

Enhanced Electrical Properties of Lithography-Free Fabricated MoS₂ Field Effect Transistors with Chromium Contacts

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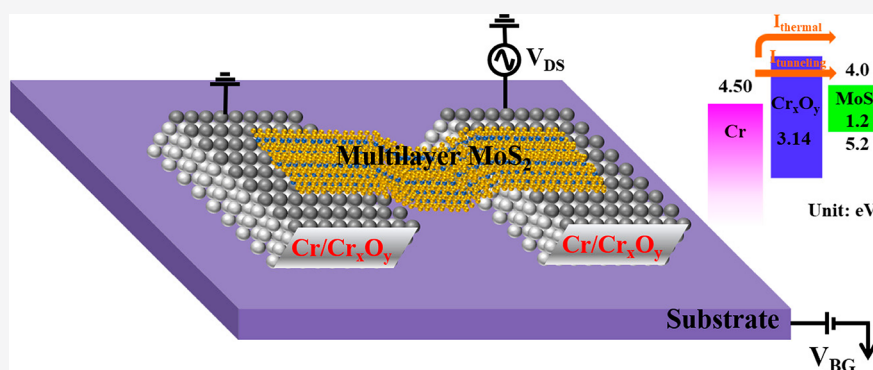
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ABSTRACT: Molybdenum disulfide (MoS₂) as a two-dimensional semiconductor material has been actively explored for field-effect-transistors (FETs). The current prevailing method for MoS₂ FET fabrication involves multiple complex steps, including electron beam (e-beam) lithography, annealing, *etc.*, which are time-consuming and require polymer resists. As a consequence, the MoS₂ exposed to chemicals during the patterning process may be unfavorably affected by residues and the performance of the final FET could be impaired while the annealing limits materials for FETs. Therefore, there is an urgent need to free the fabrication of FETs from e-beam lithography and annealing. In this study, we introduce an e-beam lithography-free method to fabricate MoS₂ FETs by employing maze-like source/drain electrodes. In addition, an ohmic contact in multilayer MoS₂ FETs using chromium (Cr) as source/drain electrodes is achieved without annealing. The underlying mechanism for contact performance is studied, and the tightness of the contact and the type of metal are found to be responsible because they determine the contact resistance. Furthermore, the long-term device degradation is explored, in which the oxidation of metal dominates. The facile fabrication process and mechanism explanation in this work might provide a new platform for future electronic devices.

Field effect transistors (FETs) based on two-dimensional (2D) thin films have attracted tremendous attention in recent years because they might enable next-generation nanoelectronic devices.^{1–6} Molybdenum disulfide (MoS₂), one of layered transition-metal dichalcogenide materials held together by van der Waals interactions, is a particularly promising channel material in the fabrication of electronic and optoelectronic devices because of its high mobility, abrupt switching, large on/off current ratio, and immunity to short channel effects.^{7–12} In addition, preliminary reports also indicate that MoS₂ FETs can be employed for sensing and energy-harvesting applications.^{13–15}

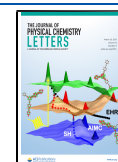
To date, contacts of MoS₂ FETs are commonly fabricated through electron beam (e-beam) or photo lithography on top of MoS₂ flakes, both of which involve multiple wet processing steps where the residue of resists is often present on the surface of the MoS₂ flakes after device fabrication.¹⁶ The resist residuals may unfavorably affect MoS₂ layer quality, thereby causing a significant variation in electrical performance of the MoS₂ FET.^{17,18} To solve this problem, several approaches have

been proposed, and among them, the lithography-free fabrication process is the most prevalent one.^{19–25} Feng's group, for example, reported the use of a dry-transfer technique by transferring a preidentified MoS₂ flake to the designated location on the prepatterned substrate and obtained the desired device geometry.²⁶ As the FETs are fabricated by transferring MoS₂ flakes onto metal electrodes, the contact between metal electrodes and MoS₂ becomes one of the major performance-limiting factors for MoS₂ FETs. To improve the performance of MoS₂ FETs, an ohmic contact is desired at the interface to facilitate carrier transfer. Currently, methods to realize the ohmic contact mainly focus on increasing the

