Enhanced field emission properties from plasma treated Ti$_3$C$_2$T$_x$ (MXene) emitters

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Abstract

MXenes are an emerging family of 2D transition metal carbides and nitrides and have already shown potential in various applications. However, up to now, studies on the field emission application of MXenes are scarce. In this study, the field emitters based on the Ti$_3$C$_2$T$_x$ (MXene) flakes were prepared by facile solution process and the effect of different plasmas (H$_2$, Ar, O$_2$) on the field emission properties of Ti$_3$C$_2$T$_x$ films was investigated. The plasma treated Ti$_3$C$_2$T$_x$ films showed significantly better field emission properties than that of as-deposited Ti$_3$C$_2$T$_x$ films. Compared to the Ar and O$_2$ plasma treated Ti$_3$C$_2$T$_x$ films, the H$_2$ plasma treated Ti$_3$C$_2$T$_x$ films displayed lower turn-on field (8.5 V/μm) and larger maximum current density (1222 μA cm$^{-2}$). The H$_2$ plasma treated Ti$_3$C$_2$T$_x$ films also showed good emission stability. These results suggested the potential of the H$_2$ plasma treated Ti$_3$C$_2$T$_x$ films as electron source of vacuum electronic devices.

1. Introduction

Electron emission sources are widely used in fields of health care, communication, industrial inspection and materials analysis. They are critical elements of various vacuum electronic devices such as displays [1], x-ray sources [2] and travelling wave tubes [3]. Compared to thermal electron sources, field emission electron sources have advantages of fast switch, pulse emission, miniaturization and operation at room temperature so that they are more promising candidates in the next generation of vacuum electronic devices. So far, field emission properties from cathodes based various 1D materials, such as carbon nanotubes (CNTs) [4, 5], ZnO nanowires/nanorods [6, 7] and BN nanotubes [8], as well as 2D layered materials, such as graphene [9–11], MoS$_2$ [12], WS$_2$ [13], MoSe$_2$ [14] and VS$_2$ [15], have been widely reported. 2D layered materials as field emitters have several advantages including abundant edge structures with a thickness of a few atomic layers, excellent electrical properties and superior mechanical strength. The present of these sharp edge structures can significantly enhance the local electric field, making the electrons more easily tunnel to vacuum.

Since Naguib et al. [16] produced 2D layered Ti$_3$C$_2$ materials from Ti$_3$AlC$_2$ powders (MAX phases) in 2011, a large family of 2D transition metal carbides and nitrides that are called MXenes have received tremendous attention from the scientific community. MXenes have a chemical formula of M$_{n+1}$X$_n$T$_x$ where M is an early transition metal, X is carbon and/or nitrogen, and T$_x$ is surface terminations such as OH, O and F and n = 1 to 3 [16]. MXenes can be synthesized by selectively etching the A element of the MAX phases [17] (M$_{n+1}$AX$_n$, A is IIIA and IVA elements such as Al and Si) using 10%–50% concentrated hydrofluoric acid (HF) [16, 18]. After etching, the A element is replaced by OH, O and F terminations [19]. Alternatively, the concentrated HF etchant can be prepared by a safer route utilizing the reaction between common, inexpensive hydrochloric acid (HCl) and fluoride salts [20]. MXenes have unique properties including metallic conductivity, tunable surface chemistry and wide range of work function strongly depended on the terminations [18, 21, 22]. From experiments and theoretic computations, they have already shown promising potential in a wide range of
applications, such as energy storage (batteries, supercapacitors) [23–25], catalysis [26], conductive electrodes [27], electromagnetic interference shielding [28], photonics [29], transistors [30], sensors [31] and photothermal therapy [32]. Additionally, MXenes can be used as field emitters due to good electrical conductivity and abundant edge structures that act as emission sites. However, up to now, studies on the field emission properties of MXenes are scarce [33]. In this study, we fabricated the Ti$_3$C$_2$Tx (MXene) films using facile solution process and treated the Ti$_3$C$_2$Tx films by plasma. The field emission properties of the Ti$_3$C$_2$Tx films after plasma treatment were examined. The results indicated that the field emission properties of the plasma treated Ti$_3$C$_2$Tx films were significantly enhanced compared to the as-deposited Ti$_3$C$_2$Tx films. In addition, the field emission properties of the Ti$_3$C$_2$Tx films treated by different plasmas, were compared.

