PHYSICAL REVIEW B VOLUME 57, NUMBER 11 15 MARCH 1998-I

Ballistic-electron-emission-microscopy studies on Au/SiO_2/n-type Si(100) and Ir/SiO_2/n-type Si(100) structures with very thin oxides

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(Received 5 November 1996)

Ballistic electron emission microscopy (BEEM) has been used to study metal-oxide-semiconductor (MOS) structures such as Au/SiO_2/n-type Si(100) and Ir/SiO_2/n-type Si(100), with the thin SiO_2 layer varying from 10 to 30 Å. As expected, the presence of a 30-Å oxide layer at the interface induces much higher electronic barriers than in the case of Schottky diodes. Samples with Au show a barrier height of $\sim$4.1 eV, while those with Ir have a barrier as high as 5.6 eV. When the interface oxide-layer thickness is reduced to $\sim$10 Å, the BEEM spectra behavior is different. For Au/SiO_2/Si junctions, although a clear threshold is always observed around 4 eV, showing that a high barrier is already formed at these small thicknesses, a leakage current is often detected below this threshold. In the case of Ir/SiO_2/Si structures, the BEEM spectra display an anomalous behavior. Below the high-energy threshold around 5.5–6 eV, a first threshold ($\sim$1.5 eV) and a broad peak ($\sim$4 eV) are observed. These structures could result from a resonant tunneling effect through the oxide layer. A striking result is also observed in the reverse BEEM (RBEEM) mode. In contrast to what is normally observed, where the RBEEM current is much weaker than the BEEM current, for these ultrathin barriers we find a broad peak at 4 eV with approximately the same intensity in RBEEM as in BEEM. [S0163-1829(98)06408-X]

I. INTRODUCTION

Metal-oxide-semiconductor (MOS) structures play a crucial role in modern semiconductor technology.[1] Due to the large band gap of SiO_2, the barrier formed at a metal/SiO_2/Si interface is much higher than the barrier obtained directly at a metal-semiconductor contact. The continuing miniaturization of semiconductor devices requires the use of thinner oxide layers and furthermore makes it increasingly important to control the formation of this oxide layer on a local scale. Oxide barriers of various kinds are also being used in numerous tunnel junction structures, where local control of the oxide is necessary. This need for a local study of oxide barriers is the main motivation for this paper.

Ballistic electron emission microscopy (BEEM) is a technique based on scanning tunneling microscopy (STM), proposed in 1988 by Kaiser and Bell[2] to study precisely and with high spatial resolution the electronic properties of a subsurface interface. In this technique, hot electrons are injected at a controlled energy from an STM tip into the thin metallic overlayer (*base* electrode) of a heterojunction to be studied. They can pass through this overlayer quasiballistically and thus probe the interface region. If they have sufficient energy, they can overcome the interface barrier and create a current in the semiconducting *collector* electrode. The threshold for nonzero current detection corresponds to the barrier height at the interface. Up to now, this technique has been applied mostly to the study of Schottky diodes,[3] where the interface barrier is quite low, around 1 eV or less, in the case of Si. No reports of BEEM measurements on MOS barriers have been made until recently, when Ludeke and co-workers[4–6] presented a BEEM study on (Pt or Pd)/SiO_2/n-type Si(100) heterostructures, where the oxide thickness ranged from 26 to 62 Å. Previously, Cuberes *et al.*[7] showed the ability to perform BEEM experiments on an insulating barrier in the Au/CaF_2/n-type Si(111) epitaxial system.

In this investigation, we focus on BEEM measurements on metal/SiO_2/Si structures with particularly thin SiO_2 layers (10–30 Å) in order to study how the barrier behavior changes as the oxide layer becomes extremely thin. We also report on studies using two different metals, Au and Ir, as base electrodes. Surprisingly, the BEEM results on these two MOS structures differ quite substantially. We first studied samples with Au and Ir deposited on a $\sim$30-Å-thick oxide layer. We investigated the effect of the SiO_2 layer at the interface, comparing barrier heights with those of a simple Schottky barrier, and studied the influence of the metal chosen for the base on the barrier height. We then studied the behavior of ultrathin barriers ($\sim$10 Å). In this case, the choice of Au or Ir as the base electrode yields strikingly different results.

