Graphitic carbon nitride nanosheet (g-C₃N₄-NS) has layered structure similar with graphene nanosheet and presents unusual physicochemical properties due to the s-triazine fragments. But their electronic and electrochemical applications are limited by the relatively poor conductivity. The current work provides the first example that atomically thick g-C₃N₄-NSs are the ideal candidate as the active insulator layer with tunable conductivity for achieving the high performance memory devices with electrical bistability. Unlike in conventional memory diodes, the g-C₃N₄-NSs based devices combined with graphene layer electrodes are flexible, metal-free and low cost. The functionalized g-C₃N₄-NSs exhibit desirable dispersibility and dielectricity which support the all-solution fabrication and high performance of the memory diodes. Moreover, the flexible memory diodes are conveniently fabricated through the fast laser writing process on graphene oxide/g-C₃N₄-NSs/graphene oxide thin film. The obtained devices not only have the nonvolatile electrical bistability with great retention and endurance, but also show the rewritable memory effect with a reliable ON/OFF ratio of up to 10⁵, which is the highest among all the metal-free flexible memory diodes reported so far, and even higher than those of metal-containing devices.

Two-dimensional (2D) materials have attracted considerable attention owing to their unique properties derived from the quantum confinement effect in plane¹⁻⁵. In particular, graphitic carbon nitride (g-C₃N₄), generally presenting a graphite-like layered structure⁶⁻⁷, has proved its importance in photo-/electro-catalysis and bioimaging associated with its tunable band-gap, large surface area and biocompatibility⁸⁻¹⁰. However, g-C₃N₄ possesses a poor electronic conductivity attributed to the band gap of about 2.6 eV and the contact resistance between the nanosheets, which limits its application to a large extent¹¹⁻¹². Nevertheless, the dielectric characteristic is essential for specific devices such as memory diodes¹³, in which the dielectric function of the active layer is highly expected since it could significantly lower the current level at “OFF” state to improve the ON/OFF ratio and thus reduce the risk of misreading during the read operation¹⁴.

Up to now, memory diodes have been studied intensively as a leading candidate for nonvolatile memory and reconfigurable logic applications typically based on the sandwich metal/insulator/metal (MIM) structure¹⁵⁻²⁰, in which a series of metallic oxides (e.g. ZnO, GeO₂, Al₂O₃, TiO₂, MoO₃ etc.) active layers serve as the insulator²¹⁻²⁶, and the ultrapure metals (e.g. Al, Cu, Au, Ag, Pt etc.) or indium tin oxide (ITO) are the widely used electrodes²⁷⁻³². However, these metal-containing materials suffered from the shortcomings of rigidity, high cost and/or limited availability in the earth³³. Consequently, metal-free 2D materials with unique electric or dielectric property have been introduced into flexible, economical and eco-friendly memory diodes. For instance, some promising memory diodes based on graphene oxide (GO) film as the active insulator layer have been presented³⁴,³⁵. However, the device reliability is an inevitable issue due to the instability of active oxygen functional groups on GO³⁶,³⁷. Meanwhile, GO layers have to be coupled with specific metal electrodes to achieve the rewritable function and enhance the ON/OFF ratio³⁸. Compared with GO, g-C₃N₄ possesses not only the improved stability under electrical field or light pulse, but also provides large numbers of lone-pair electrons that helpful for its electronic coupling with nonmetal electrodes³⁹,⁴⁰, which therefore holds the great promise as active layer for the...
construction of high-performance memory devices. As far as we know, however, there was still no report on the memory devices based on the g-C₃N₄ sheets.

Flexible devices present significant advantage for future electronic applications such as portable and wearable electronics. In particular, metal-free flexible memories have merits such as low-cost, foldable and ambient temperature manufacturing. However, one of the most important challenges for fabrication of metal-free flexible memory diodes is that the devices should be supported on nonmetal and bendable electrodes. In this regard, graphene is considered to be a proper substitute owing to its desirable carrier mobility, chemical stability and flexibility. Compared with the graphene obtained by micromechanical cleavage, epitaxial growth, and solvothermal synthesis, GO as the initial material is more compatible with the large-scale solution processing fabrication of memory device. Although the reductions of GO to graphene (also called rGO) are often facing the challenges of high temperature, toxic reagent and/or inefficiency, the recently-developed laser irradiation presents a facile, fast and efficient approach for region-confined reduction of GO to the transparent and flexible rGO film as demonstrated by us and other groups, which provides the new means for electrode fabrication.

Herein, we develop an all-solution processable strategy for metal-free, flexible and rewritable nonvolatile memory device based on the rationally assembled g-C₃N₄ nanosheets (g-C₃N₄-NSs) as the active insulator layer and direct laser writing graphene as electrodes. The as-fabricated memory device shows the nonvolatile electrical bistability and rewritable memory effect with a reliable ON/OFF ratio of up to 10⁵, which, to the best of our knowledge, is comparable to those of metal-based memory devices and better than any other metal-free flexible memory diodes reported previously (Table S1). This fabrication procedure also provides a low-cost, environment-friendly, fast and facile manufacturing process for various electronics beyond high-performance memory devices demonstrated in this study.

