

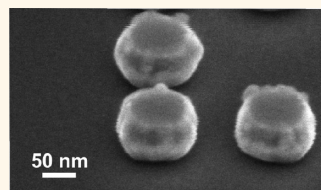
# High-Yield Transfer Printing of Metal–Insulator–Metal Nanodiodes

Mario Bareiß,<sup>†,\*</sup> Frederik Ante,<sup>‡</sup> Daniel Kälblein,<sup>‡</sup> Gunther Jegert,<sup>†</sup> Christian Jirauschek,<sup>†</sup> Giuseppe Scarpa,<sup>†</sup> Bernhard Fabel,<sup>†</sup> Edward M. Nelson,<sup>§</sup> Gregory Timp,<sup>§</sup> Ute Zschieschang,<sup>‡</sup> Hagen Klauk,<sup>‡</sup> Wolfgang Porod,<sup>§</sup> and Paolo Lugli<sup>†</sup>

<sup>†</sup>Institute for Nanoelectronics, Technische Universität München, Arcisstrasse 21, 80333 Munich, Germany, <sup>‡</sup>Max Planck Institute for Solid State Research, Heisenbergstrasse 1, 70569 Stuttgart, Germany, and <sup>§</sup>Center for Nano Science and Technology, University of Notre Dame, 275 Fitzpatrick, Indiana 46556, United States

**M**etal–insulator–metal (MIM) junctions represent promising devices for various electronic and optoelectronic applications, such as antennas for energy harvesting<sup>1,2</sup> or infrared detection,<sup>3</sup> field-emission cathodes<sup>4</sup> and switching memories.<sup>5</sup> Of particular interest are devices operating at a few THz, a frequency range where sensing<sup>6</sup> and communication<sup>7</sup> are particularly attractive. Other applications of THz devices involve biomedical investigation of DNA<sup>8</sup> or enzymes,<sup>9</sup> or night-vision cameras and security equipment. MIM structures consist of two metal layers separated by a nanometer-sized insulator. Several materials for the insulating layer have been reported, including various oxides.<sup>10–13</sup> In the field of rectennas for infrared detection, native aluminum oxide (AlOx) has been the insulating material of choice so far.<sup>14</sup> However, naturally grown oxides are not very reliable and reproducible even in simultaneously fabricated diodes (see Supporting Information). Here, we present a fabrication method of a  $\sim 3.6$  nm thick, low-defect AlOx. When applying a high-frequency alternating electrical field across the MIM junction, for example, in the terahertz (THz) regime, tunneling electrons can follow this field as the cutoff frequency of nanoscale MIM structures can reach several hundreds of THz.<sup>15</sup> Because of the nonlinear  $I$ – $V$  characteristics of a quantum mechanical tunneling current, the MIM junction can act as a rectifying device.<sup>16</sup> By using two different metals for the contacts, rectification can be achieved at zero bias. So far, only isolated nanoscale MIM diodes containing an air oxidized insulator have been reported.<sup>17</sup> We present here for the first time a high-yield scalable method for the fabrication of several millions of vertical MIM nanodiodes in one single process step, namely a temperature-enhanced nanotransfer printing process. In previous works, various

**ABSTRACT** Nanoscale metal–insulator–metal (MIM) diodes represent important devices in the fields of electronic circuits, detectors, communication, and energy, as their cutoff frequencies may extend into the “gap” between the electronic microwave range and the optical



long-wave infrared regime. In this paper, we present a nanotransfer printing method, which allows the efficient and simultaneous fabrication of large-scale arrays of MIM nanodiode stacks, thus offering the possibility of low-cost mass production. In previous work, we have demonstrated the successful transfer and electrical characterization of macroscopic structures. Here, we demonstrate for the first time the fabrication of several millions of nanoscale diodes with a single transfer-printing step using a temperature-enhanced process. The electrical characterization of individual MIM nanodiodes was performed using a conductive atomic force microscope (AFM) setup. Our analysis shows that the tunneling current is the dominant conduction mechanism, and the electrical measurement data agree well with experimental data on previously fabricated microscale diodes and numerical simulations.