II. EXPERIMENT

Substrates of n-type Si(100) (P doped to $\sim 10^{15}$ cm^{-3}), back implanted for Ohmic contacts and thermally preoxidized to 1300 Å of SiO_2, were etched in a HF(48%)/methanol 1:4 solution to prepare clean hydrogen-terminated surfaces,[8] rinsed in methanol, and introduced in the ultrahigh vacuum (UHV) system. Afterwards, all preparation steps and the BEEM measurements were performed *in situ*, except in the first few experiments with a Au base, where the samples were taken out for a short time to repair the connecting lead to the base. The subsequent measurements made totally *in situ* gave exactly the same results as for the cases where we had to take out the sample, and thus we conclude that the short exposure to air did not influence the results. The clean silicon surfaces were oxidized during 1 h under a pressure of 100 mbar O_2 (99.998% purity). This oxidation takes place at

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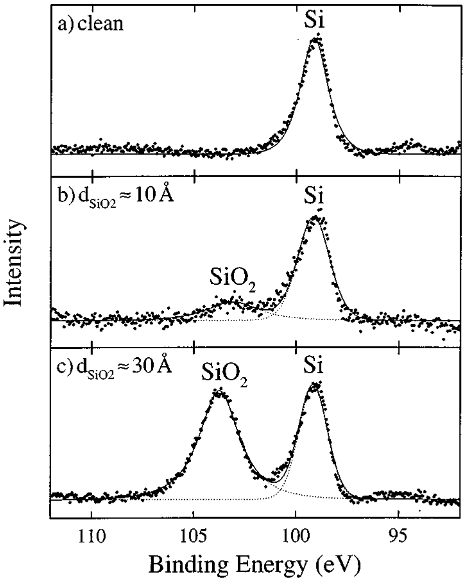

FIG. 1. XPS spectra of the Si $2p$ region of the Si substrate and the SiO$_2$ overlayer (a) before the oxidation, (b) after oxidation at 500 °C (*ultrathin* oxide layer: $d_{\mathrm{ox}}\approx 10$ Å), and (c) after oxidation at 650 °C (*thin* oxide layer: $d_{\mathrm{ox}}\approx 30$ Å). Solid lines are fits to mixed Gaussian-Lorentzian product functions, after the subtraction of a linear background; the components of each peak are given by the dotted lines.

low temperature (below 800 °C) and the oxidation temperature defines the oxide thickness,[9] which was estimated by x-ray photoelectron spectroscopy (XPS) measurements,[10,11] as can be seen in Fig. 1. Two groups of samples were prepared. The first one was oxidized at 650 °C, giving an oxide thickness of $d_{\mathrm{ox}}\approx 30$ Å, and the second one at 500 °C, with $d_{\mathrm{ox}}\approx 10$ Å; they are referred to as *thin* and *ultrathin* oxide layers, respectively. Details of the thickness estimate are given in Ref. 11. The base metal film (Au or Ir) was then deposited on the top of the SiO$_2$ at room temperature by dc magnetron sputtering through a shadow mask to define a junction area of ~ 0.29 mm^2. Gold films (50 or 100 Å thickness) were deposited at a pressure of 0.02 mbar Ar (99.9999% purity), at a rate of 5 Å/s, and iridium films (100 Å thickness) at 0.31 mbar Ar, at a rate of 1.1 Å/s. The base pressure in the chamber was 5×10^{-9} mbar and the deposition rate was determined with a quartz microbalance. The impedance of these MOS junctions was on the order of 1 MΩ or more, allowing low-noise BEEM measurements. For comparison, Au/n-type Si(100) Schottky diodes ($d_{\mathrm{Au}}=50$ Å) were also prepared with the same procedure, except for the oxidation step.