2. Experiment

2.1. Sample preparation

Ti$_3$C$_2$Tx was synthesized by etching Ti$_3$AlC$_2$ powders using a mixed solution of HCl and LiF, whose process has been reported in the literature [20, 34]. 1.56 g of LiF was added to 9 M HCl solution (20 ml) to form mixed solution. 1 g of Ti$_3$AlC$_2$ powders (Nanjing XFNANO Materials Co., Ltd) were slowly added to the mixed solution and the mixture was magnetic stirred at room temperature for 30 h. Subsequently, the mixture was washed using distilled water and then centrifugated at 5000 rpm for 15 min. The supernatant was decanted. The above washing process was repeated until the supernatant reached a PH of about 6-7. After that, the sediment (Ti$_3$C$_2$Tx flakes) was dispersed in 50 ml of distilled water. The mixture was sonicated in ice water bath under Ar atmosphere for 30 min and then centrifuged at 3500 rpm for 30 min. The stable supernatant of Ti$_3$C$_2$Tx was collected. The Ti$_3$C$_2$Tx films were prepared by the following process. 5 ml of the Ti$_3$C$_2$Tx solution was diluted by distilled water to obtain a concentration of 2.5 mg/ml. Low-resistance n-type Si substrates were successively ultrasonically cleaned by acetone and alcohol for 10 min. 20 μl of the Ti$_3$C$_2$Tx solution was carefully dropped to the Si substrate by pipette and then dried at 45 °C for 1 h in vacuum, forming the as-deposited Ti$_3$C$_2$Tx films with a circular area of about 0.18 cm$^2$. Subsequently, the as-deposited Ti$_3$C$_2$Tx films were treated by plasma in a microwave plasma chemical vapor deposition device (Nanomaster, NPE4000). The chamber was cleaned and then vacuumized to a pressure of 2 × 10$^{-5}$ torr. After that, the gas was pumped into the chamber to obtain a pressure of about 0.35 torr. Three gases (H$_2$, Ar, O$_2$) were used. The microwave power used to produce the plasma was 1.2 kW. The negative bias applied to the sample stage was 300 V. The plasma treated time was 25 min. After the plasma treatment, all the samples were stored in vacuum for further characterizations and field emission measurements.

2.2. Characterization

The surface morphologies of the Ti$_3$C$_2$Tx flakes, plasma-treated and as-deposited Ti$_3$C$_2$Tx films were characterized by field emission scanning electron microscope (FE-SEM, Carl Zeiss SUPRA 55) and transmission electron microscope (TEM, JEOL JEM-3200FS). X-ray diffraction (XRD, Bruker D8 discover) was used to analyze the phase composition of the Ti$_3$C$_2$Tx flakes. X-ray photoelectron spectrum (XPS, Thermo Fisher ESCALAB 250Xi) was employed to acquire elemental composition of the plasma-treated and as-deposited Ti$_3$C$_2$Tx films. In addition, the field emission properties of the plasma-treated and as-deposited Ti$_3$C$_2$Tx films were measured using a diode configuration in a vacuum chamber with pressure of less than 5 × 10$^{-5}$ Pa. The

![Figure 1. (a) XRD pattern of the exfoliated Ti$_3$C$_2$Tx flakes; (b) TEM image of the Ti$_3$C$_2$Tx flakes. The inset is the high-magnification image.](image-url)
Ti$_3$C$_2$Tx films and ITO-coated glass with a distance of 340 μm were used as the cathode and the anode, respectively. All the measurements were performed using a high-voltage DC power (Keithley 248) combined with a current recording program based on LabVIEW.

3. Results and discussion

Figure 1 (a) shows the typical XRD pattern of the exfoliated Ti$_3$C$_2$Tx flakes deposited on the Si substrate from the Ti$_3$C$_2$Tx solution in the range of 4°–30°. The observed sharp diffraction peaks indicate a relatively good crystalline state of Ti$_3$C$_2$Tx flakes. The most intense peak at 2θ = 6.9° corresponds to the (0002) lattice plane of Ti$_3$C$_2$Tx [35]. When the Ti$_3$AlC$_2$ is etched to Ti$_3$C$_2$Tx, the (002) characteristic peak at 2θ = 9.5° of Ti$_3$AlC$_2$ would

![Figure 2. SEM images of the as-deposited (a) and H$_2$ (b), Ar (c) and O$_2$ (d) plasma treated Ti$_3$C$_2$Tx film.](image)

![Figure 3. The XPS spectra of the as-deposited and plasma treated Ti$_3$C$_2$Tx films.](image)
be clearly seen that the plasma treated Ti3C2Tx flakes were clearly broadened and shifted to a lower angle, which originates from the enlarged layer spacing along c axis and can be explained by the structural expansion due to etching and substitution of Al with OH, F and O terminations [36]. Furthermore, there is no typical sharp peak of TiO2 at 2θ = 25°, indicating that very few Ti3C2Tx flakes are oxidized to form TiO2 [37]. In addition, the (004) and (006) characteristic peak of Ti3C2Tx can be seen at 2θ = 21.7° and 27.4°, respectively. Figure 1(b) displays the representative TEM image of the Ti3C2Tx flakes. The inset is the high-magnification TEM image. Thin 2D layered structures can be clearly observed, demonstrating the formation of Ti3C2Tx flakes from the successfully etching of Ti3AlC2.