**KEYWORDS:** metal–insulator–metal nanodiodes · nano transfer printing · terahertz rectifier · conductive atomic force microscope · tunneling current

microscopic scale devices have been successfully transfer-printed.<sup>18–21</sup> In particular, we presented recently transfer-printed MIM diodes (MHz rectifiers)<sup>22</sup> which also feature spatial dimensions in the micrometer range. A transfer-printed electrical nanodevice has not been demonstrated so far. A conductive atomic force microscope (C-AFM) setup<sup>23</sup> is used to electrically characterize individual MIM nanodiodes. We demonstrate the functioning of the nanodevices and the fabrication method by comparing the current–voltage characteristics of the printed nanoscale MIM diodes to those of printed microscale diodes, which we previously reported. The diode operation is analyzed by numerical simulations. Finally, the THz performance of the transfer-printed MIM structure is estimated.

\* Address correspondence to Bareiss@nano.ei.tum.de.

Received for review January 27, 2012 and accepted March 2, 2012.

Published online March 02, 2012  
10.1021/nn3004058

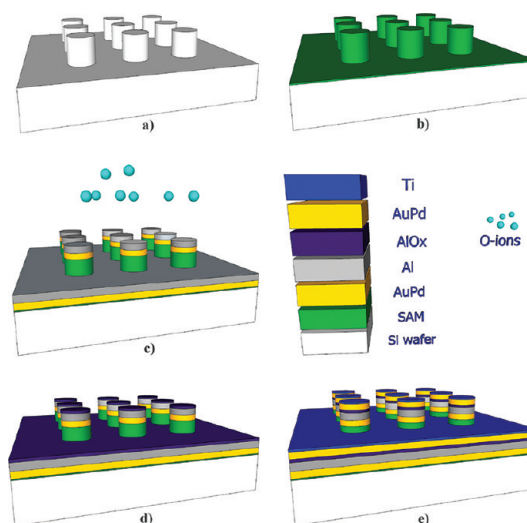
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## DISCUSSION

**Temperature-Enhanced Nanotransfer Printing of MIM Nanodiodes.** Electron-beam lithography (EBL)<sup>24,25</sup> is a commonly used method for patterning materials and fabricating devices with dimensions between 30 and 100 nm. However, EBL lacks the fabrication speed and cost efficiency required for the processing of large areas. In contrast, nanotransfer printing (nTP) offers the possibility to print large, dense arrays of nanoscale devices on arbitrary flat surfaces, provided the adhesive and cohesive forces, surface energies, and material properties are properly tailored.<sup>26</sup> Using temperature-enhanced nTP, we have fabricated an array of approximately 4 million MIM pillar diodes, each with a diameter of less than 100 nm, suitable for electronic and optoelectronic applications.

First, a stamp was designed and fabricated from a silicon wafer by EBL and highly anisotropy reactive ion etching (RIE). The stamp contains an array of cylindrical mesas covering a square area of  $300 \times 300 \mu\text{m}^2$  (see Figure 1a for a schematic, and Figure 2a for an electron microscopy image). Successful transfers with stamps up to  $1 \text{ cm}^2$  have also been performed. The mesas have a height of 80 nm to provide good physical stability during the printing process. The diameter of the mesas and the spacing between adjacent mesas was designed between 50 and 100 nm.

