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## Reducing adhesion energy of micro-relay electrodes by ion beam synthesized oxide nanolayers

Bivas Saha,<sup>1,2</sup> Alexis Peschot,<sup>3</sup> Benjamin Osoba,<sup>3</sup> Changhyun Ko,<sup>1</sup> Leonard Rubin,<sup>4</sup> Tsu-Jae King Liu,<sup>3</sup> and Junqiao Wu<sup>1,2,a</sup>

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Reduction in the adhesion energy of contacting metal electrode surfaces in nano-electro-mechanical switches is crucial for operation with low hysteresis voltage. We demonstrate that by forming thin layers of metal-oxides on metals such as Ru and W, the adhesion energy can be reduced by up to a factor of ten. We employ a low-energy ion-beam synthesis technique and subsequent thermal annealing to form very thin layers ( $\sim 2$  nm) of metal-oxides (such as RuO<sub>2</sub> and WO<sub>x</sub>) on Ru and W metal surfaces and quantify the adhesion energy using an atomic force microscope with microspherical tips. © 2017 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution (CC BY) license (<http://creativecommons.org/licenses/by/4.0/>). [<http://dx.doi.org/10.1063/1.4978436>]

Nano-electro-mechanical (NEM) switches (relays) have attracted significant attention in recent years due to their potential to overcome the energy-efficiency limit of complementary metal-oxide semiconductor (CMOS) digital logic circuits and technology.<sup>1-4</sup> Since relays operate with zero off-state leakage current and abrupt switching characteristics, overcoming the limitation of 60 mV/decade room temperature subthreshold swing for CMOS transistors, they can be used to dramatically lower the energy consumption of digital electronic circuits.<sup>3,4</sup> Several theoretical analyses and experimental works have been performed to design and demonstrate relays driven by piezoelectric,<sup>2</sup> electrostatic,<sup>5</sup> or magnetic forces<sup>6</sup> and to achieve lower operating voltage.<sup>7</sup> As all relays operate on the basic principle of formation and breaking of mechanical contacts, contact adhesion energy plays an important role in determining the performance and energy efficiency of a relay.<sup>8,9</sup> Studies also have been performed to understand the effects of contact area scaling and the dynamics of contact opening/closing.<sup>9-11</sup> A general understanding gained from such analyses<sup>7,11</sup> is that the minimum energy needed to switch a relay is proportional to the contact adhesive force ( $F_{ad}$ ). Therefore, reduction of the adhesion energy ( $E_{Ad}$ ) is necessary to improve the energy efficiency of a relay.

Adhesion is dependent on many parameters, including the contacting electrode material(s). Process limitations as well as device performance requirements impose additional constraints on the choice of the material(s). For high endurance and good performance, we have used hard metals with high electrical conductivity, such as W and Ru, as contacting electrode materials in logic relays.<sup>7</sup> With a motivation to reduce the adhesion energy per unit area without dramatically reducing the relay on-state conductance, we show herein that ultra-thin metal-oxides (such as RuO<sub>2</sub> and WO<sub>x</sub>) on Ru and W metal surfaces can significantly decrease adhesion energy, by up to an order of magnitude.

The Ru and W thin films (approximately 30 nm in thickness) were deposited onto 100 nm of oxidized silicon wafer substrates at room temperature, using the reactive rf-sputtering technique

