×10^{-12}	6.5×10^{-13}
C ₂ H ₄	3.2×10^{-11}	3.5×10^{-13}	6.0×10^{-11}	2.3×10^{-13}	1.1×10^{-13}
C ₂ H ₆	3.5×10^{-12}	2.5×10^{-14}	3.9×10^{-11}	1.9×10^{-13}	5.5×10^{-14}
C ₃ H ₈	5.0×10^{-12}	2.5×10^{-14}	2.3×10^{-11}	4.0×10^{-13}	9.0×10^{-14}

TABLE X. Partial Pressure of Residual Gases During Processing of Tungsten Specimen 87-1.

Gas	Partial Pressure, torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake-out	Cryogenics In	Resistive Heating (typical)	Silicon Deposition	After Deposition
H ₂	3.5×10^{-8}	1.6×10^{-11}	1.3×10^{-10}	1.6×10^{-8}	1.1×10^{-10}
He	1.1×10^{-10}	2.4×10^{-8}	3.2×10^{-11}	4.8×10^{-11}	4.9×10^{-12}
CH ₄	4.1×10^{-10}	3.7×10^{-13}	5.0×10^{-13}	1.8×10^{-12}	3.9×10^{-13}
H ₂ O	3.2×10^{-11}	9.7×10^{-13}	1.2×10^{-12}	1.1×10^{-12}	3.1×10^{-13}
Ne	5.1×10^{-9}	1.3×10^{-11}	1.5×10^{-11}	1.5×10^{-11}	8.2×10^{-12}
N ₂	1.1×10^{-9}	3.6×10^{-14}	2.0×10^{-12}	2.3×10^{-12}	2.6×10^{-12}
CO	1.1×10^{-9}	5.0×10^{-13}	5.5×10^{-12}	1.2×10^{-11}	6.4×10^{-12}
O ₂	7.0×10^{-12}	8.6×10^{-13}	7.9×10^{-13}	4.2×10^{-13}	4.9×10^{-15}
Ar	2.8×10^{-11}	8.3×10^{-15}	8.0×10^{-15}	4.2×10^{-13}	2.0×10^{-14}
CO ₂	4.1×10^{-11}	6.1×10^{-13}	1.2×10^{-12}	4.0×10^{-12}	4.0×10^{-13}
C ₂ H ₄	1.5×10^{-11}	2.8×10^{-13}	3.1×10^{-13}	3.3×10^{-13}	3.7×10^{-13}
C ₂ H ₆	1.8×10^{-11}	9.8×10^{-14}	1.1×10^{-13}	1.1×10^{-13}	1.2×10^{-13}
C ₃ H ₈	3.0×10^{-12}	8.5×10^{-14}	8.2×10^{-14}	5.1×10^{-14}	2.1×10^{-13}

TABLE XI. Partial Pressure of Residual Gases During Processing of Tungsten Specimen 87-2.

Gas	Partial Pressure, torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake out	Cryogenics In	Resistive Heating (typical)	Silicon Deposition	After Deposition
H ₂	3.5×10^{-8}	1.6×10^{-11}	1.3×10^{-10}	3.2×10^{-8}	1.1×10^{-10}
He	1.1×10^{-10}	2.4×10^{-8}	3.2×10^{-11}	1.1×10^{-10}	4.9×10^{-12}
CH ₄	4.1×10^{-10}	3.7×10^{-13}	5.0×10^{-13}	3.0×10^{-12}	3.9×10^{-13}
H ₂ O	3.2×10^{-11}	9.7×10^{-13}	1.2×10^{-12}	2.0×10^{-12}	3.1×10^{-13}
Ne	5.1×10^{-9}	1.3×10^{-11}	1.5×10^{-11}	2.8×10^{-11}	8.2×10^{-12}
N ₂	1.1×10^{-9}	3.6×10^{-14}	2.0×10^{-12}	1.7×10^{-12}	2.6×10^{-12}
CO	1.1×10^{-9}	5.0×10^{-13}	5.5×10^{-12}	3.1×10^{-11}	6.4×10^{-12}
O ₂	7.0×10^{-12}	8.6×10^{-13}	7.9×10^{-13}	5.4×10^{-13}	4.9×10^{-15}
Ar	2.8×10^{-11}	8.3×10^{-15}	8.0×10^{-15}	3.9×10^{-13}	2.0×10^{-14}
CO ₂	4.1×10^{-11}	6.1×10^{-13}	1.2×10^{-12}	6.1×10^{-12}	4.0×10^{-13}
C ₂ H ₄	1.5×10^{-11}	2.8×10^{-13}	3.1×10^{-13}	5.1×10^{-13}	3.7×10^{-13}
C ₂ H ₆	1.8×10^{-11}	9.8×10^{-14}	1.1×10^{-13}	1.4×10^{-13}	1.2×10^{-13}
C ₃ H ₈	3.0×10^{-12}	8.5×10^{-14}	8.2×10^{-14}	1.2×10^{-13}	2.1×10^{-13}

TABLE XII. Partial Pressure of Residual Gases During Processing of Tungsten Specimen 87-3.