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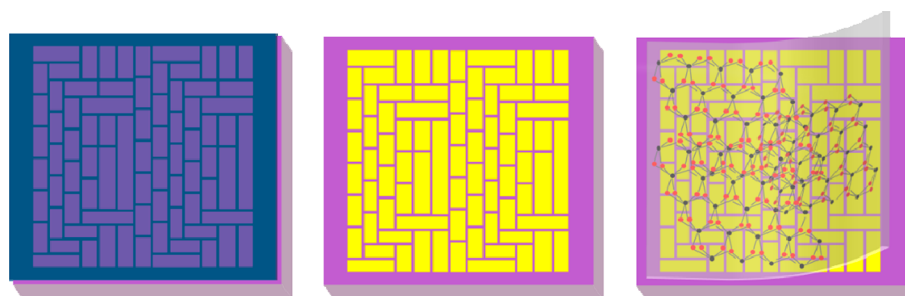


Figure 1. Schematic illustration of the MoS₂-based FETs fabrication process with maze-like source/drain electrode prepatterned wafer. (a) Patterned maze-like source/drain electrodes by photolithography, (b) deposition of the metal electrodes, and (c) mechanical exfoliation of MoS₂ flakes.

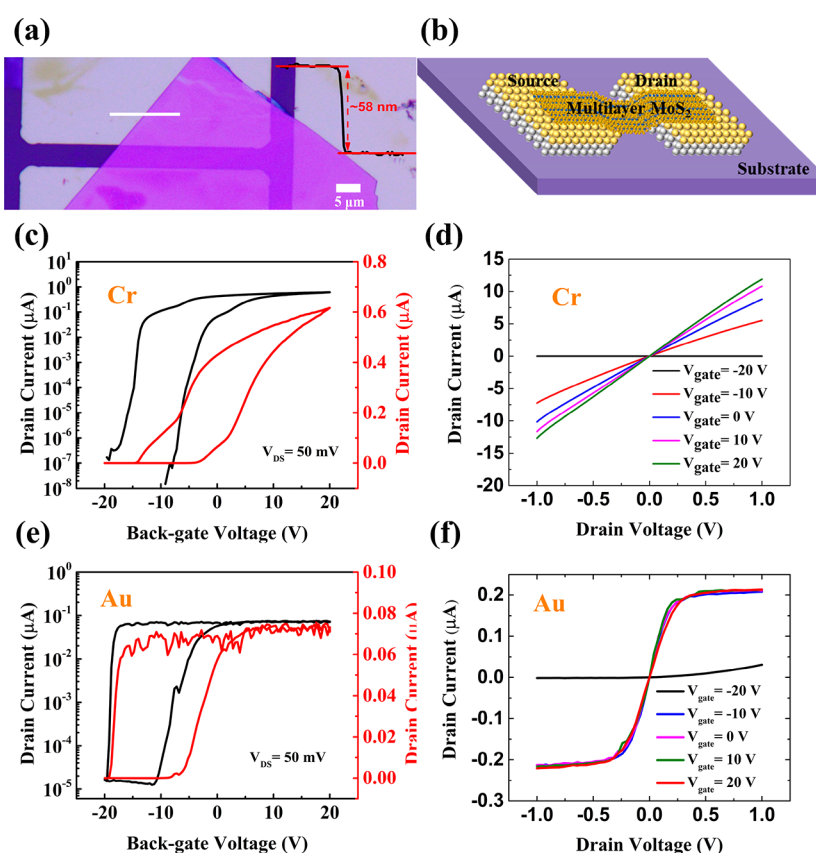


Figure 2. (a) Optical micrograph of a typical multilayer MoS₂ FET with Cr/Au (5 nm/50 nm) as source/drain electrodes. (b) Scheme of multilayer MoS₂ FET with Cr/Au (5 nm/50 nm) as source/drain electrodes. (c) Transfer curves of Cr-MoS₂ FET. (d) Output curves of Cr-MoS₂ FET. (e) Transfer curves of Au-MoS₂ FET. (f) Output curves of Au-MoS₂ FET.

interactions between metal electrode and MoS₂ by annealing,^{26,27} whereas the electrode itself has been less studied.