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inside a high vacuum chamber ( $3 \times 10^{-6}$  Torr). To promote adhesion, a thin (1.5 nm thick) layer of  $\text{TiO}_2$  was deposited onto the  $\text{SiO}_2$  surface by atomic layer deposition (ALD) prior to Ru deposition. No adhesive layer was used for W, since it adheres well to  $\text{SiO}_2$ . To form oxide on a metal surface, low-energy oxygen-ion implantation followed by thermal annealing at  $450^\circ\text{C}$  for 30 min in forming gas (95%  $\text{N}_2$  and 5%  $\text{H}_2$ ) was employed. The ion implantations were performed with  $7^\circ$  tilt angle and acceleration energies of 3 keV for Ru and 3.5 keV for W samples, respectively. These energies were selected based on Stopping and Range of Ions in Matter (SRIM) simulations,<sup>12</sup> to achieve a projected implant range of  $\sim 2\text{--}3$  nm. (See Fig. S1 in the [supplementary material](#) for SRIM-simulated range vs. energy, and an example of oxygen depth distribution in Ru. It should be noted that for such low acceleration energies, the longitudinal and lateral straggles are comparable in magnitude to the projected range.) Two values of  $\text{O}_2$  implant dose were investigated for each metal:  $4.5 \times 10^{15}$   $\text{cm}^{-2}$  and  $7.3 \times 10^{15}$   $\text{cm}^{-2}$  for Ru;  $4.2 \times 10^{15}$   $\text{cm}^{-2}$  and  $7.0 \times 10^{15}$   $\text{cm}^{-2}$  for W. As an additional experimental split condition, the substrate temperature was either cooled or not cooled during implantation. Atomic force microscopy (AFM) was used to measure adhesive force between contacting materials (metal/metal-oxide and AFM tip) in the force spectroscopic mode.<sup>13</sup> The measured adhesive force and the RMS surface roughness were used to calculate the adhesion energy. Material properties of the oxides, such as their composition and thicknesses, were determined by X-ray photoelectron spectroscopy (XPS).

Microspherical  $\text{SiO}_2$  tips (inset in Fig. 1(a)) with  $2\ \mu\text{m}$  tip diameter were used for the AFM measurements. The bare  $\text{SiO}_2$  tips were later coated with Ru or W using the same recipe as used to deposit these metals onto oxidized silicon wafer substrates, for measurements of adhesive force between related materials (for example, Ru-Ru contact or  $\text{RuO}_2$ -Ru contact). The relatively large tip diameter provides for consistent measurements of contact adhesive force, in contrast to more conventional sharp AFM tips with smaller diameters (less than 10 nm) that are used for tapping-mode measurements of surface topology. Using a small-diameter tip, we observed significant variations in measured adhesive force from area to area, and even within the same area of the sample. (See Fig. S2 in the [supplementary material](#) for details.)

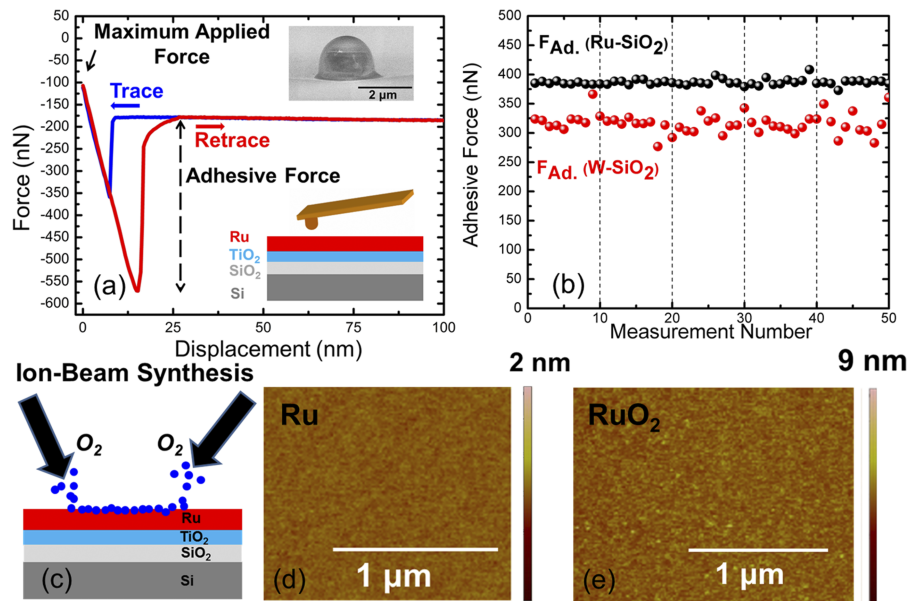


FIG. 1. (a) Representative force-vs.-displacement plot for AFM-based adhesion measurement, with the adhesive force and maximum applied force indicated. Upper inset shows a scanning electron microscopy image of the  $\text{SiO}_2$  microspherical AFM tip with  $2\ \mu\text{m}$  diameter. Lower inset shows a schematic of the measured sample structure. (b) Measured adhesive force across 50 measurements, for Ru and W metal films contacted with a bare  $\text{SiO}_2$  AFM tip. (c) Schematic illustration of the ion-beam synthesis process to form an ultra-thin layer of oxide on the metal surface. (d) AFM image of the Ru metal film surface. (e) AFM image of the oxidized Ru ( $\text{RuO}_x$ ) film surface. The RMS roughness of the surfaces was measured using conventional tapping-mode AFM with a sharp tip of  $\sim 8$  nm diameter.