Figure 2 presents the SEM images of the as-deposited and plasma treated Ti3C2Tx films. In figure 2(a), the as-deposited Ti3C2Tx films show a homogeneous and flat surface morphology with a small amount of edge structures. After the plasma treatments, all the Ti3C2Tx films (figures 2(b)–(d)) display much rougher surfaces than that of the as-deposited Ti3C2Tx films due to the effect of plasma etching. A large number of randomly oriented edge structures can be clearly observed. These edge structures can act as efficient emission sites and significantly improve the field enhancement of the Ti3C2Tx films in the course of field emission. In addition, compared to the Ar plasma treated Ti3C2Tx films, the H2 and O2 plasma treated Ti3C2Tx films seem to have higher degree of roughness.

Figure 3 shows the XPS spectra of the as-deposited and plasma treated Ti3C2Tx films. For all the four samples, the core level peaks of C, Ti, O and F elements corresponding to C1s, Ti2p, O1s and F1s respectively can be obviously observed. For the as-deposited Ti3C2Tx films, a very small peak of Cl element coming from the etchant (LiF and HCl) can be seen. In the survey spectrum of the as-deposited Ti3C2Tx films, the C1s shows a double peak, indicating that the as-deposited Ti3C2Tx films are covered with graphite-like carbon. The survey spectra of the plasma treated Ti3C2Tx show that the C1s peak on the low binding energy side is removed after plasma treatments, indicating the removal of the graphite-like carbon. It is also clear that the plasma treatment using heavier Ar damages the Ti3C2Tx films since the survey spectrum shows a feature at the low binding energy side of the Ti2p that is not present in the survey spectra of H2 and O2 plasma treated Ti3C2Tx films. Furthermore, the binding energy shift and peak broadening of the O1s peak for the O2 plasma treated samples indicate the formation of TiO2. In addition, It is found that after plasma treatments, the content of F element on the surface of the Ti3C2Tx films decreases significantly, while the content of O element increases. The ratio of atomic percentage of O and F (O/F) is used to evaluate the change of the relative content of these two elements. For the as-deposited and H2, Ar and O2 plasma treated Ti3C2Tx films, the values of O/F ratio are 1.67, 8.88, 4.45 and 8.67, respectively. As previously reported, the work function of Ti3C2Tx is significantly dependent on its surface terminations that strongly affect the surface dipole [22, 38, 39]. Schultz et al [40] demonstrated from the experiments that the Ti3C2Tx with reduced F content (higher O/F ratio) exhibited lower work function, which was basically in agreement with their theoretical calculations. For the F-poor Ti3C2Tx, the experimental work function value from the ultraviolet photoelectron spectra was about 4.0–4.2. This indicates that after the plasma treatments, the work function of the Ti3C2Tx films may be lower, which is favorable for field emission.

Figure 4(a) presents the emission current density (J) as a function of electric field (E) for the as-deposited and plasma treated Ti3C2Tx films. Table 1 shows the summary of the field emission properties of the samples. It can be clearly seen that the plasma treated Ti3C2Tx films show considerably better field emission properties than those of as-deposited Ti3C2Tx films. The turn–on field (required to generate a current density of 100 μA cm−2) of the Ti3C2Tx films reduces from greater than 10.3 V/μm to 8.5 V/μm and the maximum current density increases from 78 μA cm−2 to 1222 μA cm−2. Moreover, compared to the Ar and O2 plasma treated Ti3C2Tx films, the H2 plasma treated Ti3C2Tx films display lower turn–on field and much higher maximum current.
density. For the as-deposited Ti3C2Tx films, the poor field emission properties may be due to the graphite-like carbon covered on the films, as shown in figure 3. After the plasma treatments, the graphite-like carbon is removed and the field emission properties of Ti3C2Tx films are significantly enhanced. Moreover, the heavy Ar and O2 plasma would damage the Ti3C2Tx films in the plasma treatment through preferential sputtering and oxidation, respectively, while the light H2 plasma would burn off the graphite-like carbon without damaging the Ti3C2Tx films. Thus, compared to the Ar and O2 plasma treated Ti3C2Tx films, the H2 plasma treated Ti3C2Tx films show the best field emission properties. In addition, the enhanced field emission properties of the plasma treated Ti3C2Tx film may partly result from the reduced work function that is due to the decrease of F content.