For the BEEM measurements, samples were transferred in UHV to the surface analysis STM chamber, with a base pressure in the low 10^{-11}-mbar range. The metal film was contacted to ground by gently pressing a gold wire onto it. We used a home-built UHV STM that was designed to place the tip above any point on a 1-cm^2 surface, using a scanning electron microscope to determine the position of the tip.[12] For these measurements Au or PtIr tips were used, with the results being independent of the tip material. BEEM spectra were obtained by measuring the collector current as a function of the tip bias, keeping the tunnel current constant. In order to reduce the noise in our spectra, for each measurement typically 20–60 spectra were accumulated and averaged. If charging effects[6,13] took place during these measurements, they would be hidden in the spectra, and therefore we cannot totally exclude that they could influence the effects reported in this paper. However, we never observed any direct indication that such charging effects are present. Also, we cannot exclude that the spectra represent a spatial average over distances of some nanometers due to thermal drift of the STM tip above the surface. But no significant changes in the shape of the spectra were observed during any given measurement. For all the data presented in this paper, we typically took averaged spectra on 20–50 different positions inside an area of about 200×200 nm^2. This operation was then repeated three or four times on very different areas over the sample surface about 10–100 μm apart. Tunnel currents I_t varied between 1 and 10 nA, and the tip bias was increased up to 10 V with both polarities. The high tip bias did not induce modifications in the shape of the spectra as a function of time, which might have been observed if the high voltage had damaged the surface. All measurements were done without biasing the MOS junctions, i.e., without modifying the barrier shape at the interface.

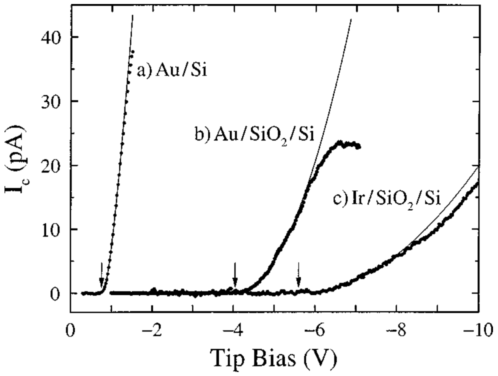

FIG. 2. (a) BEEM spectrum measured on a Au/Si sample without an oxide barrier, with $d_{\mathrm{Au}}=50$ Å and $I_t=1$ nA. The solid line is a fit to the Bell and Kaiser model giving $eV_b=0.76$ eV. (b) and (c) BEEM spectra measured on samples with a thin oxide barrier ($d_{\mathrm{ox}}\approx 30$ Å). The solid lines are fits to a quadratic law $I_C\propto(V-V_b)^2$. (b) Case of a Au base with $d_{\mathrm{Au}}=100$ Å and $I_t=10$ nA, giving $eV_b=4.0$ eV. (c) Case of an Ir base with $d_{\mathrm{Ir}}=100$ Å and $I_t=10$ nA, giving $eV_b=5.6$ eV. The arrows indicate the threshold position.

III. RESULTS AND DISCUSSION

A. Thin oxide layers (~30 Å)

Figures 2(a) and 2(b) show a comparison of BEEM spectra measured on samples with a Au base with and without an additional oxide barrier. As expected, the presence of an oxide layer induces a higher barrier at the interface, changing from $eV_b\approx 0.8$ eV, the values usually expected in the case of a Au/Si Schottky barrier,[2] to $eV_b\approx 4$ eV, for the Au/SiO$_2$/Si MOS junction. For voltages very close to the barrier height,[8,14] within a few tenths of a volt or even less, the BEEM current is expected to be dominated by a quadratic law, $I_C\propto(V-V_b)^2$. The range of validity of this approximation is too small to allow fitting, but we were able to use a quadratic law to extract the threshold position V_b from the spectra, as our data fit empirically to such a law on a wider range than expected by the theory. In the case of Au/SiO$_2$/Si junctions, the range chosen for the fit was $V_b \pm 1$ V, but changing it by ~ 0.5 V or even more on either side did not shift the threshold by more than a few hundredths of a volt. In the case of Ir/SiO$_2$/Si junctions, the fit could be easily made on a larger interval, and the range chosen was $V_b \pm 2$ V. Again changing it by ~ 0.5 V on either side did not shift the threshold by more than 0.2 V. These fitting variations of the threshold are thus less than the variations of V_b measured at different positions on the samples, expressed by the standard deviation for these values and indicated by the errors on V_b in the following results. Generally, the barrier heights measured on the Au/SiO$_2$/Si junctions are distributed around $eV_b \approx 4.1 \pm 0.2$ eV. The barrier height is very close to those obtained by other techniques probing macroscopic scales, as C-V characteristics or internal photoemission, on samples with much thicker oxide layers ($d_{\mathrm{ox}} = 500$ Å).[15,16] It is also in agreement with the values obtained in the recent BEEM study by Ludeke and co-workers[4–6] on the same kind of samples ($d_{\mathrm{ox}} = 26$–62 Å) with a Pt or a Pd base ($eV_b = 3.9$ eV). The spectrum measured on the Au/Si junction was fitted by the model proposed by Bell and Kaiser[17] for the Schottky diodes, which gives a very good description of the data for these kinds of samples.