A typical force-*vs.*-displacement plot obtained with a microspherical SiO<sub>2</sub> tip and Ru film is presented in Fig. 1(a), wherein the trace and retrace curves correspond to the approach and retraction of the tip to/from the Ru surface, respectively. For each of the adhesion measurements, the maximum applied force was 100 nN. The rationale for this is discussed below. The adhesive force was determined from the retrace curve as shown in Fig. 1(a). To check for reproducibility, 10 measurements were performed at each of 5 randomly chosen spots on each sample. The results for Ru(film)-SiO<sub>2</sub>(tip) contact and W(film)-SiO<sub>2</sub>(tip) contact are presented in Fig. 1(b). For Ru, the measurements are very consistent at 386 nN on average, with a standard deviation of 5 nN. For W, there is a greater variation, with a standard deviation of 16 nN for an average adhesive force of 316 nN. This is attributed to the larger RMS surface roughness of the W film (0.45 nm) as compared against the Ru film (0.21 nm), as deduced from AFM measurements (e.g., Fig. 1(d)), since larger surface roughness results in larger differences in the contact environment from measurement to measurement. Nevertheless, the variation in measured values of adhesive force is relatively small (less than 5%), for all of the samples studied in this work.

Adhesive force between materials has several origins including (1) van der Waals force, (2) chemical interaction forces due to bonding between the atoms of the materials, (3) electrostatic interactions, and (4) capillary force due to external contaminants such as water and polymers on the sample surfaces.<sup>14</sup> While the first three are intrinsic, the fourth can be mitigated with proper care. Hence the adhesion measurements were performed in a dry-N<sub>2</sub> environment. (Prior to measurement, the sample was *in situ* heated to 140 °C for several hours and then cooled to room temperature under dry-N<sub>2</sub> flow.) The capillary force was found to contribute less than 12% to the total adhesive force (see Fig. S3 in the [supplementary material](#)).

The adhesion energy per unit area, which is an absolute gauge of the adhesion property of a material, was calculated using the Derjaguin-Muller-Toporov (DMT) continuum mechanics model.<sup>14,15</sup> The effect of surface roughness was accounted for using the modified-Rumpf model<sup>16,17</sup> since it is best suited for solid materials with surface roughness values less than 2 nm.<sup>17</sup> In this model, the roughness is approximated as a single hemispherical asperity on a flat surface; the radius of the asperity depends only on the RMS roughness of the surface.<sup>15,17</sup> The equation combining the DMT and modified-Rumpf model is<sup>13</sup>

$$W_{Ad.} = \left( \frac{F_{Ad.}}{2\pi R_{tip}} \right) \frac{\left( 1 + \frac{R_{tip}}{1.48R_{atomic}} \right)^{-1} + \left( 1 + \frac{1.48R_{atomic}}{z_0} \right)^{-2}}{\left( 1 + \frac{R_{tip}}{1.48R_q} \right)^{-1} + \left( 1 + \frac{1.48R_q}{z_0} \right)^{-2}}, \quad (1)$$

where  $W_{Ad.}$  is the adhesion energy per unit area,  $F_{Ad.}$  is the adhesive force determined from AFM measurements,  $R_{tip}$  is the radius of the AFM tip,  $R_{atomic}$  and  $R_q$  are the RMS roughness values of the sample surface and the tip, respectively, and  $z_0$  is the equilibrium separation distance between the tip and the sample when they are in contact. A value of 0.32 nm was used for  $z_0$  for all contact cases in this work, which represents the *c*-axis lattice parameter<sup>18</sup> of RuO<sub>2</sub>. For the bare SiO<sub>2</sub> tip, a roughness value of 0.3 nm was used. A sensitivity test of the SiO<sub>2</sub> RMS roughness was performed, which showed that the percentage adhesion energy reduction by oxidation of the metal is insensitive to the tip roughness, as expected. However, the adhesion energy values of the individual sample-tip contacts depend on the tip roughness value assumed. For the Ru- or W-coated tip, the roughness value of the metal thin film was used. The RMS roughness of the implanted and annealed RuO<sub>2</sub> sample was measured to be 0.55 nm, while the WO<sub>x</sub> film has an RMS roughness of 1.51 nm.