Herein, we present a simple and e-beam lithography-free approach for the fabrication of multilayer MoS₂ FETs, which not only obviates the undesirable wet chemistry steps but also has a high device yield. Moreover, this approach could be potentially more scalable than the normal lithography methods currently used for MoS₂ electronic and optoelectronic devices. Furthermore, we found that ohmic contact was achieved directly without annealing treatment when chromium (Cr) was used as source/drain electrodes. Our study was complemented by comparing the results obtained from FETs with Au as source/drain electrodes. It was found that the quality of interface, especially intimate contact, played an important role in the formation of ohmic contact. We applied microwave

annealing (MWA) to improve the contact of metal/MoS₂ interfaces to further confirm our conclusion, and results showed that MWA could significantly reduce the Schottky barrier at the MoS₂/Au interface. After MWA annealing at 840 W in nitrogen atmosphere, both the field effect mobility and on-state current of the FETs based on Au electrodes increased by 1–3 orders of magnitude.

In addition, the long-term performance of MoS₂ FET based on metallic Cr contact was studied, where a declined performance over time was observed. Analysis of the device performance and the metal thin film was conducted to explain the phenomenon. It was found that the rapid performance declination of MoS₂ FET mainly resulted from the thickness increase of Cr_xO_y.

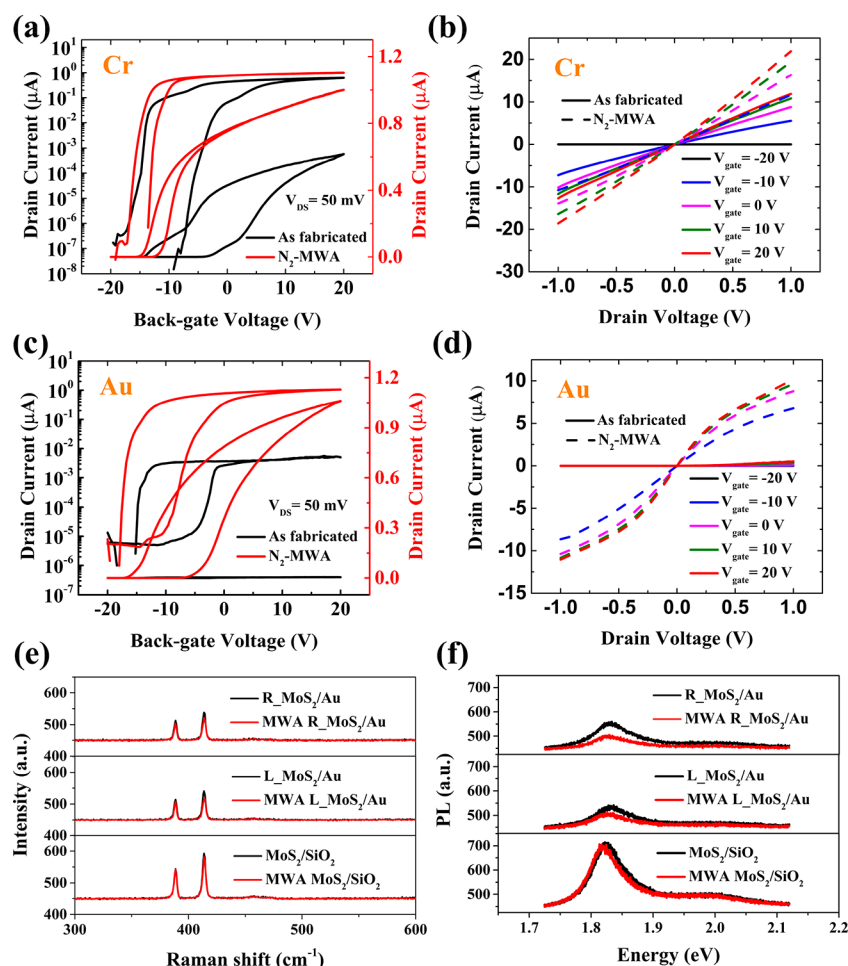


Figure 3. (a) Transfer curves of Cr-MoS₂ FET before and after MWA treatment. (b) Output curves of Cr-MoS₂ FET before and after MWA treatment. (c) Transfer curves of Au-MoS₂ FET before and after MWA treatment. (d) Output curves of Au-MoS₂ FET before and after MWA treatment. (e) Raman measurements for multilayer MoS₂ on SiO₂/Si substrate and on Au electrodes. (f) Photoluminescence (PL) measurements for multilayer MoS₂ on SiO₂/Si substrate and on Au electrodes.