As can be seen in Fig. 2(c), the BEEM spectra measured on MOS junctions with an Ir base show a threshold at higher energy, corresponding to a barrier height of $eV_b \approx 5.6 \pm 0.4$ eV. This barrier is very high and cannot be explained solely by the difference of work functions between gold and iridium. Charges present near the interfaces could be responsible for this enhancement, but it is interesting that it does not take place with gold. Such an explanation would imply that charges play a more important role in the Ir case. For both types of samples (Au and Ir), the collector current below the threshold is essentially zero, being lower than the current detection limit, of the order of some tenth of pA to 1 pA. On the other hand, the transmission threshold is always well defined. For measurements with a positive bias on the tip, in the reverse BEEM (RBEEM) mode,[18] for both types of samples (Au and Ir), the collector current is also zero, within the measurement noise limits, up to 10 V bias.

The surface topography of all these samples, seen by STM, is granular, with grains about 5–10 nm in diameter and a few nanometers in height. The collector current images, measured simultaneously at a tip bias higher than the barrier height at the interface, show a correlation with the topographic images. The collector current is more intense at the grain boundaries, where the metal film is probably thinner. This is illustrated in the case of an Ir base in Fig. 3. As the hot-electron inelastic mean free path in the metal is very short (a few nanometers) at high energies,[19,20] thickness variations of this order of magnitude are enough to induce intensity variations in the collector current. Other mechanisms may also be at work, since there is not a strict correlation over the whole image.

B. Ultrathin oxide layers (~10 Å)

The analysis of the samples with an ultrathin oxide layer of about $d_{\mathrm{ox}} \approx 10$ Å reveals a different behavior. Figure 4 shows some BEEM spectra measured at different places on various Au/SiO$_2$/Si samples with an ultrathin oxide layer. As for the thin oxides ($d_{\mathrm{ox}} \approx 30$ Å), these spectra reveal a clear threshold around 4 eV. Some spectra [Fig. 4(a)] are very similar to those measured on samples with a ~ 30 Å oxide layer, with no detectable current below the threshold. However, this current often increases gently before a sharp onset at the threshold [Figs. 4(b) and 4(d)]. This current contribution below the threshold can be associated with a small proportion of electrons crossing through the oxide barrier. However, it does not seem that it can be due to electron tunneling through the barrier, for this would induce an exponential increase of the current with the tip bias, as opposed to the observed linear dependence for the leakage current. One possible origin of this current is a hopping transport through the barrier, linked to impurities present in the oxide layer, although the exact process is not known. Leakage currents have also been observed by Ludeke, Bauer, and Cartier.[5] In their case, they appeared in certain defect regions possibly related to impurities embedded in the oxide. In our measurements, this leakage current is never very intense [spectrum (d) in Fig. 4 represents in some way an extreme case] and it is always much lower and more uniform than those reported by Ludeke and co-workers in their defect regions. It does

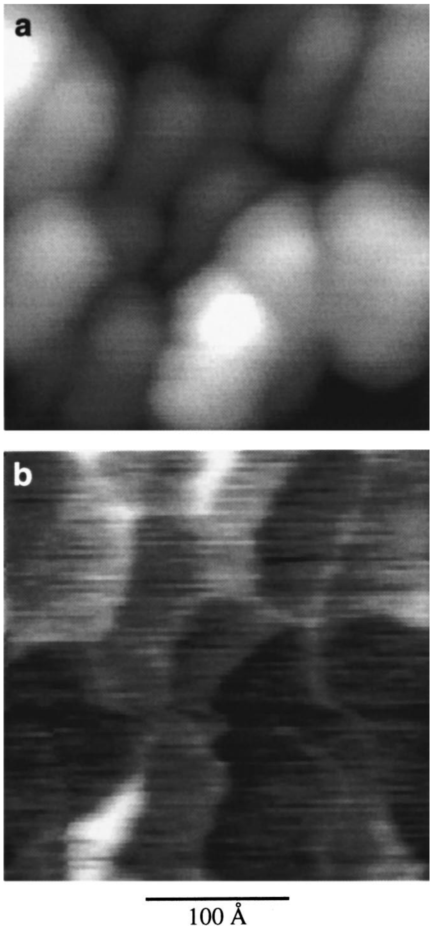

FIG. 3. (a) 300×300 Å^2 STM topographic image and (b) simultaneously measured collector current image (filtered image) for an Ir/SiO$_2$/Si sample ($d_{\mathrm{Ir}} = 100$ Å, $d_{\mathrm{ox}} \approx 30$ Å). Black-white ranges are 45 Å and 3.2 pA, respectively. $V_{\mathrm{tip}} = -8$ V, $I_t = 5$ nA.