Calculated values of adhesion energy per unit area for contacts between metal or metal-oxide thin films and AFM tips (bare SiO<sub>2</sub>, or coated with Ru or W) are presented in Fig. 2. The adhesion energy between Ru and SiO<sub>2</sub> is 92 mJ/m<sup>2</sup>, whereas the adhesion energy between W and SiO<sub>2</sub> is about 3× lower, at 30 mJ/m<sup>2</sup>. Fig. 2 also shows that the adhesion energies are ~3× and ~5× smaller for RuO<sub>2</sub> and WO<sub>x</sub> in contact with SiO<sub>2</sub> (27 mJ/m<sup>2</sup> for RuO<sub>2</sub>-SiO<sub>2</sub> and 5 mJ/m<sup>2</sup> for WO<sub>x</sub>-SiO<sub>2</sub>). Clearly, oxide formation on metal surfaces reduces adhesion energy. The results for RuO<sub>x</sub> samples formed with different implantation conditions are very similar (within the error ranges—see Fig. S4 in the [supplementary material](#)). This is not altogether surprising, since the samples were annealed together after O<sub>2</sub> implantation so that the chemical compositions of the oxides formed on the surfaces are

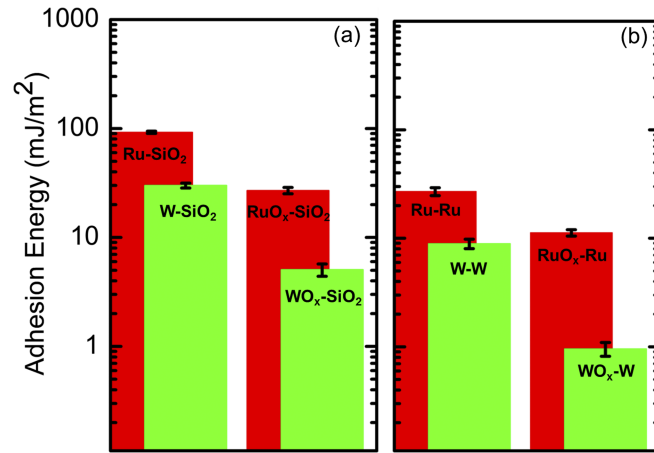


FIG. 2. Comparison of calculated values of adhesion energy per unit area for Ru, RuO<sub>x</sub> (with  $x = 2$  at the surface), W and WO<sub>x</sub> contacted with (a) a bare SiO<sub>2</sub> AFM tip or (b) a metal-coated SiO<sub>2</sub> AFM tip.

expected to be very similar. Moreover, the RMS roughness values of the samples were found to be very similar. The adhesion energies for Ru-Ru and W-W contacts (i.e., deduced from measurements using an AFM tip coated with metal) are 27 mJ/m<sup>2</sup> and 9 mJ/m<sup>2</sup>, respectively. Oxidation of the metal thin film surface reduces these to very low values of 11 mJ/m<sup>2</sup> and 1 mJ/m<sup>2</sup>, respectively, similarly as for SiO<sub>2</sub> contacts. While the measurements and calculated results clearly show a decrease in adhesion energy of oxidized vs. pristine surfaces, one should not compare measurements made with different tips (e.g., SiO<sub>2</sub> tip versus Ru tip), as deposition of coating material on the tip will change the tip properties and alter the nature of tip-sample interaction, and hence influence the obtained adhesion values.