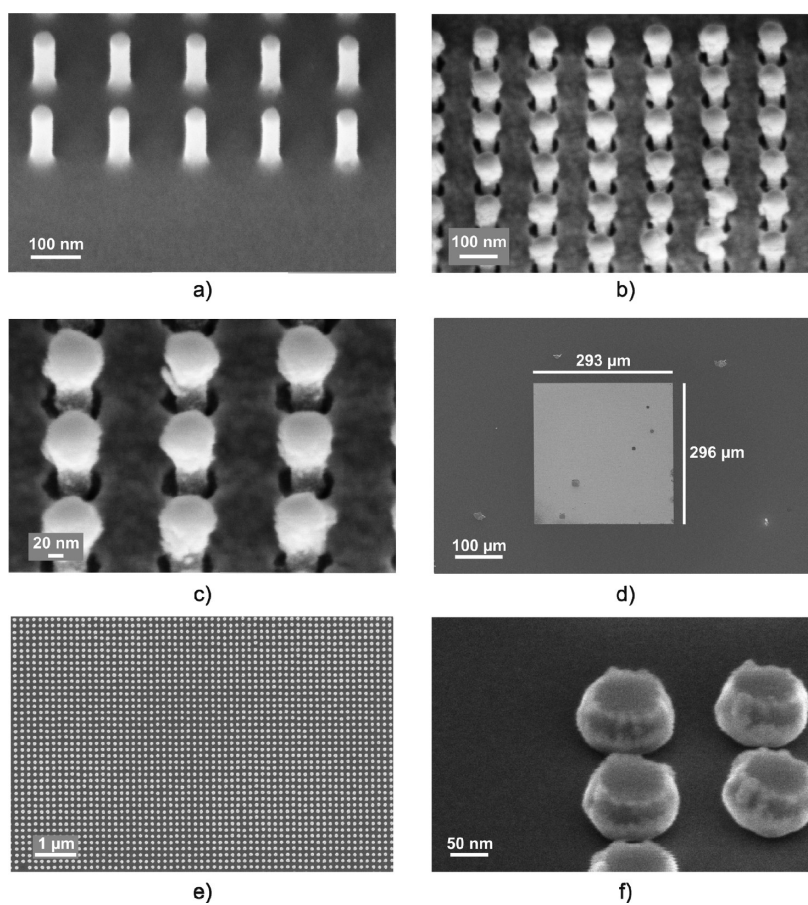
The preparation of the stamp prior to printing is outlined in Figure 1b–e. First, the stamp was covered with an alkylsilane self-assembled monolayer (SAM) to reduce its surface energy. Next, a 15 nm thick layer of AuPd was deposited by thermal evaporation onto the SAM-covered stamp to provide a delamination layer from which the MIM pillars can later be easily transferred to the target substrate. In the next step, a 25 nm thick layer of aluminum with a surface roughness of less than 1 nm was deposited as the first electrode. The stamp was then briefly exposed to oxygen plasma to form a high-quality aluminum oxide tunnel barrier with a thickness of 3.6 nm.<sup>27</sup> Compared with aluminum oxide obtained by native oxidation in air, the oxygen-plasma-grown oxide has been found to provide substantially better reliability and reproducibility. To complete the MIM structure, a 15 nm thick AuPd layer was then deposited as the second electrode. Finally, a 4 nm thick layer of titanium was deposited by evaporation to provide an adhesion promoter for the nanotransfer printing process. To facilitate good physical contact between the stamp and the target substrate, the layer stack deposited onto the stamp must be as smooth as possible. Therefore, metals with a small grain size, such as Al, AuPd, and Ti, are more desirable than, for example, Au. During the metal evaporations the stamp was held perpendicular to the metal source in order to minimize the deposition of metals on the sidewalls of the mesas (see Figure 2b) and Figure 2c).



**Figure 1.** Sequence of process steps to prepare the stamp for transfer-printing of the nanoscale MIM pillars. (a) An array of mesas with a height of 80 nm and a diameter of 50 to 100 nm is fabricated on the surface of the silicon stamp by electron-beam lithography and highly anisotropic silicon etching. (b) The silicon stamp is covered with an alkylsilane self-assembled monolayer (SAM). (c) 15 nm of AuPd and 25 nm of Al (first electrode of the MIM diodes) are deposited by thermal evaporation. (d) The Al surface is exposed to oxygen plasma to form a  $\sim 3.6$  nm thick AlOx tunnel barrier. (e) Finally, 15 nm of AuPd (second electrode of the MIM diodes) and 4 nm of Ti (to promote adhesion to the target substrate) are deposited by thermal evaporation.