Figure 1 shows the schematic diagram of fabrication processes of MoS₂-based FETs, including a pattern of maze-like electrodes by photolithography (Figure 1a), deposition of the metal electrodes (Figure 1b), and mechanical exfoliation of MoS₂ flakes (Figure 1c). The use of the maze-like source and drain structure can greatly improve the density of the source and drain electrodes in a unit area, thereby effectively improving the yield of fabricated devices. Suitable flakes with uniform thickness were selected by examining the contrast of color under an optical microscope (see the Supporting Information, Figure S1). In the present study, we mainly focused on devices based on MoS₂ thicker than 10 nm rather than few-layer or monolayer ones, because most reported devices used multilayer MoS₂ flakes as channel material.²⁸ Using this simple method, we fabricated MoS₂-based FETs and investigated the contact performance of mechanically exfoliated MoS₂ flakes with Cr and Au. The metal was grown by thermal evaporation (Nano-36 thermal evaporator, Kurt J. Lesker).

Figure 2a shows an optical image of a typical multilayer MoS₂ FET located on a 90 nm thin film of SiO₂ with heavily doped Si as the substrate, the film thickness was measured to be ~58 nm. The corresponding schematic structure of the multilayer MoS₂ FET is shown in Figure 2b. First, we studied the effect of Cr and Au as source/drain electrodes on the

contact performance of the fabricated devices. Cr (20 nm) and a metal stack of Cr/Au (5 nm/50 nm) were used to fabricate the source/drain contacts employing photolithography. The transfer and output characteristics of multilayer MoS₂ FET made of Cr electrodes (Cr-MoS₂ FET) are presented in Figure 2c,d, and that of multilayer MoS₂ FET made of Cr/Au electrodes (Au-MoS₂ FET) are presented in Figure 2e,f. From Figure 2d,f, we can clearly see that an ohmic contact was realized when Cr was used as source/drain electrodes, while Au failed to form ohmic contact with multilayer MoS₂. Using the equation $\mu_{FE} = g_m L / (W C_{OX} V_{DS})$, where L , W , g_m , and C_{OX} are the channel length, width, transconductance, and gate capacitance at $V_{DS} = 50$ mV, respectively, the electric field mobility (μ_{FE}) of FET can be extracted. It was found that the μ_{FE} and on-state current (I_{ON}) of Cr-MoS₂ FETs were larger than that of Au-MoS₂ FETs. It is reasonable to presume that the higher mobility of Cr-MoS₂ FETs resulted from the low contact resistance at the MoS₂/Cr electrode interface, thus leading to a higher carrier injection efficiency. As e-beam lithography was not used in the device fabrication process, a clean channel and contact interface could be obtained. For both Cr-MoS₂ FETs and Au-MoS₂ FETs, the minimum subthreshold slopes (SS_{min}) of the device were at a very low level (see the Supporting Information, Figure S2). In order to explore the key factor in the formation of ohmic contact