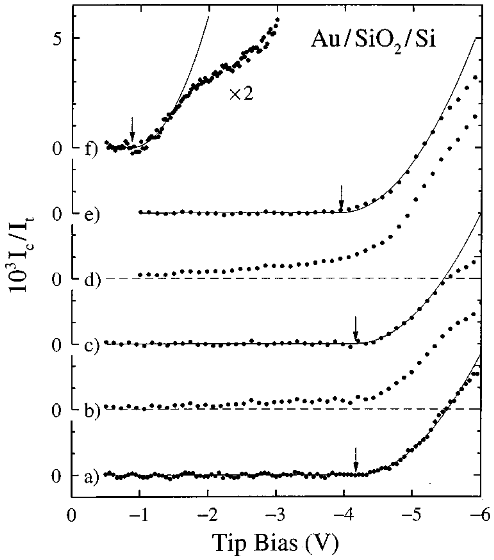

FIG. 4. BEEM spectra measured at different places on various Au/SiO$_2$/Si samples, with an ultrathin oxide layer ($d_{ox} \approx 10$ Å). The solid lines are fits to a quadratic law $I_C \propto (V-V_b)^2$. (a) $d_{Au} = 100$ Å, $I_t = 1$ nA, $eV_b = 4.2$ eV. (b) $d_{Au} = 100$ Å, $I_t = 1$ nA. (c) Same spectrum as (b), after subtraction of a linear current component, of 0.10 pA/V; $eV_b = 4.2$ eV. (d) $d_{Au} = 50$ Å, $I_t = 3$ nA. (e) Same spectrum as (d), after subtraction of a linear current component, of 0.67 pA/V; $eV_b = 4.0$ eV. (f) $d_{Au} = 100$ Å, $I_t = 1$ nA, $eV_b = 0.9$ eV. The arrows indicate the threshold position.

nonetheless vary from place to place on distances of a few tenths of a nanometer to a few nanometers, which could be simply due to variations in the base thickness, as discussed for the thin oxides, or to small variations in the oxide thickness or in its structure. In the RBEEM mode, with a positive bias on the tip, no detectable collector current is measured up to 10 V, although these data are more noisy.

Spectra (c) and (e) in Fig. 4 are obtained from spectra (b) and (d), respectively, after subtracting a linear component of the current, attributed to the leakage current. These corrected spectra look much like those measured in the case of the slightly thicker oxide layers (~ 30 Å), allowing an easier determination of the threshold for transmission above the barrier. A clear collector current threshold observation around 4 eV shows that for oxide layers as thin as 10 Å, a high barrier is already formed at the interface.

Sometimes, but very rarely, spectra indicating a low barrier around 0.9 eV were observed on one region [cf. Fig. 4(f)], whereas on other regions the threshold was at ~ 4 eV. Such a behavior could be associated with a local absence of the oxide layer, so that we observe directly the Au/Si Schottky barrier at the interface. We infer that this kind of region is very rare, since such spectra were seldom measured.

The behavior of the BEEM spectra measured on Ir/SiO$_2$/Si samples with an ultrathin oxide layer is very different from what is observed on the same kind of sample with a thicker oxide layer, or on samples with an ultrathin oxide layer and a gold base. Figure 5 shows some spectra measured at different places on Ir/SiO$_2$/Si samples with an ultrathin oxide layer of ~ 10 Å [Figs. 5(b)–5(e)], compared to a spectrum measured on the same kind of sample with a ~ 30-Å oxide layer [Fig. 5(a)]. These spectra are typical and could be observed with a high reproducibility at several places on various samples, using either Au or PtIr tips. Although the relative intensity of the structures varies from one spectrum to the other, all spectra present a first threshold around 1.5 eV, a broad peak around 4 eV, and a second threshold around 5.5–6 eV. When a positive bias is applied to the tip, the RBEEM current is of the same sign as the direct BEEM current and is very similar to it, both in shape and intensity. The absolute peak height varies from place to place, but its relative intensity between BEEM and RBEEM spectra is approximately constant. The striking fact is that the peak intensity in BEEM and RBEEM spectra have nearly the same value. The structures in the reverse BEEM and in the direct BEEM spectra appear exactly at the same energy. However, the current increase after the second threshold is much smaller in the RBEEM mode. The second threshold corresponds more to the end of the broad peak.