As target substrate we have used a p-type silicon wafer covered with an AuPd layer (to electrically connect the bottom electrodes of all diodes) and a thin titanium layer (to provide good adhesion of the printed layer stack). Immediately prior to printing, the titanium-coated surfaces of the stamp and the target substrate were physically activated (the parameters can be found in the Supporting Information). When the titanium-coated MIM pillars on top of the mesas of the stamp are brought into physical contact with the titanium-covered target substrate, individual and isolated MIM nanodiodes are transferred from the stamp to the substrate, resulting in a high density of devices. The transfer process is facilitated by applying a temperature of 200 °C during printing. We found that such a high temperature promotes the formation of covalent bonds between the titanium layers on the top of the stamp and on the substrate due to the removal of water molecules.<sup>28</sup>

**Morphological Characterization of Printed Nanoscale MIM Diodes.** To investigate the quality of the nTP process, three nominally identical samples were fabricated and investigated by scanning electron microscopy (SEM). The transfer yield, which we define as the number of properly transferred MIM diode stacks divided by the number of pillars on the stamp, was found to be 98%, which means approximately 4 million diodes were successfully transferred. During the metal and insulator deposition process, material was deposited not only on



**Figure 2.** Stamp preparation and large area out-of-plane printed nanodiodes. (a) A scanning electron microscope (SEM) image of an edge of the Si stamp is shown at a tilt angle of  $45^\circ$ . The pillars feature a height of 80 nm and a diameter of 50 nm. (b) Metal layers and an insulator are fabricated on the stamp that is covered with a hydrophobic self-assembled monolayer. (c) No material was deposited on the sidewalls of the pillars as we used collimator plates during the evaporation. (d) After covering the stamp with metals and the insulator, almost the whole array of MIM nanodiodes was transfer-printed on a target substrate. (e) A zoom of the printed area covered with nanodiodes is presented here. (f) 3-Dimensional individual diodes can be seen by tilting the electron beam by  $54^\circ$ . The surface of the printed nanodiodes is very smooth allowing contacting the top with a conductive atomic force microscope.

the top of the pillar structures of the stamp, but also around the periphery of the previously deposited metal stack (although not on the sidewalls of the pillars). Therefore, the area of the MIM structure increases with increasing metal layer thickness. Indeed, the diameter of the transferred quantum devices was found to be larger by about 45 nm than the original feature size on all three stamps. Taking these effects into consideration, the diode area and the distance from each other can be tailored very precisely, and nanoscale structures with a very high density can be fabricated. To compete with high resolution EBL fabrication techniques, the morphology and height of the printed devices have to be homogeneous and must remain unchanged after the printing process. The fidelity of the printing process is proven by the perfect match between the height of the transfer-printed MIM diodes, measured with an atomic force microscope (AFM), and the evaporated material thicknesses on the stamp. The printed quantum pillars have a height of 60 nm and feature a stack shape expected from the

original layer depositions on the stamp. The top surfaces of the transferred tunneling diodes are very smooth, thus providing an excellent contact area for electrical characterization with a conductive AFM tip.

We have therefore demonstrated that several millions of MIM nanodiodes can be fabricated by a few evaporation steps and a fast printing process, without need of resists or other patterning steps (other than those used to make the stamp), followed only by some plasma treatments. Thus, temperature-enhanced nTP provides a solution to the challenge of fabricating large-scale and high-density homogeneous arrays of nanoscale devices in a fast and economic way. The stamp can be used several times before degradation occurs.