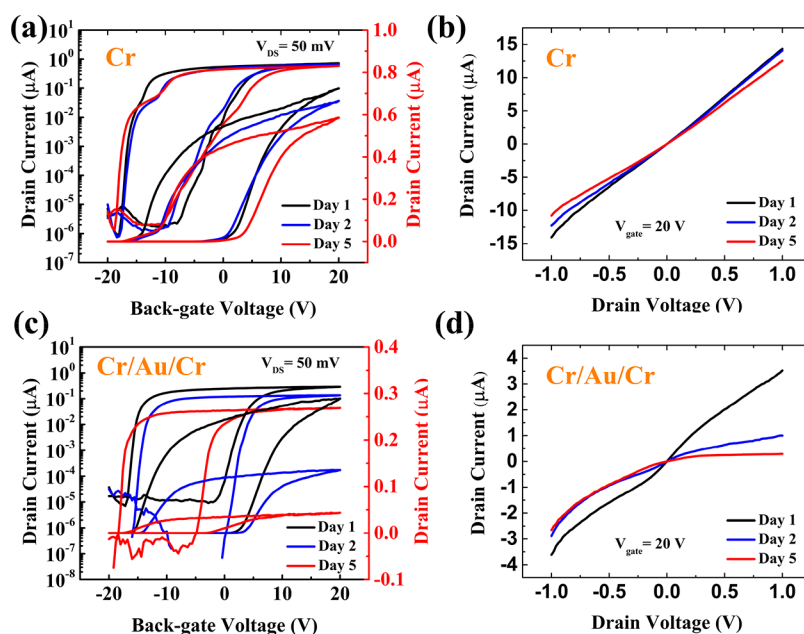


Figure 4. (a) Time dependence of transfer curves of MoS₂ FET using 20 nm Cr as source/drain electrodes. (b) Time dependence of output curves of MoS₂ FET using 20 nm Cr as source/drain electrodes. (c) Time dependence of transfer curves of MoS₂ FET using 5/15/20 nm Cr/Au/Cr as source/drain electrodes. (d) Time dependence of output curves of MoS₂ FET using 5/15/20 nm Cr/Au/Cr as source/drain electrodes.

between metal and multilayer MoS₂, a control experiment using liquid assisted exfoliation to fabricate MoS₂ FETs was conducted (see the Supporting Information, Figure S3). The multilayer MoS₂ fabricated from liquid-assisted exfoliation formed a more intimate contact with Au electrodes.²⁹ As shown in Figure S3b,c, this control multilayer Au-MoS₂ FET exhibited better transfer and output characteristics, indicating the reduced Schottky barrier. This revealed the importance of the intimate contact between multilayer MoS₂ and metal electrodes in the formation of the ohmic contact.

Annealing is widely adopted to improve the quality of contact between transition metal dichalcogenide (TMD) materials and metal as annealing can clean the TMD surface and subsequently induce intimate contact.^{30–33} Here, we used microwave annealing (MWA) to investigate the effect of annealing on the contact performance of fabricated multilayer MoS₂ transistors. Referring to the process parameters of the MWA process from ref 34, the multilayer MoS₂ FETs were annealed in nitrogen atmosphere with 840 W power for 10 min. As shown in Figure 3a–d, the electrical performance of Au-MoS₂ FET was remarkably improved, while there was no significant performance improvement of Cr-MoS₂ FET. After annealing, both the field effect mobility and on-state current of the Au-MoS₂ FETs increased by 1–3 orders of magnitude (see the Supporting Information, Figure S4). The different effect of MWA on Cr-MoS₂ FET and Au-MoS₂ FET could further confirm that the contact between MoS₂ and Cr electrodes was more intimate than that between MoS₂ and Au electrodes; therefore, the MWA process could not significantly improve the electrical performance of the Cr-MoS₂ FET. For the Au-MoS₂ FET, the contact was not that intimate, and the MWA annealing effect was remarkable.

We further used photoluminescence (PL) and Raman spectroscopy to investigate the crystal quality of MoS₂ on top of the Au electrode and that on SiO₂/Si substrate before and after MWA.^{27,35} The nearly identical PL and Raman peak intensities before and after annealing (Figure 3e,f) indicated

that MWA did not change the intrinsic material properties of MoS₂ on SiO₂/Si substrate. In contrast, the decreased PL and Raman intensities of MoS₂ on Au electrodes after MWA revealed the property change. The possible cause for such difference could be the varied effect of MWA on the contact and substrate.³⁶ For the relatively loose MoS₂/Au contact, MWA could make it significantly more intimate and increase the defect states of multilayer MoS₂, thus reducing the PL and Raman peak intensities.^{37,38} For the multilayer and SiO₂/Si interface, MWA has little effect on improving it and the PL and Raman peak intensities showed little variation after annealing. From the above results, we could conclude that surface cleaning and intimate contact dominated the improvement of Au-MoS₂ FET performance. The great difference in the properties between Cr-MoS₂ FET and Au-MoS₂ FET might result from three possible causes: (1) A layer of natural chromium oxide (Cr_xO_y) was formed on the surface of Cr electrode in air, which helped to form a more intimate contact with molybdenum disulfide than Au.^{39,40} (2) The lower work function of Cr reduced the Schottky barrier and contact resistance.¹¹ (3) The naturally formed very thin Cr_xO_y layer lowered the Fermi pinning effect of multilayer MoS₂.