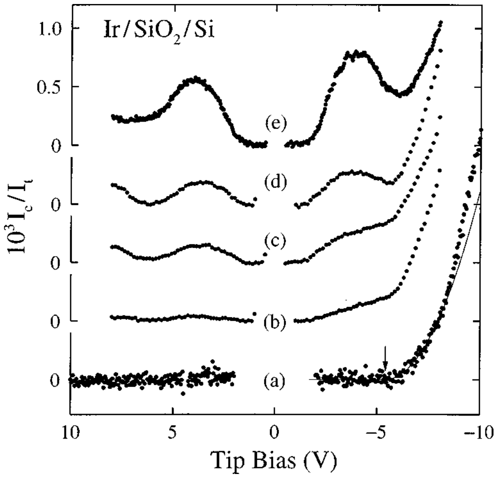

FIG. 5. BEEM and RBEEM spectra measured at different places on various Ir/SiO$_2$/Si samples ($d_{Ir} = 100$ Å). (a) Sample with a thin oxide layer ($d_{ox} \approx 30$ Å, $I_t = 10$ nA). The solid line is a fit to a quadratic law $I_C \propto (V-V_b)^2$ with $eV_b = 5.4$ eV, and the arrow indicates the threshold position. (b)–(e) Spectra on various samples with an ultrathin oxide layer ($d_{ox} \approx 10$ Å, $I_t = 5$ nA).

The second threshold (around 5.5–6 eV) is strikingly similar to the one observed with the thicker oxides. We therefore interpret this high-energy structure as the transport of the electrons above the SiO$_2$ barrier through the SiO$_2$ conduction band, since in fact it appears at the same energy as the threshold found for the thicker SiO$_2$ barriers ($eV_b \approx 5.6$ eV). Thus, even in these ultrathin films, the oxide barrier is clearly present.

As a result, if the broad peak corresponds to an energy below the oxide barrier height, we must find a mechanism allowing the electron to cross through the barrier. A simple tunnel effect cannot explain such a high intensity, compared to the current due to electrons passing above the barrier in the oxide conduction band above 5.5 eV. Such a mechanism could be a resonant tunneling effect linked to the presence of defect-related states in the SiO$_2$ energy gap. Ricco, Azbel, and Brodsky[21] put forward such a mechanism to explain oxide breakdown appearing at a bias lower than expected in MOS structures with $d_{ox} < 100$ Å. The presence of resonant states must be a consequence of the choice of an Ir base, considering that such structures do not appear with a Au base. They could be due to a chemical interaction of Ir with SiO_2 or they could arise from Ir diffusion through the oxide, especially since the oxide layer is very thin and disordered. The absence of such resonant states for Au films can be explained by the well-known low diffusivity of Au in SiO_2.[22]

Of course, one could be tempted to explain the first threshold, at low energy, as a result of the transport of the electrons above the Schottky barrier at the bare Ir/Si interface, where the oxide layer has disappeared. If this was the case, the structures above this threshold (broad peak and second threshold) would result from the electron transport above the barrier. Note, however, that these spectra are very different from the exceptional case discussed for a Au base [Fig. 4(f)]. In the case of an Ir base, the shape above the first threshold is very different, and furthermore these spectra are observed reproducibly everywhere on the surface of the junction. Two other facts lead us to reject this hypothesis. First, the low-energy threshold appears at ~ 1.5 eV, whereas the Schottky barrier height for Ir/n-type Si is 0.9 eV.[23] Second, for BEEM measurements on Schottky diodes, the RBEEM current near the threshold is always weaker at least by one order of magnitude than the direct BEEM current.[8,18] In the present measurements, the RBEEM current is of the same order of magnitude as the direct BEEM current.

An effect of dynamic charge trapping or detrapping in the oxide can also be ruled out, since these structures appear with the same shape, independent of the voltage sweep rate or direction during the acquisition of the spectra.