Reduction in adhesion energy due to oxide formation on a metal surface could be due to several factors. We believe that the reduction of chemical interaction forces plays an important role in the reduction mechanism. In addition, van der Waals forces and Casimir forces could also be reduced as a result of oxide formation and the resultant lower electrical conductivity of the surface. The adhesion energy per unit area of an interface between two materials A and B is given by the following equation:<sup>19</sup>

$$W_{Ad.} = \sigma_A + \sigma_B - \gamma_{A-B}, \quad (2)$$

where  $\sigma_A$  and  $\sigma_B$  are the surface energies of the materials A and B, respectively, and  $\gamma_{A-B}$  is the energy of the interface between materials A and B. Most metals have high surface energy and low interface energy due to the presence of dangling bonds on the surface. Electrostatic attraction between conduction electrons of the contacting metals makes metal-metal bonds easy to form across the interface.<sup>20</sup> A phenomenological expression for the interaction potential of two similar metallic surfaces is<sup>21</sup>

$$W(D) = 2\sigma \left[ 1 - \frac{D - D_0}{\lambda_M} \right] e^{-\frac{(D-D_0)}{\lambda_M}}, \quad (3)$$

where  $\lambda_M$  is a characteristic decay length of the metal material,  $D$  is the distance between the metal surfaces, and  $D_0$  is the equilibrium distance. Under the equilibrium condition, the adhesion energy is  $W(D) = 2\sigma$ . As a result, metal contacts generally have large adhesion energies. In contrast, oxides (like most ceramics) usually have nearly saturated surface bonds so that they have low surface energies. Also, oxide-oxide bonds are more covalent in character and are more difficult to form than metallic bonds. Therefore, oxide contacts typically have lower adhesion energies compared to metal contacts. Given this general description, it is not surprising that the formation of oxides on metal surfaces was found in this work to considerably reduce adhesion energies.

To determine the stoichiometry and thicknesses of the ion-beam synthesized oxides on the metal surfaces, we performed detailed XPS analyses to compare the O and Ru depth profiles in

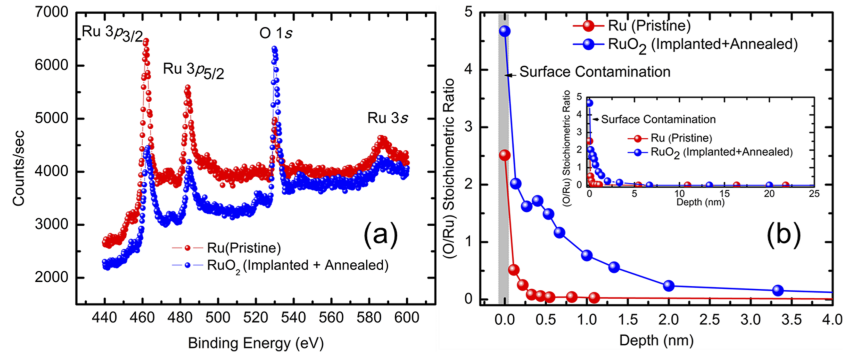


FIG. 3. (a) X-ray photoelectron spectra (XPS) of Ru and RuO<sub>2</sub> films (collected after gently removing the surface contamination layer) showing that the oxygen signal is significantly higher in the O<sub>2</sub>-implanted and annealed sample vs. the pristine Ru sample, indicative of oxide formation in the former. (b) Comparison of O/Ru stoichiometric ratio depth profiles, showing that the surface of pristine Ru sample is not oxidized, in contrast to the O<sub>2</sub>-implanted and annealed Ru sample.

a pristine Ru sample vs. an implanted and annealed sample. The results presented in Fig. 3 indicate that the pristine-Ru sample is very pure, with negligible native oxide on the surface; this is consistent with our previous experiments demonstrating that native RuO<sub>x</sub> formation is a self-limiting process.<sup>22</sup> The results for the implanted and annealed sample indicate an oxide thickness of ~2 nm with a stoichiometry of RuO<sub>2</sub> at the surface, confirming the accuracy of the SRIM simulations.