Interestingly, an unusual attenuation of electrical performance of Cr-MoS₂ FETs was discovered during the experiments. The electrical performance of Cr-MoS₂ FET declined remarkably as time went by, and the decline rate was much higher than that of Au-MoS₂ FET. Previous studies have revealed that an ultrathin metal oxide insulator can reduce the Schottky barrier height (SBH), while further increasing the thickness of the insulator introduces a tunneling barrier that leads to the decrease of current.⁴¹ In our experiment, there was a natural chromium oxide (Cr_xO_y) layer between Cr metal and MoS₂ film in the multilayer MoS₂ FET. Therefore, we proposed that it was the increasing thickness of Cr_xO_y over time that contributed to the rapid degradation of the electrical performance of Cr-MoS₂ FET. To validate our hypothesis, we prepared devices with two different metal electrodes: 20 nm Cr

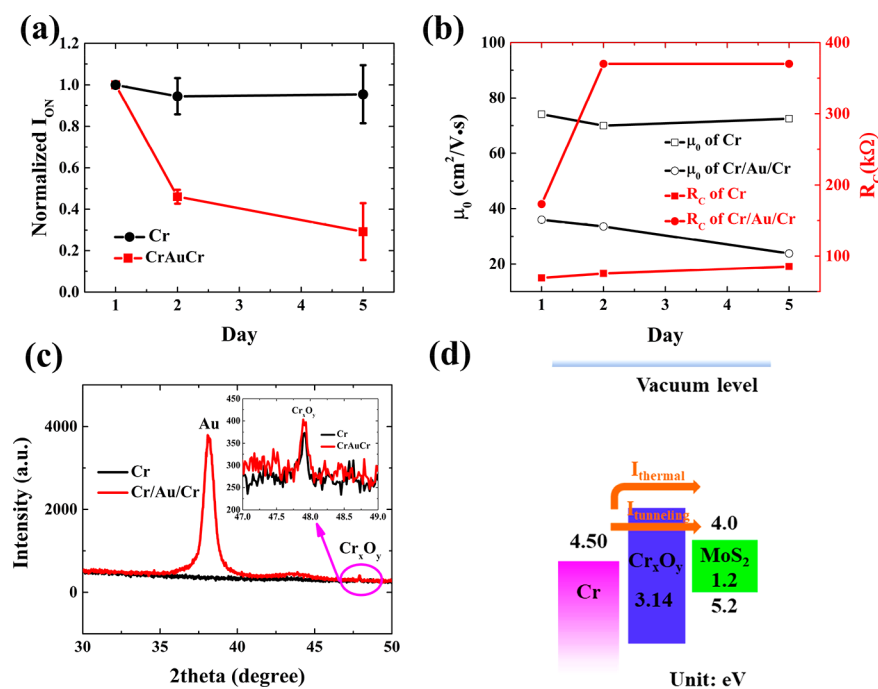


Figure 5. (a) Normalized on-state current (I_{ON}) of Cr-MoS₂ FETs and Cr/Au/Cr-MoS₂ FETs changing against time. Each set of data came from 3 separate MoS₂ FETs. (b) Time dependence of intrinsic mobility and contact resistance of Cr-MoS₂ FET and Cr/Au/Cr-MoS₂ FET. The electrical parameters of Cr-MoS₂ FET and Cr/Au/Cr-MoS₂ FET were extracted from the transfer curves of FETs in panels a and c of Figure 4, respectively. (c) XRD image of Cr and Cr/Au/Cr films on the fifth day. (d) Schematic energy band diagrams of a Cr/Cr_xO_y/MoS₂ structure. All band edge energies are with reference to vacuum; unit: eV.