**Electrical Characterization with Conductive AFM Tips and Numerical Simulation.** The electrical properties of the transfer-printed MIM nanodiodes have been quantified using a conductive atomic force microscope (C-AFM). With this high spatial resolution characterization method, several nanodiodes have been tested individually, and

the static device parameters of single nanodevices have been extracted, allowing conclusions about the underlying electron transport mechanism. The conductive AFM tips consisted of silicon covered by a Pt–Ti alloy and featured a tip radius of 15 nm. The electrical setup allowed a local application of voltages up to 10 V and measurement of the current at the same time. The sample with the printed rectifiers was placed on a ceramic holder, wherein the AuPd layer on the substrate was connected through a magnetic gold clamp from the top (see Figure 3b). As the AuPd electrode of the printed MIM structure was attached to the conductive metal layer on the substrate, the bottom of the transfer-printed diode was directly connected to the gold clamp. The top of the diode, consisting of the Al and the AuPd delimitation layers, was electrically connected *via* the C-AFM tip. The conductive tip and the gold clamp on the substrate were connected to a parameter analyzer which was located in the head of the C-AFM setup (see Figure 3a). This circuit was used for the electrical characterization of single MIM nanodiodes. The voltage was applied on the C-AFM tip, and the current was measured at the same time.

As the area of the MIM diode is in the nanometer range, currents from  $10^{-19}$  A up to  $10^{-6}$  A are expected. Although the C-AFM setup was in a temperature, humidity, and vibration controlled environment, electrical noise still occurred, and the lowest measurable currents were in the 100 pA-range which is similar to C-AFM measurements reported in literature.<sup>29,30</sup> We investigated several nanodiodes, and only one diode showed a short circuit behavior. In all other diodes, an exponential  $I$ – $V$  characteristic was measured (see Figure 4a). An asymmetric tunneling current behavior with respect to the applied bias was observed as expected due to the asymmetric diode structure with different metal electrodes. In a comparison of the measured current density of the nanoscale tunneling diodes with previously transfer-printed diodes featuring an area in the micrometer range,<sup>31</sup> the current densities are in excellent agreement to each other proving the quality of the materials and fabrication process. The electrical stability under an applied voltage was improved by a factor of at least 2 by reducing the area of the tunneling diodes. The previously presented tunneling diodes showed a breakdown behavior around 5 to 6 V. In contrast, the nanoscale diodes worked at least up to 10 V. We believe that the improved break down behavior is related to the reduced number of defects found in the insulating layers as the diode area is scaled down. This is another indication of the high quality of the ultrathin AlOx layer fabricated by the plasma treatment.

To extract static device parameters and to determine whether or not tunneling is the dominant transport mechanism, numerical simulations were carried out.<sup>32</sup>

Hole currents can be neglected since the tunnel barrier for holes is much higher than for electrons. By assuming that direct/Fowler-Nordheim tunneling is the dominant tunneling process, the current density can be modeled by the Tsu–Esaki formula:<sup>33</sup>

$$J = \frac{em_e k_B T}{2\pi^2 \hbar^3} \int P(E) \ln \left\{ \frac{1 + \exp(-E/k_B T)}{1 + \exp[-(eV + E)/k_B T]} \right\} dE$$