Results
Morphology and chemical states of g-C₃N₄-NSs. The bulk g-C₃N₄ was treated by concentrated acid and oxidant to achieve the intercalation and exfoliation as shown in Fig. 1. The obtained g-C₃N₄-NSs presented the size ranging from 20 nm to 50 nm (Fig. 2A and B) and a typical topographic height of about 0.4 nm (Fig. 2C and D), which were significantly smaller than those of the intercalated g-C₃N₄ (Fig. S1 and S2), suggesting that the g-C₃N₄ were exfoliated to atomically thick sheets rather than multi-layers. The thickness of the single layer g-C₃N₄-NSs was slightly larger than the theoretical value of 0.326 nm, most probably due to the presence of functional groups (Fig. 2E).

The X-ray photoelectron spectroscopy (XPS) provided direct evidence of chemical state for g-C₃N₄-NSs. As shown in Fig. 2E, the...
g-C₃N₄-NSs exhibited C 1s, N 1s, O 1s, S 2s and S 2p signals with a N/C atomic ratio of 1.21, close to the ideal g-C₃N₄ composition (N/C = 1.33) and almost the same to that of its parent bulk g-C₃N₄ sample (N/C = 1.23), suggesting that the chemical composition and the coordination of carbon and nitrogen in g-C₃N₄-NSs were retained. But a much higher O peak was observed for the as-prepared g-C₃N₄-NSs than bulk g-C₃N₄, indicating the possible occurrence of oxidation during sample preparation. High resolution C 1s spectrum (Fig. S3A) revealed the existence of C-OH (285.6 eV) and C=O (288.8 eV) and O-C=O (289.3 eV), C-NH (286.8 eV), C≡N and C-N bonds of sp² hybridized carbon in tri-s-triazine rings (287.4 and 288.1 eV)⁹,¹⁰, which, in consistency with the analysis of N 1s and O 1s XPS (Fig. S3B and C), confirmed that the g-C₃N₄-NSs had the intrinsic structure of g-C₃N₄ with oxygen functional groups⁹. In addition, the high resolution S 2p peak (Fig. S3D) verified the absence of S-C and S-N bonds, suggesting that there was no covalent bonding between the H₂SO₄ molecules and g-C₃N₄-NSs⁴⁸.

X-ray diffraction patterns (XRD) of as-prepared g-C₃N₄-NSs presented one predominant (002) peak at ca. 26.6° similar with their bulk counterparts, while the low-angle reflection peak of bulk g-C₃N₄ located at 13.3° disappeared for the g-C₃N₄-NSs (Fig. 2F)¹¹,¹²,¹³. With respect to the (002) peak stemmed from the periodic stacking of layer structure⁹, a slight shift from 27.6° for bulk g-C₃N₄ to 26.6° for g-C₃N₄-NSs occurred, suggesting a similar intrinsic crystal structure as the bulk one but with an increased interlayer spacing due to the presence of functional groups on the surface of g-C₃N₄-NSs. The peak located at 13.3° was originated from the in-planar repeat period of tri-s-triazine units with small tilt angularity in bulk g-C₃N₄, which was absent for g-C₃N₄-NSs due to the decreased planar size and structural defects⁹,⁴⁶, as observed from TEM and XPS mentioned above.

The normalized ultraviolet visible (UV-vis) absorption spectra and photoluminescence (PL) spectra showed an obvious blue shift of the g-C₃N₄-NSs with respect to the bulk g-C₃N₄, suggesting that the band gap of g-C₃N₄ was enlarged (Fig. 2G)⁹,¹⁰,⁴⁶. The band gaps of g-C₃N₄-NSs and its parent g-C₃N₄ were determined from the (ahv)² versus photon-energy plots (Fig. S4) to be about 3.85 eV and 2.61 eV, respectively. Compared with bulk g-C₃N₄, the broadened energy gap of g-C₃N₄-NSs suggested the enhanced dielectricity⁹. It was speculated that this phenomenon was attributed to in-plane electron-confined effect of small-sized 2D nanostructure⁹, electron-withdrawing effect of oxygen-containing groups⁹ and electron-trapping effect of structural defects¹².