Using the DMT model,<sup>14,15</sup> we have also estimated the effects of other parameters such as the contact radius ( $a$ ), sample deformation ( $\delta$ ), and contact pressure ( $P$ ) underneath the tip during measurement. The contact radius ( $a$ ) for the AFM tip is given by the following expression:<sup>15</sup>

$$a = \sqrt[3]{\frac{R_{tip}}{E_{total}}(F + 2\pi R_{tip}W_{Ad.})}, \quad (4)$$

where  $E_{total}$  is the reduced elastic modulus, which is a combined elastic modulus that considers both the tip material and the sample material through the relation presented in Eq. (5), where  $\nu_s$ ,  $E_s$ ,  $\nu_t$ , and  $E_t$  are the Poisson's ratio and Young's moduli of the sample and tip, respectively,

$$\frac{1}{E_{total}} = \frac{3}{4} \left( \frac{1 - \nu_s^2}{E_s} + \frac{1 - \nu_t^2}{E_t} \right), \quad (5)$$

$F$  is the applied force, and  $R_{tip}$  and  $W_{Ad.}$  are the tip radius and the adhesion energy, respectively. We find that the contact radius of the AFM tip is approximately 25 nm, which gives a contact area of ~2000 nm<sup>2</sup> for 100 nN of applied force in a Ru-SiO<sub>2</sub> contact. Considering that the AFM tip radius is 1  $\mu$ m, only a small portion (about 2.5%) of the tip is actually in contact with the sample surface when the applied force is 100 nN. However, our measurements show that 2000 nm<sup>2</sup> of contact area is sufficient for reproducible bond formation and breaking (from measurement to measurement), despite the aforementioned effect of surface roughening to increase variability. The deformation of the sample surface ( $\delta$ ) is given by the expression  $\frac{a^2}{R_{tip}}$  and is approximately 0.63 nm for our measurements. Such a small deformation ensures that the materials are in the elastic regime, for accurate measurement of adhesion energy. The pressure underneath the tip is calculated to be ~350 bars during the measurements.

Adhesive force per unit area in a functional NEM relay has been extracted by Yaung *et al.*<sup>8</sup> and Lee *et al.*<sup>23</sup> using indirect methods such as electrical characterization and indirect AFM-based analysis wherein the force is applied on the movable electrode of the relay. While the results extracted from the electrical characterization showed an adhesive force of 0.02 nN/nm<sup>2</sup> between the W electrodes, the indirect AFM measurement showed a much lower adhesive force of  $1.6 \times 10^{-5}$  nN/nm<sup>2</sup>. The direct AFM based experimental analysis performed here between W-W contacts shows that the adhesive force is about 0.17 nN/nm<sup>2</sup>, which is an order of magnitude higher than the estimates by Yaung *et al.*<sup>9</sup> We believe that this difference is due to the assumptions used in estimating the

contact area: While Young *et al.*<sup>8</sup> have used the dimple (apparent) contact area for the calculation of force per unit area, here we use the DMT model to estimate the microscopic (real) contact area.

As the contact area increases with applied force, so should chemical interaction forces. To investigate this effect, we performed an experiment in which the applied force was increased from 100 nN to 1  $\mu$ N. The results presented in Fig. 4 show that the RuO<sub>2</sub>-SiO<sub>2</sub> adhesion energy increases with the applied force, by about 25%. Using the DMT model, we estimate that the corresponding increase in the contact area is about 44%. This suggests that the sample is plastically deformed for 1  $\mu$ N of applied force. Therefore, an applied force of 100 nN was used to obtain all of the data presented in Fig. 2. (An applied force of 50 nN was found to be too small to yield reproducible measurements.) It should be noted that the contact force in NEM relays is a strong function of the relay operation mode and body-biasing condition and could range from a few micro-newtons to very small values (theoretically approaching zero when the body-bias voltage is close to the release voltage). Thus, the 100 nN applied force in our AFM measurements is within the range relevant for relay operation.

Logic relays should be stable and operate with high endurance, more than 10<sup>14</sup> cycles,<sup>24</sup> in order to have a broad application. In the previous work,<sup>25</sup> we demonstrated that relays with Ru contacts can operate with sufficient on-state conductance for at least 10<sup>8</sup> cycles, under low vacuum conditions. Stability of contact adhesion is also critical for achieving highly reliable relay operation, since a large adhesive force can cause a relay to be stuck in the on state. We have therefore studied the evolution of adhesive force in a RuO<sub>2</sub>-SiO<sub>2</sub> contact over the course of many operating cycles. Due to the low frequency (0.25 Hz) of the AFM measurement technique, we were able to monitor the contact for only 1000 cycles; nevertheless, the results provide insight into the stability of adhesion energy. The results presented in Fig. 4(b) show that the adhesive force remains stable over the 1000 operating cycles that is expected to result in efficient device performance. Adhesion cycling tests performed on bare-Ru/SiO<sub>2</sub> (shown in the [supplementary material](#)) indicate an increase in adhesive force by ~6% over the 1000 contact cycles, probably due to wearing of the tip, highlighting the benefit of oxides in terms of contact stability.