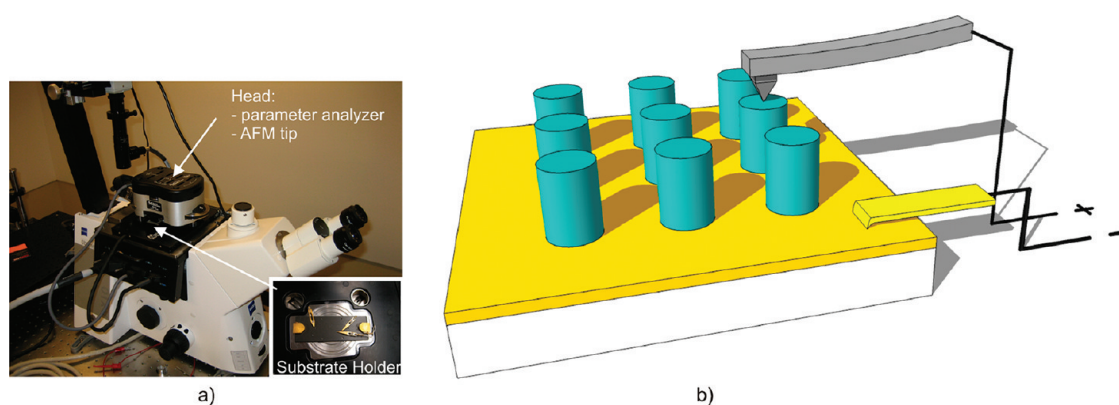
where  $P(E)$  is the transmission coefficient for electrons with energy  $E$ . An excellent fit of the simulation for voltages up to 6 V was accomplished when considering an energy dependent effective tunneling mass and using tunnel barrier heights of 4.2 eV for the AuPd electrode and 2.8 eV for the Al electrode. The value of 4.2 eV at the AuPd–AlOx interface corresponds to the barrier height of Au in the Schottky limit. As the barrier height at the Al–AlOx interface is known to be dependent on the growth mechanism, a smaller barrier height of 2.8 eV than the expected value of 3.1 eV can be explained by the formation of a dipole layer produced by charge transfer between the Al electrode and interfacial gap states in the AlOx which is known to reduce the oxide barrier height.<sup>34,35</sup> For higher voltages, the simulation predicts higher values for the current density. However scattering processes like electron–phonon interactions are not included in the simulation which are expected to reduce the tunneling current.<sup>36</sup>

Currently, we are working on a carbon-nanotubes (CNT)/graphene transfer process in order to place a highly conductive film on the top MIM layer for addressing not only single MIM nanodiodes, but an array of diodes.

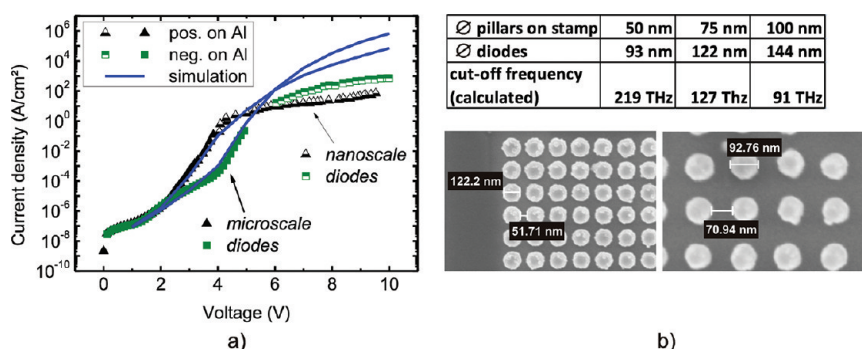
**Estimation of the Nanodiodes Cut-off Frequency.** The cut-off frequency ( $f_{\text{cut}}$ ) is the maximum frequency at which an electrical device still works properly.<sup>37</sup> By approximating a nanodiode as a parallel-plate-type capacitor,  $f_{\text{cut}}$  is defined as

$$f_{\text{cut}} = \frac{1}{2\pi RC} = \frac{d}{2\pi R \epsilon_0 \epsilon_r A}$$

where  $R$  is the resistance of the lead lines (here the external wires and the AuPd substrate), and  $C$  is defined by the capacitive area  $A$  and dielectric thickness  $d$ . Here, the tunnel resistance of the diode can be neglected as it is much higher and in parallel to the resistance of the lead lines.<sup>16</sup> The increase of the thickness of the dielectric leads to an increase of the cutoff frequency. However, the tunneling current is exponentially dependent on the tunnel barrier thickness. Thus, a dramatic current decrease can be expected when increasing the thickness of the dielectric. In general, a direct measurement of the permittivity of MIM nanodiodes is not possible with a C-AFM setup. However, we have previously determined the permittivity of the MIM diodes comprising a microscale area



**Figure 3.** Conductive atomic force microscope setup. (a) A photograph of the conductive atomic force microscope (AFM) setup: The parameter analyzer and the AFM tip are implemented in the head, and the substrate holder comprises ceramic plate and gold clamps for connecting the sample substrate. (b) A schematic view of the AFM setup: The MIM pillars (turquoise) that are transfer-printed on a Si wafer (white) comprising a conductive AuPd layer (orange) are contacted from the top via a conductive AFM tip (here, positive voltage). The AuPd-layer on the substrate is connected via a gold clamp (here, negative voltage).