### Sandwich structure of rGO/g-C₃N₄-NSs/rGO thin film.

Based on the dielectricity, desirable dispersibility, and abundant functional groups, the g-C₃N₄-NSs present the great potential as active insulator layer for memory diode. For this purpose, we developed a new solution processable approach for constructing the sandwich-structured memory diodes with the graphene electrodes. As schematically shown in Fig. 3, a metal ring of stannum wire was employed as the support to prepare GO thin films from 1.5 mg/mL GO solution (step 1 to 2 and Fig. S5)⁴⁰, which was then transferred onto a poly(ethylene terephthalate) (PET) substrate (step 3) by wet-transferring process⁴⁰. The formed GO film was crackless with some wrinkles (Fig. S6) like the common graphene-based film⁵⁰,⁵¹. The g-C₃N₄-NSs film was formed on GO layer through the sprayed fog of g-C₃N₄-NSs aqueous solution in virtue of a commercial humidiﬁer (step 4). It is worth mentioning that, due to the good hygroscopicity of GO⁴⁴, the GO film would allow the compact contact between g-C₃N₄-NSs fog and GO layer. As a result, a seamless g-C₃N₄-NSs layer was formed and its thickness was limited by the negligible hygroscopicity of g-C₃N₄-NSs surface. (Fig. 4A). Then another GO film was rapidly covered on the g-C₃N₄-NSs layer, where a droplet of ethanol was absorbed in advance (step 5). After dried in warm air of about 60°C for 30 min, the uniform GO/g-C₃N₄-NSs/GO complex film was obtained, which was subsequently converted into the rGO/g-C₃N₄-NSs/rGO (insert of step 6 and Fig. 4B) by direct laser scanning within the confined region (step 6). In this way, both the top and bottom GO layers were reduced simultaneously, while the g-C₃N₄-NSs layer remained laser-transparent. The laser induced rGO electrodes were 3 mm × 1 mm in size. Notably, because of high water swelling rate of GO⁴⁴, these wrinkles on GO film to some extent protected it from cracking during the absorption of g-C₃N₄-NSs fog. On the other hand, compact g-C₃N₄-NSs layer was spontaneously formed between the surfaces of GO films once dried (Fig. 4C) due to their interface compatibility. The whole thickness of the sandwich structure was ca. 150 nm (Fig. 4D), where g-C₃N₄-NSs layer was only ca. 30 nm (Fig. S7). Upon the laser irradiation, GO was highly reduced (Fig. S8), while the g-C₃N₄-NSs layer maintained its morphology and composition (Fig. S9).

### Reversible and reliable electrical bistability of g-C₃N₄-NSs based device.

The current-voltage (I-V) characteristics of the device presented the typical memory effect (Fig. 5A). Initially, the current gradually increased with a negative increase of applied voltage (stage...
When the voltage approached the switching threshold of ca. 4.87 V (stage II), the current abruptly jumped from 7.85 \times 10^{-10} to 4.34 \times 10^{-5} A, indicating the device achieved the write process in the data storage operation by resistive switching from a high resistance state (HRS, i.e., OFF state) to a low resistance state (LRS, i.e., ON state). The current ON/OFF ratio was about 10^5. After the resistive switching, the device retained the ON state during the subsequent sweep (stage III, IV and V). Even if the power was turned off, it still preserved the nonvolatile nature. Impressively, the OFF state could be recovered when the voltage approached the positive switching threshold of ca. 4.01 V (stage VI), implying the function of erasable data storage. Moreover, stage VII and VIII exhibited stable OFF state similar to that of stage I, indicating that the erase process in the data storage operation was achievable in this device.

The ON/OFF ratio determines the misreading probability during the practical operations of the memory devices. Fig. 5B presents an overall comparison of the previously reported flexible sandwich-structure memory diodes with the current work. As can be seen, based on the metal-containing electrodes, and metal oxides or other insulator layers (blue and green bars), few of the devices could reach the high ON/OFF ratios of ca. 10^5–10^6 with rewritable feature. In contrast, for the metal-free devices fabricated previously (red bars), the achieved ON/OFF ratio is only ca. 10^3 at best. As a result, only 8% the studies were focused on the metal-free memory devices (inset of Fig. 5), indicating the extreme challenge for development of the memory diodes with high ON/OFF ratio and outstanding characteristics of flexibility and rewritability. Herein, the developed rGO/g-C_3N_4-NSs/rGO device exhibits the record high ON/OFF ratio for metal-free, flexible and rewritable memory diodes so far.

To investigate the reliability of the memory device, the retention, endurance and flexibility tests were conducted in the ON and OFF states. Fig. 6A showed the retention property of the device for a test period of 5000 s under ambient conditions. This device maintained the ON/OFF ratio at the level of ca. 10^5 without significant variation at a bias of −1 V, indicating the great retention ability. The endurance performance was demonstrated with steady operation for 50 cycles and the resistance values were read out at −1 V (Fig. 6B). Although the current values showed slight fluctuations, the ON/OFF ratio was retained. Obviously, the device exhibited excellent sustainability that might be attributed to the stability of g-C_3N_4-NSs insulator layer, the electrical inertia of the rGO electrode and the compact contact between them.