The fact that these structures are only observed on samples with an Ir base might indicate that they are related to the peculiar shape of the Ir density of states, which has a peak at 0.8 eV above the Fermi level.[24] However, this energy is too low to correspond either to the broad peak in the BEEM and RBEEM spectra, or to a structure in the derivative of these spectra. An effect of the Si density of states is unlikely, as such spectra are not measured when gold replaces iridium.

However, the hypothesis of the electron transmission through a resonant tunneling process still does not give a straightforward explanation for the RBEEM spectra. In measurements on Schottky diodes, the threshold for BEEM and RBEEM spectra appears at the same energy, but in the case of RBEEM, it is smoother and the collector current intensity is much lower than for BEEM spectra. For an oxide barrier, even if the process generating the RBEEM current can explain the appearance of the structures in the RBEEM spectra at the same energy as in the BEEM spectra, it cannot explain either the similar shape of the spectra or the comparable intensities. Whereas in BEEM the hot electrons are injected in the base directly from the tip, the RBEEM current results from secondary electrons excited in the base through electron-hole scattering, similar to an Auger process. As a result both the momentum and energy distributions of the hot electrons in the base are expected to be different between BEEM and RBEEM. As seen in the measurements on slightly thicker oxides, the inelastic mean free path at the energies involved by the high barrier structures is sufficiently low, and accordingly the scattering is sufficiently large to give an isotropic momentum distribution in both modes. However, we expect an important intensity difference between these two modes as the energy distribution of the holes in the base participating to the RBEEM process is very different from that of the electrons in the direct BEEM process. The hot holes are coming from the tail of the distribution and are clearly fewer than the most energetic electrons near the tip Fermi level, at the maximum of the electron distribution. This effect is consistent with the absence of measurable current for RBEEM spectra on thin oxides [see Fig. 5(a)]. Thus the reason for the appearance of the broad peak in the RBEEM spectra for ultrathin oxides remains unexplained, and possibly the mechanism that determines the broad peak both in the BEEM and the RBEEM modes may be altogether different from what is described in conventional models. Finally, we believe that the understanding of these structures may uncover new information on the properties of these ultrathin oxides.

IV. CONCLUSIONS

Using the BEEM technique, we have locally studied MOS junctions with high barriers being seen even for very thin oxide layers (<30 Å). For Au/SiO_2/Si junctions, the results are in good agreement with large area C-V and internal photoemission measurements on thicker oxides. For Ir/SiO_2/Si junctions, the barrier is higher and cannot be explained solely by the work-function difference.

For transmission studies through the ultrathin oxides, we have found a striking behavior. In the case of a Au base, as the oxide thickness is reduced to ~ 10 Å, a clear threshold is still observed, although a leakage current, whose origin is uncertain, is often measured below the threshold. For Ir/SiO_2/Si junctions with an ultrathin oxide layer, anomalous spectra are observed in the BEEM and also especially the RBEEM measurements. The broad peak observed below the high-energy threshold in the BEEM spectra could result from a resonant tunneling effect through the oxide layer. However, it seems that the intensity and the similarity of the structures appearing in the RBEEM spectra cannot be explained directly through conventional BEEM models. Further measurements with different oxide thicknesses and metals other than Ir or Au as the base electrode should be done.

Finally, although the oxide layer can be very thin (~ 10 Å), we can say that neither in the case of gold (except for some rare exceptions) nor in the case of iridium, do we see simply the BEEM characteristic of a Schottky junction (i.e., no oxide layer). Already for these oxide thicknesses, at least in the case of a gold base, a high barrier seems to be formed, as in the case of thicker oxide layers. Although a complete explanation of the observed features is still missing, we expect that the elucidation of the origin of these features will contribute to a more complete understanding of transport through very thin insulators and shall allow BEEM to become a sensitive technique for the study of such interface barrier formation.

ACKNOWLEDGMENTS

We would like to acknowledge stimulating discussions with M. Decroux, critical comments on the manuscript by

Ch. Ahn, the efficient technical assistance of J.-G. Bosch, and the high-quality work of the mechanical workshop of the Physics Section of the University of Geneva. We also thank Katalin Solt of Landis & Gyr, Zug (now with the Eidgenössische Technische Hochschule, Zürich), for supplying the back implanted and thermally preoxidized Si wafer. This work was supported by the Swiss National Science Foundation, through Program No. PNR 24.

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