

Scale-up of MXene Synthesis

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The family of two-dimensional (2D) transition metal carbides and nitrides, MXenes, has been expanding rapidly since the discovery of Ti₃C₂ MXene in 2011 [1]. More than 20 different MXenes have been synthesized, and the structure and properties of numerous other MXenes have been predicted using density functional theory calculations [2]. Two-dimensional (2D) materials with a thickness of a few nanometers or less can be used as single sheets due to their unique properties or as building blocks, to assemble a variety of structures. MXenes properties can be tunable for a large variety of applications [3] that directly lead to their use for electromagnetic shielding [4], transparent conductors, light-to-heat energy conversion, new advanced lasers and photothermal therapy.

Synthesis of MXene typically begins with etching the A-element atomic layers (for example, aluminum) in a MAX phase (for example, Ti₃AlC₂) with HF solution and/or a mixture of fluoride salts and acids at room temperature or slightly higher temperature. After the etching is finished (complete removal of the A-element layers), washing must be applied to remove residual acid and reaction products (salts) and achieve a safe pH (~6). After the pH is increased to ~6, and eventual intercalation of large organic molecules and subsequent delamination completed, the multilayered MXene flakes or single nanosheets can be collected via vacuum-assisted filtration and then dried in vacuum [5]. MXenes can be deposited from solution by spin, spray, or dip coating, painted or printed, or fabricated in a variety of ways. Synthesis conditions used to produce MXenes influence the resulting properties and thus are directly related to the performance of MXenes in their applications [5]. In the laboratory, researchers synthesize MXene in gram quantities, and it is very difficult to repeat the synthesis conditions in order to obtain a material with the same repeatable properties. For scaling up the laboratory process and to obtain material in

larger quantities (up to 200 g per batch) of good quality with repeatable properties, a pilot laboratory line was developed [5]), which allows us to control the etching process and adjust its basic parameters - temperature, mixing speed, recording and storing all necessary data for analysis or to repeat the conditions during subsequent syntheses to obtain a MXene with repeatable properties. In addition, since the acidic etching process is accompanied by the release of heat, a specially developed sealed reactor allows safe and reliable synthesis. The computer control system provides the desired precursor feed rate and the optimal synthesis temperature profile [6].

References

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