



## Experiment Report Form



<b>Experiment title: In situ XRD study of Mxene synthesis using reaction of T3AlC2 (Max-phase) with molten zinc chloride</b>	<b>Experiment number:</b> A20-1-860	
<b>Beamline:</b> BM20	<b>Date of experiment:</b> from: 10 May 2023 to: 15 May 2023	<b>Date of report:</b> 14 August 2023
<b>Shifts:</b> 9	<b>Local contact(s):</b> Christoph Hennig	<i>Received at ESRF:</i>

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### Report:

The goal of this project is to study recently reported MXene synthesis method *in situ* using reaction of T<sub>3</sub>AlC<sub>2</sub> (MAX-phase) with Lewis acid molten salts. This method has huge advantages over previously used MXene synthesis methods, as it does not involve highly dangerous HF. Until our experiments at ESRF the reaction products were studied only using ex situ methods after cooling back to ambient temperature and melt solidification.

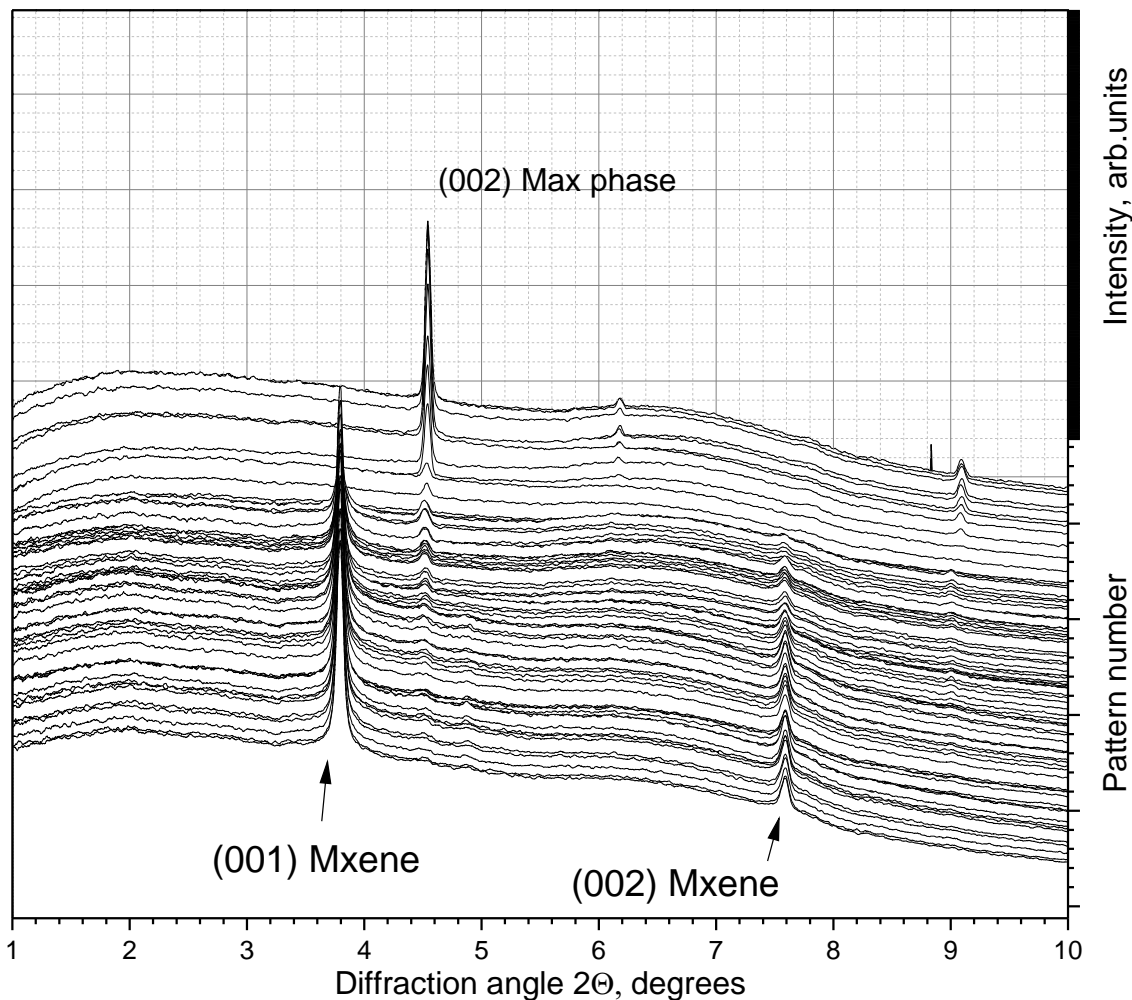
Experiments were performed at BM20 beamline using powder samples of Max phase pre-mixed with powder of ZnCl<sub>2</sub> in different proportions. The samples were heated rapidly over the melting point of ZnCl<sub>2</sub> (290<sup>0</sup>C), above melting point the heating was done with 2 degrees/min up to 550<sup>0</sup>C. Our experiments confirm that the reaction of Max phase with molten ZnCl<sub>2</sub> takes several hours. When the reaction was complete or the changes in structure stopped (~5-7 hours), the samples were cooled slowly to the point of salt solidification and after that rapidly cooled back to ambient temperature. In some experiments the heating was repeated after shifting the capillary to new (not yet melted) spot in order to verify reproducibility.

Summarizing results, experiments were successfully performed with samples of Max phase immersed in molten ZnCl<sub>2</sub> demonstrating expected transformation into new Mxene phase reported in published studies (performed ex situ). The reaction could be observed visually due to formation of bubbles of gases (AlCl<sub>3</sub>) and

change in the sample color. Bubbles disturbed some experiments if the powder sample was moved slightly leading to changes in absolute intensity and background.

XRD patterns confirmed complete transformation of MAX phase into Zn-Mxene which occurs according to proposed mechanism by etching away Al from  $Ti_3AlCl_2$ . New set of XRD reflections was observed to emerge in a sample  $ZnCl_2:Max$  (6:1, g/g) over period of several hours at  $550^{\circ}C$ . The relative intensity of new peaks was increasing during the annealing and intensity of Max phase decreasing. In the best sample we observed complete transformation (Figure.1). After preliminary analysis we have not found any evidence for formation of intermediate  $Ti_3ZnCl_2$  phase (Max phase with Al substituted with Zn). This phase was suggested in some literature reports but evidence by XRD presented in these studies was weak.

We performed also similar experiments with etching Max phase by another salt not yet tested in literature,  $SnCl_2$ . In situ experiments demonstrated that etching of Max phase by  $SnCl_2$  is also possible, but we did not achieved complete transformation into Mxene, some XRD reflections from pristine Max phase were still present even after prolonged high temperature annealing. Additional experiments with larger loading of  $SnCl_2$  are possibly needed to achieve complete transformation.



**Figure 1.** In situ XRD data recorded during annealing of MAX phase in molten  $ZnCl_2$ . The data recorded at  $550^{\circ}C$  (60s per frame) over the period of ~5 hours. Initial pattern shows reflections of MAX-phase, new set of reflections from Mxene emerge and after several hours completely substitutes reflections from precursor phase. Experiment shows co-existence of both precursor and Mxene phase over period of several hours.

More detailed analysis of XRD data will be performed until the end of 2023. Overall we consider our experiment be successful and expect to use collected data for publication. All the goals specified in the proposal were achieved and some additional completely new data recorded.