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HUMIDITY SENSING PROPERTIES OF $Ti_3C_2T_x$ MXENE FILMS

MXenes are a large family of 2D transition metal carbides, nitrides, and carbonitrides derived mainly through the selective etching of MAX phases with the general formula of $M_{n+1}AX_n$, where M stands for an early transitional metal (Ti, V, Mo, Ta, etc), A is an element of IIIA and IVA groups and X is carbon and/or nitrogen, n=1-4 [1]. MXenes have drawn considerable interest in the field of materials science owing to its good conductivity and other useful properties [1]. In particular, the high surface area and hydrophilic nature of MXenes make this material suitable for sensor applications such as a humidity sensor [2]. Herein, we have demontrated the humidity sensing propetries of MXene films prepared by drop-casting methods. The effect of the thickness of MXene films on their sensitivity to the adsorption of water molecules has been revealed.

The Ti_3AlC_2 powder (MAX phase) was prepared by calcination of the precursors (Ti, Al and TiC) under vacuum in a high temperature muffle furnace LHT4/18 (Nobertherm, Germany). Then, the prepared Ti_3AlC_2 phase was etched in a mixture of LiF and HCl, which react to form hydrofluoric acid. After etching, the mixture was washed several times with deionized water by centrifugation to precipitate the MXene powder. The multilayer MXene particles were then delaminated using LiCl to form a MXene sol. The MXene concentration in the colloid was determined by gravimetry and was about 5.6 mg/ml.

MXene films on glass substrates were prepared by applying a definite volume of the MXene colloid followed by drying under vacuum. The conductivity of the obtained films was about 4000 S/cm. The structure, morphology and thickness of the samples were characterized by XRD and SEM. The resistance of the MXene films during the humidity tests was measured using a multimeter APPA 505 True RMS connected to a computer. The sensing experiments were carried out by rapidly immersing the samples in a chamber with 100 % relative humidity at room temperature.

Successful synthesis of $Ti_3C_2T_x$ MXene particles was confirmed by the absence of characteristic peaks of the Ti_2AlC_2 MAX phase and increased interlayer distance evidenced by shifting of the *00l* peaks toward a lower 2θ value. SEM data showed that MXene flakes stack on top of each other to form a highly conductive film. The thickness of the obtained MXene films was 0.6±0.1, 0.9±0.15 and 1.2±0.2 μm.

Figure 1 shows the change in resistance of the obtained MXene films during humidity sensing experiments. Depending on the MXene film thickness, the resistance increased 20-60 times as the relative humidity (RH) increased from 30 % to 100 %. The resistance was reversibly restored when the relative humidity dropped to 30%. Interestingly, the rise in resistance was significantly slower than the subsequent fall in resistance (Fig. 1). This result indicates a reversible change in resistance due to water adsorption/desorption rather than oxidation of MXene sheets. Moreover, the humidity sensing properties are improved with increasing the film thickness.

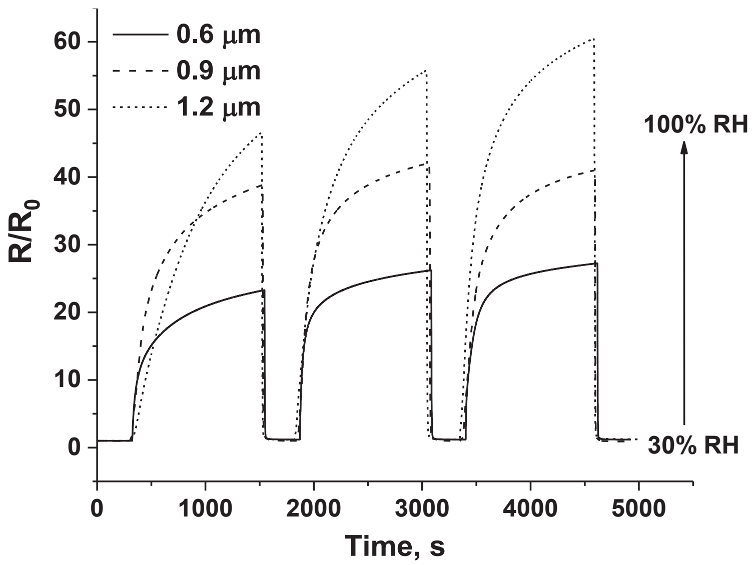

Fig. 1. Dynamic response-recovery curves for the $Ti_3C_2T_x$ MXene films of different thickness to different relative humidity

In line with response-recovery mechanism described in [3], the intercalation of water molecules into $Ti_3C_2T_x$ interlayers leads to an decrease of electrical conductivity of the MXene films. The rise in electrical resistance is associated with the hindered out-of-plane electron transport in swollen MXene films.

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