electrode and 5/15/20 nm Cr/Au/Cr electrode. Figure 4 shows the electrical properties of Cr-MoS₂ FETs and Cr/Au/Cr-MoS₂ FETs in different time periods. It could be found that the degradation rate of electrical properties of Cr/Au/Cr-MoS₂ FETs was faster than that of Cr devices. Figure 5a shows the decay of the on-state current of the two contacts with time, and three FETs were tested for each electrode. It was obvious that the performance of Cr/Au/Cr-MoS₂ FETs declined faster. We also extracted the time-dependent relationship between the intrinsic mobility (μ_0) and contact resistance (R_C) of these two FETs in Figure 4 (for extraction methods of μ_0 and R_C , see note 1 of the Supporting Information).^{42,43} The μ_0 of the FET decreased with time, indicating the declined film quality. However, this still could not explain the sharp decrease of the on-state current, and we further analyzed the contact resistance. The results showed that the R_C of Cr/Au/Cr-MoS₂ FET increased greatly during the next day and then remained relatively stable. However, the R_C of the Cr-MoS₂ FET increased stably for 5 days. We measured the XRD patterns of the Cr film and Cr/Au/Cr film prepared in the same batch of the FETs on the fifth day to determine the reason for the resistance variation. The results showed that the Cr_xO_y peak of Cr/Au/Cr film was stronger than that of Cr film on the fifth day. Therefore, we inferred that Cr on the surface of Au/Cr was easier to be oxidized, and the oxidation process was almost finished on the second day. We also tested the XRD patterns of these two films on day 8 (see the Supporting Information, Figure S5). The Cr_xO_y peak intensity of the two films was basically at the same level and was consistent with the peak intensity of Cr/Au/Cr film on the fifth day. Therefore, it was inferred that the Cr metal could reach a stable oxidation level after 8 days. As mentioned above, the existence of ultrathin Cr_xO_y could reduce gap states and

provide a depinning effect, thus enhancing the electrical performance of multilayer MoS₂ FETs. However, a tunneling barrier would be introduced, and it would become the dominant component when the thickness of Cr_xO_y further increased with time (Figure 5d).^{41,44} More work is still needed to compare Cr_xO_y with different thicknesses to further understand the depinning effect of Cr_xO_y/MoS₂ interface in the future.

In conclusion, we have demonstrated an e-beam lithography-free method to fabricate MoS₂ FET by employing maze-like source/drain electrodes. In addition, an ohmic contact was realized using Cr as source/drain electrodes, while Au failed to form ohmic contact with multilayer MoS₂. After MWA, the electrical performance of Au-MoS₂ FETs was remarkably improved, while there was no significant performance improvement of Cr-MoS₂ FETs. The great difference in the properties between Cr-MoS₂ FETs and Au-MoS₂ FETs might result from three possible aspects: (1) A layer of natural chromium oxide (Cr_xO_y) was formed on the surface of the Cr electrode in air, which helped to form a more intimate contact with molybdenum disulfide than Au. (2) The lower work function of Cr reduced the Schottky barrier and contact resistance. (3) The naturally formed very thin Cr_xO_y layer lowered the Fermi pinning effect of multilayer MoS₂. Furthermore, the long-term device degradation of the Cr-MoS₂ FET was explored, and we found it was the increasing thickness of Cr_xO_y over time that contributed to the rapid degradation of the electrical performance of Cr-MoS₂ FETs. The facile fabrication process and mechanism explanation in this work could offer a new efficient route toward high-quality MoS₂ FETs.

■ ASSOCIATED CONTENT

SI Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acs.jpcllett.1c00231>.

Figures showing optical microscopy image of maze-like source/drain electrodes, statistics of Cr-MoS₂ FETs and Au-MoS₂ FETs in terms of μ_{FE} and SS_{min} , fabrication of Au-MoS₂ FETs using liquid-assisted exfoliation method and electrical performance of a typical Au-MoS₂ FET, statistics of Au-MoS₂ FETs before and after MWA in terms of μ_{FE} and I_{ON} , and XRD image of Cr and Cr/Au/Cr films on the eighth day; supplementary notes, including extraction of intrinsic mobility and contact resistance (PDF)

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Notes

The authors declare no competing financial interest.

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