Fowler and Nordheim (F-N) model is further employed to investigated the field emission properties of the Ti3C2Tx films. The F-N model \[41\] can be expressed by following equation:

\[ J = \frac{a(\beta E)^2}{\varphi} \exp\left(\frac{-b\varphi^{3/2}}{\beta E}\right) \]  

where \( J \) is emission current density, \( E \) is applied electric field, \( \varphi \) is work function and \( \beta \) is field enhancement factor. The coefficients are \( a = 1.54 \times 10^{-6} \text{ A V}^{-2} \) and \( b = 6.83 \times 10^7 \text{ V eV}^{-3/2} \text{ cm}^{-1} \). The field enhancement factor \( \beta \) can be calculated by taking natural logarithm of the above equation (1) and expressed as following:

\[ \beta = -\frac{b\varphi^{3/2}}{\kappa_{\text{F-N}}} \]  

where \( \kappa_{\text{F-N}} \) is the slope of F-N plot. Figure 4(b) presents the plots of \( \ln(J/E^2) \) versus \( 1/E \) (F-N plot) for the H2 and O2 plasma treated Ti3C2Tx films. Due to the limitation of current readout accuracy of the measuring equipment, as well as the very low emission current from as-deposited and Ar plasma treated Ti3C2Tx films, the plots of \( \ln(J/E^2) \) versus \( 1/E \) for these two samples are messy and linear fittings are not possible. Thus, they are not included in figure 4(b). It can be seen that both the F-N plots display linear correlation. Here, we assume the work function of Ti3C2Tx is 4.2 eV \[40\]. The field enhancement factors of the H2 and O2 plasma treated Ti3C2Tx films are calculated to be about 1605 and 1425, respectively.

Electron emission stability of field emitters is an important property for practical applications. Figure 5 displays the emission stability of H2 plasma treated Ti3C2Tx films at the initial current densities of 78 \( \mu \text{A cm}^{-2} \) and 194 \( \mu \text{A cm}^{-2} \) over a period of 150 min. At both initial current densities the H2 plasma treated Ti3C2Tx films show good emission stability without obvious current degradation. In addition, similar to other 2D layered

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| Sample          | Turn-on field at 100 \( \mu \text{A cm}^{-2} \) (V/\( \mu \text{m} \)) | Maximum current density (\( \mu \text{A cm}^{-2} \)) | Field enhancement factor |
|-----------------|-------------------------------------------------|-----------------------------------------------|--------------------------|
| As-deposited    | 10.3 (at 50 \( \mu \text{A cm}^{-2} \))         | 79                                            | /                        |
| H2 plasma       | 8.5                                             | 1222                                          | 1605                     |
| Ar plasma       | 10.9                                            | 189                                           | /                        |
| O2 plasma       | 10.0                                            | 428                                           | 1425                     |

Figure 5. Emission stability of the H2 plasma treated Ti3C2Tx films.
materials, such as graphene, the Ti$_3$C$_2$T$_x$ films exhibit spike-like current fluctuations, which are due to the adsorption/desorption of residual gas adsorbates changing the work function, as well as the variation of geometrical factor by reason of ion bombardment from residual gas ionization by current. The good emission stability of H$_2$ plasma treated Ti$_3$C$_2$T$_x$ films may be attributed to the large number of edge structures for electrons emitting that reduce the current from each tip and prevent them from burning out quickly. Furthermore, the large lateral size of the Ti$_3$C$_2$T$_x$ flakes increases the adhesion of the emitters with regard to the substrate, which also contributes to the good field emission stability.

4. Conclusion

We have fabricated the field emitters based on the Ti$_3$C$_2$T$_x$ (MXene) flakes by facile solution process and significantly improved the field emission properties of Ti$_3$C$_2$T$_x$ films by H$_2$ plasma treatment. After the H$_2$ plasma treatment, the Ti$_3$C$_2$T$_x$ films display much lower turn-on field, considerably increased maximum emission current density and good emission stability. Considering their relatively low turn-on field and good emission stability, the H$_2$ plasma treated Ti$_3$C$_2$T$_x$ films show potential as electron source of vacuum electronic devices.

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