**Figure 4.**  $I$ – $V$  Characteristics of MIM nanodiode. (a) The  $I$ – $V$  characteristic of a transfer-printed nanoscale MIM pillar (half-open symbols), measured using a conductive AFM setup, is presented in comparison with previously fabricated microscale MIM diodes (full symbols), characterized with a probe station, and numerical simulations (line). For a better comparison the positive and the negative polarity (on aluminum) are both shown on the positive  $V$ -axis, and the absolute logarithmic scale is used for the current density for the same reason. The nanoscale and microscale MIM structures show an excellent agreement, and the simulation could extract static device parameters. (b) The calculated cutoff frequencies of three different transfer-printed MIM diodes comprising areas between 93 and 144 nm are shown in the table, and two SEM images show the spatial dimensions of MIM diode arrays.

in a low noise probe station and the diodes showed a capacitance density of  $C/A = 1.19 \times 10^{-6} \text{ F/cm}^2$ .<sup>31</sup>

We showed there that the dielectric constant  $\epsilon_r$  is strongly dependent on the insulator thickness, although it is independent of the area  $A$ . The resistance  $R$  of the AuPd substrate that took over the lead lines was determined to be  $8.93 \Omega$ . The area of the MIM tunneling diodes was measured via SEM, and the diameter of the smallest printed pillars was determined to be 93 nm. A cut-frequency of 219 THz for the smallest MIM diodes can therefore be estimated. A further increase of the cutoff frequency is possible by reducing the diode area; however, because of the vertical thickness of the MIM nanodiodes, scaling down below 50 nm appears to be challenging so far. A better way to improve the performance of the device is the use of a low- $k$  material, for example, with a permittivity below the value of 4.8 that we determined for the 3.6 nm thin AlOx.

## CONCLUSION

We have presented a sophisticated large-area nanoscale device fabrication method for the transfer-printing of metal–oxide–metal tunneling nanodiodes which feature asymmetric  $I$ – $V$  characteristics. We showed that the efficient fabrication of several million tunneling nanodiodes within one process step is possible. An implementation to CMOS technology can realistically be considered, as the transfer-printing process is applicable for almost every substrate and the temperature required is rather low. We can tailor the cutoff frequency by varying the diode area, as the general conducting mechanism and the static device parameters are independent of the diode area. The AlOx insulator has a thickness of 3.6 nm and retains its high quality during the printing process, so that even voltages up to 10 V can be applied without destroying the quantum device. The electrical performance of the nanoscale devices is in good agreement

with previously fabricated microdiodes. Numerical simulation shows that direct Fowler–Nordheim tunneling is the main conducting mechanism for the electrons, and static device parameters were extracted. Since the cutoff frequency of the smallest diodes is estimated as 219 THz, MIM nanodiodes are

promising for extremely high-speed electronic and optoelectronic applications. Moreover, the fabrication of these nanoscale devices in dense arrays over large areas provides the possibility to fabricate terahertz rectifiers or also other devices in an economic and fast way.

## METHODS

**Hydrophobization of the Stamp.** The silicon stamp was cleaned and activated by exposing it for 30 s to an oxygen plasma and then placed along with 0.5 mL of 1H,1H,2H,2H-perfluorooctyltrichlorosilane (Alfa Aesar, Ward Hill, USA) into a desiccator at a pressure of 10 mbar for 15 min. This results in the formation of a hydrophobic self-assembled monolayer (SAM) on the surface of the stamp. The SAM was annealed by placing the stamp on a hot plate at a temperature 140 °C for 30 min.