Having discussed the adhesion properties of the metals and metal-oxides, we now comment briefly on their electrical properties. Previous reports<sup>22</sup> indicate that RuO<sub>2</sub> (the only Ru oxide that is stable in atmospheric ambient) is electrically conductive, since it has a resistivity that is only a factor of seven higher than that of pure Ru, at room temperature. Considering that the ion-beam synthesized RuO<sub>2</sub> is only ~2 nm thick, and that a contact resistance of ~1 m $\Omega$  was measured between two RuO<sub>2</sub> disks with 1 cm diameter,<sup>26</sup> we would not expect it to dramatically increase the contact resistance. Also, oxide formation on Ru electrodes should help stabilize the stiction coefficient as a function of

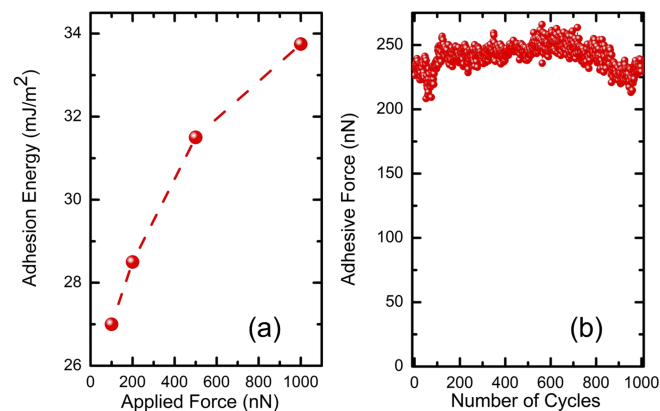


FIG. 4. (a) Adhesion energy for RuO<sub>2</sub>-SiO<sub>2</sub> contact as a function of the applied force during the AFM measurement. (b) Evolution of contact adhesive force with the number of cycles, for a RuO<sub>2</sub> thin film contacted by a bare SiO<sub>2</sub> AFM tip with 0.25 Hertz frequency. Further experiments (not presented here) show that the characterization frequency of the AFM measurements does not affect the nature of the cycling test and the reliability of the measurements.



cycles under hot-switching conditions (see [supplementary material](#)). In contrast,  $\text{WO}_3$  is known to be electrically insulating,<sup>27</sup> so the stoichiometry and thickness of the  $\text{WO}_x$  layer must be carefully controlled (by adjusting the  $\text{O}_2$  ion implant dose and acceleration energy) to ensure a sufficiently high on-state electrical conductance.

In conclusion, we have demonstrated that low-energy ion beam synthesis and subsequent thermal annealing result in nanolayers of metal oxides (such as  $\text{RuO}_2$  and  $\text{WO}_x$ ) on Ru and W metal surfaces, and the adhesion energy of the metals can be reduced by up to a factor of ten through the formation of the oxide nanolayers. XPS analysis shows that the oxide nanolayers are  $\sim 2$  nm thick and stoichiometric (for  $\text{RuO}_2$  on the Ru metal surfaces), which is expected to result in low on-state resistance needed for relay operations. The adhesion energy is quantified using an AFM with microspherical tips. Reduction of the adhesion in NEM contact metals through oxide nanolayers is important not just for the relay switch applications, but also for several other types of MEMS devices including RF MEMS switches that rely on the contact between a conductive membrane and an insulating dielectric. Therefore, this technique can be used to improve the energy efficiency of a broad class of MEMS and NEMS devices.

See [supplementary material](#) for the details about SRIM simulation results, adhesion measurements with AFM tip of small diameter, quantification of capillary forces, effects of ion-implantation process parameters on adhesion, sensitivity test of  $\text{SiO}_2$  probe tip roughness, contact cycling results of bare-Ru/ $\text{SiO}_2$  adhesive force, effects of AFM contact frequency on measurements, possible effects of electrical current and triboelectric effect on contact adhesion, and possible packaging conditions of the NEM relays.

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