The flexibility test was performed by the repetitive bending and relaxing of this device with a bent radius of 8 mm for 1000 times (Fig. S10). It was observed that there was no electrical degradation at either ON or OFF state at the reading voltage of −1 V (Fig. 6C). Since the g-C_3N_4-NSs were single layered 2D material, whose self-formed orientation and relative slip caused by stress enabled the strain tolerance, and the graphene films could keep its resistance stable in both the longitude and transverse direction under stretching, the outstanding mechanical endurance of the fabricated device was well deserved.

**Discussion**

To improve the dispersability and tune the dielectric property of g-C_3N_4, we have prepared the functionalized g-C_3N_4-NSs as schematically shown in Fig. 1, which have stoichiometric N/C atomic ratio and exist in the form of single atomic layers. Briefly, bulk g-C_3N_4 was firstly intercalated by sulphuric acid (H_2SO_4). The formed multilayer
advantageous g-C3N4-NSs as active layer, the experimental data were fitted with classical models. In low-bias region, the plot of ln (I) vs. V2/3 from 0 to -0.71 V was fitted to a straight line with Equation S1, suggesting that the conduction mechanism probably arose from the thermionic emission (Fig. S11)76. With the continuous increase of voltage, the conductive behavior changed (Figuer S12) while the device was maintained in OFF state. Fig. S13 showed the ln (I/V2) vs. I/V plot to support the resistive switching based on Fowler-Nordheim (F-N) tunneling model78, where the constant K0 (opposite number of slope of the fitting curve, Equation S4) was measured to be 320.31, implying that the ON state achieved by tunneling current with a reasonable barrier height of ca. 0.8 eV calculated from Equation S5. Therefore, the switching of our device can be attributed to the space charges accumulated at the wrinkles and interfaces between rGO and g-C3N4-NSs. When the traps were filled with electrons beyond ~4.35 V, the tunneling process was occurred and resulted the switching. In addition, the devices fabricated with crossbar model and without active layer further confirm that the memory effect in this work is only attributed to g-C3N4-NSs for active insulator layer (Fig. S14 to S16).

In conclusion, we develop an all-solution processable strategy for metal-free, flexible and rewritable nonvolatile memory device based on the rationally assembled g-C3N4-NSs as the insulator layer and direct laser writing graphene as electrodes. The as-prepared g-C3N4-NSs have atomic thickness, excellent dispersibility, desirable stability and considerable dielectricity. Accordingly, the fabricated memory device shows the nonvolatile electrical bistability and rewritable memory effect with a reliable ON/OFF ratio of up to 105, better than all the metal-free flexible memory diodes reported previously. This device fabrication procedure also provides a low-cost, fast and facile process for manufacturing various electronics beyond high performance memory devices demonstrated in this study.

**Methods**

**Preparation of bulk g-C3N4.** The bulk g-C3N4 was prepared by low temperature thermal condensation of analytical grade melamine. Generally, 10 g obtained powder were then put into a 50 mL alumina crucible, which was subsequently placed in the center region of a quartz tube furnace for heat treatment. The g-C3N4 was obtained after heating melamine at a 600°C for 2 h. After cooling to room temperature, the yellow product was taken out and ground into fine powders in the agate mortar.

**Preparation of GO.** Graphene oxide (GO) suspension is prepared by modified Hummers method as we have reported79. GO films were produced by direct drying the foam of GO captured with metal rings80.

**Fabrication of flexible metal-free memory devices.** The flexible memory devices were fabricated on the PET substrate. Firstly, a GO film was transferred on PET substrate. Then the GO/PET film was covered on a beaker which is filled with g-C3N4-NSs fog (or intercalated g-C3N4 and as exfoliated g-C3N4 in controlled experiment) for 1 min, where the fog was produced by 0.3 mg/mL aqueous solution of g-C3N4-NSs with commercial humidifier. The absorption step was controlled by g-C3N4-NSs fog flow with a flux of ca. 5 mL/min for 2 min. Herein, this step should be repeated 3 times to guarantee that the GO film was completely covered by g-C3N4-NSs. Another GO film was then transferred onto the surface of the g-C3N4-NSs film which was absorbed a droplet of ethanol in advance. Finally, a 458 nm laser with a power of 3 W was employed to write the devices by reducing the GO layers of g-C3N4-NSs/GO structure.

**Characterization.** The morphology of the samples was examined by scanning electron microscope (SEM, JSM-7001F), transmission electron microscopy (TEM, JEOL-2100) and atomic force microscope (AFM, Veeco D3100 atomic force microscope). X-ray photoelectron spectroscopy (XPS) data were recorded on an ESCALAB 250 photoelectron spectrometer (ThermoScientific Scientific) with Al Kz (1486.6 eV). X-ray diffraction (XRD) patterns were obtained by using a
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