**Metal Deposition onto the Stamp.** A stack of AuPd (15 nm thick) and Ti (4 nm thick) was deposited onto the SAM-covered silicon stamp in a Leybold UNIVEX 450 vacuum evaporator. To minimize the amount of metal deposited onto the sidewalls of the pillars, collimator plates with an aperture diameter of 5 cm were placed over the evaporation sources, and the substrate was placed at a relatively long distance of about 30 cm from the sources. The background pressure during the evaporations was below  $10^{-6}$  mbar and the deposition rate was 0.4 Å/s. Following the deposition of the AuPd/Ti stack, a 25 nm thick Al layer was deposited in a Leybold UNIVEX 300 vacuum evaporator at a background pressure of about  $10^{-7}$  mbar.

**Insulator Growth on the Stamp.** The AlOx insulator was obtained by oxidizing the surface of the 25 nm thick Al layer in an Oxford Plasma Technology reactive ion etch (RIE) system operated at an oxygen pressure of 10 mTorr and with a plasma power of 200 W for 30 s. This results in the formation of a dense, 3.6 nm thick AlOx insulator.

**Metal Deposition onto the Target Substrate.** As the target substrate, a doped silicon wafer was employed. A stack of Ti (4 nm thick), AuPd (25 nm thick), and Ti (4 nm thick) was deposited onto the wafer in a Leybold UNIVEX 350G vacuum evaporator. The background pressure during the evaporations was  $2.5 \times 10^{-6}$  mbar and the deposition rate was 0.4 Å/s. To obtain a uniform film thickness, the wafer was rotated during the evaporations.

**Transfer Printing of MIM Nanodiodes.** The Ti layers on the surfaces of the stamp and the target wafer were activated (hydrophilized) by exposing them to an oxygen plasma for 10 s (stamp) and 2 min (wafer) in a microwave plasma generator operated at an oxygen flow rate of 80 mL/min and with a plasma power of 200 W. The stamp and the substrate were then placed in an Obducat NIL 2.5 nanoimprinter, heated to a temperature of 200 °C, and then pressed against each other for 3 min with a pressure of 50 bar. After allowing the stamp and the substrate to cool to a temperature of 90 °C, the stamp was released, thus transferring the MIM nanodiodes to the target substrate.

**Electrical Characterization of MIM Nanodiodes.** We used an Asylum Research MFP-3D atomic force microscope with an ORCA holder that features two transimpedance amplifiers with a gain of either  $1 \times 10^6$  V/A or  $1 \times 10^9$  V/A. The sample was clamped via the conductive substrate to a gold electrode, which was also connected to the cantilever holder (head), thus creating a complete circuit between the sample and the cantilever tip. We used Ti/Pt (5/20)-coated silicon tips (OMCL-AC240TM, Olympus, Japan) for the measurement. More details on these measurements can be found in the Supporting Information.

**Conflict of Interest:** The authors declare no competing financial interest.

**Acknowledgment.** The authors thank Tatyana Orlova at the Notre Dame Integrated Imaging Facility (NDIIF) for her invaluable help and IMS Chips for their support. The research leading

to these results has received funding from the Institute for Advanced Study (IAS), the International Graduate School for Science and Engineering (IGSSE) at the Technische Universität München, the European Community's Seventh Framework Programme (FP7/2007-2013) under Grant Agreement No. 228673, the NSF through Grant CCF-1129098, and the German Excellence Cluster 'Nanosystems Initiative Munich' (NIM).

**Supporting Information Available:** Theoretical and experimental sections including five figures: S1, theoretical background of metal–insulator–metal diodes; S2, nanotransfer technology; S3, measurement setup for conductive AFM tips; S4, simulation; S5, air-oxidized MIM diodes. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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