Gas	Partial Pressure, torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake-out	Cryogenic In	Resistive Heating (typical)	Silicon Deposition	After Deposition
H ₂	3.5×10^{-8}	1.6×10^{-11}	1.3×10^{-10}	2.4×10^{-8}	1.1×10^{-10}
He	1.1×10^{-10}	2.4×10^{-8}	3.2×10^{-11}	8.0×10^{-11}	4.9×10^{-12}
CH ₄	4.1×10^{-10}	3.7×10^{-13}	5.0×10^{-13}	2.4×10^{-12}	3.9×10^{-13}
H ₂ O	3.2×10^{-11}	9.7×10^{-13}	1.2×10^{-12}	1.5×10^{-12}	3.1×10^{-13}
Ne	5.1×10^{-9}	1.3×10^{-11}	1.5×10^{-11}	2.2×10^{-11}	8.2×10^{-12}
N ₂	1.1×10^{-9}	3.6×10^{-14}	2.0×10^{-12}	2.0×10^{-12}	2.6×10^{-12}
CO	1.1×10^{-9}	5.0×10^{-13}	5.5×10^{-12}	2.1×10^{-11}	6.4×10^{-12}
O ₂	7.0×10^{-12}	8.6×10^{-13}	7.9×10^{-13}	4.8×10^{-13}	4.9×10^{-15}
Ar	2.8×10^{-11}	8.3×10^{-15}	8.0×10^{-15}	4.1×10^{-13}	2.0×10^{-14}
CO ₂	4.1×10^{-11}	6.1×10^{-13}	1.2×10^{-12}	5.1×10^{-12}	4.0×10^{-13}
C ₂ H ₄	1.5×10^{-11}	2.8×10^{-13}	3.1×10^{-13}	4.2×10^{-13}	3.7×10^{-13}
C ₂ H ₆	1.8×10^{-11}	9.8×10^{-14}	1.1×10^{-13}	1.2×10^{-13}	1.2×10^{-13}
C ₃ H ₈	3.0×10^{-12}	8.5×10^{-14}	8.2×10^{-14}	9.0×10^{-14}	2.1×10^{-13}

TABLE XIII. Partial Pressure of Residual Gases During Processing of Hafnium Specimen 107-1.

Gas	Partial Pressure torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake-out	Cryogenics In	Resistive Heating	Silicon Deposition	After Deposition
H ₂	3.3×10^{-9}	1.4×10^{-9}	6.8×10^{-11}	2.8×10^{-9}	2.0×10^{-10}
He	3.8×10^{-8}	1.2×10^{-10}	1.7×10^{-10}	2.0×10^{-10}	5.0×10^{-12}
CH ₄	5.0×10^{-11}	4.1×10^{-13}	8.0×10^{-13}	2.2×10^{-12}	6.1×10^{-13}
H ₂ O	4.1×10^{-12}	2.8×10^{-12}	7.1×10^{-12}	1.1×10^{-11}	3.5×10^{-13}
Ne	5.4×10^{-10}	9.4×10^{-12}	1.4×10^{-11}	2.9×10^{-11}	4.7×10^{-12}
N ₂	2.1×10^{-10}	1.1×10^{-11}	9.3×10^{-12}	2.7×10^{-11}	3.1×10^{-12}
CO	1.2×10^{-10}	2.0×10^{-11}	2.3×10^{-11}	5.0×10^{-11}	7.5×10^{-12}
O ₂	7.9×10^{-14}	1.5×10^{-13}	2.7×10^{-13}	6.5×10^{-13}	3.5×10^{-14}
Ar	7.8×10^{-12}	1.6×10^{-13}	1.1×10^{-12}	1.2×10^{-12}	3.0×10^{-14}
CO ₂	8.8×10^{-12}	2.4×10^{-12}	9.8×10^{-12}	7.9×10^{-12}	5.2×10^{-13}
C ₂ H ₄	2.6×10^{-12}	1.3×10^{-13}	2.2×10^{-13}	4.8×10^{-13}	2.5×10^{-13}
C ₂ H ₆	1.4×10^{-12}	4.2×10^{-14}	1.2×10^{-13}	2.3×10^{-13}	9.1×10^{-14}
C ₃ H ₈	3.8×10^{-13}	2.3×10^{-14}	1.9×10^{-14}	5.4×10^{-14}	5.1×10^{-14}

TABLE XIV. Partial Pressure of Residual Gases During Processing of Tantalum Specimen 107-2.

Gas	Partial Pressure, torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake-out	Cryogenics In	Resistive Heating	Silicon Deposition	After Deposition
H ₂	3.3×10^{-9}	1.4×10^{-9}	4.4×10^{-10}	2.7×10^{-7}	2.0×10^{-10}
He	3.8×10^{-8}	1.2×10^{-10}	4.1×10^{-10}	6.4×10^{-9}	5.0×10^{-12}
CH ₄	5.0×10^{-11}	4.1×10^{-13}	8.1×10^{-12}	5.6×10^{-12}	6.1×10^{-13}
H ₂ O	4.1×10^{-12}	2.8×10^{-12}	1.1×10^{-11}	1.8×10^{-11}	3.5×10^{-13}
Ne	5.4×10^{-10}	9.4×10^{-12}	3.4×10^{-11}	6.0×10^{-10}	4.7×10^{-12}
N ₂	2.1×10^{-10}	1.1×10^{-11}	2.4×10^{-13}	3.0×10^{-11}	3.1×10^{-12}
CO	1.2×10^{-10}	2.0×10^{-11}	4.1×10^{-11}	2.1×10^{-10}	7.5×10^{-12}
O ₂	7.9×10^{-14}	1.5×10^{-13}	2.7×10^{-13}	8.3×10^{-13}	3.5×10^{-14}
Ar	7.8×10^{-12}	1.6×10^{-13}	1.0×10^{-12}	2.1×10^{-12}	3.0×10^{-14}
CO ₂	8.8×10^{-12}	2.4×10^{-12}	1.9×10^{-11}	1.5×10^{-11}	5.2×10^{-13}
C ₂ H ₄	2.6×10^{-12}	1.3×10^{-13}	5.2×10^{-13}	6.9×10^{-13}	2.5×10^{-13}
C ₂ H ₆	1.4×10^{-12}	4.2×10^{-14}	4.2×10^{-13}	2.5×10^{-13}	9.1×10^{-14}
C ₃ H ₈	3.8×10^{-13}	2.3×10^{-14}	6.3×10^{-14}	1.3×10^{-12}	5.1×10^{-14}

TABLE XV. Partial Pressure of Residual Gases During Processing of Tungsten Specimen 107-3.

Gas	Partial Pressure, torr				
	(1)	(2)	(3)	(4)	(5)
	After Bake-out	Cryogenics In	Resistive Heating	Silicon Deposition	After Deposition
H ₂	3.3×10^{-9}	1.4×10^{-9}	3.6×10^{-8}	5.3×10^{-7}	2.0×10^{-10}
He	3.8×10^{-8}	1.2×10^{-10}	1.7×10^{-9}	1.3×10^{-8}	5.0×10^{-12}
CH ₄	5.0×10^{-11}	4.1×10^{-13}	4.3×10^{-12}	8.9×10^{-12}	6.1×10^{-13}
H ₂ O	4.1×10^{-12}	2.8×10^{-12}	4.1×10^{-11}	2.5×10^{-11}	3.5×10^{-13}
Ne	5.4×10^{-10}	9.4×10^{-12}	1.4×10^{-10}	1.2×10^{-9}	4.7×10^{-12}
N ₂	2.1×10^{-10}	1.1×10^{-11}	9.4×10^{-11}	3.3×10^{-11}	3.1×10^{-12}
CO	1.2×10^{-10}	2.0×10^{-11}	1.7×10^{-10}	3.7×10^{-10}	7.5×10^{-12}
O ₂	7.9×10^{-14}	1.5×10^{-13}	1.5×10^{-12}	1.0×10^{-12}	3.5×10^{-14}
Ar	7.8×10^{-12}	1.6×10^{-13}	1.1×10^{-12}	3.1×10^{-12}	3.0×10^{-14}
CO ₂	8.8×10^{-12}	2.4×10^{-12}	8.5×10^{-11}	2.3×10^{-11}	5.2×10^{-13}
C ₂ H ₄	2.6×10^{-12}	1.3×10^{-13}	1.3×10^{-12}	8.9×10^{-13}	2.5×10^{-13}
C ₂ H ₆	1.4×10^{-12}	4.2×10^{-14}	5.8×10^{-13}	2.8×10^{-13}	9.1×10^{-14}
C ₃ H ₈	3.8×10^{-13}	2.3×10^{-14}	2.0×10^{-13}	2.6×10^{-12}	5.1×10^{-14}