Circularity of MAX phases: from worn parts and broken samples to 2D functional materials

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1. Introduction

MAX phases are a group of ternary compounds with the general formula $M_{n+1}AX_n$, where M stands for early transition metal, A – group 13 or 14 element and X – carbon or nitrogen atoms. Due to their unique mechanical properties, these materials has been extensively researched for over 20 years and have been proposed for multiple demanding applications [1], where high temperature oxidation and corrosion might occur [2]. By selective etching the A element of MAX phase, 2D structures – MXenes might be crated [3]. Those nanomaterials exhibit excellent functional properties making them promising materials for energy storage, EMI shielding, lubrication and many more applications [4]. As the M transition metals require energy-consuming refinement processes, and the MAX phases synthesis is also conducted by high-temperature synthesis, employing the circularity of MAX phase parts and samples will provide a cost-efficient MAX phase precursor source.

2. MAX phase synthesis

Synthesis of MAX phases is most commonly based on elementary powders mixing and high temperature sintering of said mixture[5]. While different mixing and sintering techniques may be applied, the reaction synthesis between the substrate materials results in crystallization of MAX phase ternary compounds, with some degree of secondary phases, like MX carbides or M-A intermetallic phases. Currently, numerous research papers report work on increasing the synthesis process yield, productivity as well as purity of the MAX phases.

3. Turning bulk sintered parts and worn elements back into powder

In order to create a bulk body, pressure is applied to promote the bonding between powder particles during sintering process. The exerted load upon the sintering tools tend to wear them down, increasing the possibility of their failure and loss of large quantities of costly material (Figure 1).

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Figure 1 Result of a failed sintering densification of MAX phase (about 600g of material)

Turning bulk bodies into powder is mainly conducted using a high temperature atomization process, that melts the material and quickly cools it by means of gas or liquid cooling agent. In the case of MAX phases, melting the material would result in the MAX phase decomposition to MX carbides, due to outward A element diffusion [6]. Such process is also energy-intense, as it requires to heat the substrate material to extreme temperatures in a short period of time. In order to avoid using a high temperature process, crushing may be applied using jaw crusher (Figure 2) in order to turn bulk bodies back into a powder form, ready for further processing.

Figure 2 Laboratory-grade jaw crusher with tungsten carbide crushing jaws

This process may be applied to failed sinters, electrical discharge (ED) machining leftovers (Figure 3), machining scraps as well as worn elements after surface cleaning process. This allows the implementation of fully circular approach that will result in increased cost-efficiency and eco-friendliness of MAX phase products and limit the usage of Critical Raw Materials such as titanium and chromium transition metals.

Figure 3 Leftover sintered preform after ED machining

4. MXene etching

In order to ensure the sustainability of the entire MXene supply chain it is necessary to provide an acid-free A element etching route, that ensures good quality of etched products as well as increased yield of the process. The SAFARI project aims to develop safe and sustainable by design ways to provide MXenes and their hybrid systems with aim to improve various end user applications, such as biodetectors with increased sensitivity, coating with improved EMI shielding performance and more [7].

5. Conclusions

The authors have proposed a cost-efficient route for fabrication of MAX phase precursor powders out of worn parts, failed sinters and process leftovers to be used for 2D MXene etching, reducing the cost of the final products. The crushing process has not affected the purity of the feedstocks in a significant way, confirming that recycled MAX phases may be employed without diminishing the functional properties of the products.

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7. References

[1] J. Gonzalez-Julian, “Processing of MAX phases: From synthesis to applications,” *Journal of the American Ceramic Society*, vol. 104, no. 2, pp. 659–690, Feb. 2021, doi: https://doi.org/10.1111/jace.17544.

[2] X. Lei and N. Lin, “Structure and synthesis of MAX phase materials: a brief review,” *Critical Reviews in Solid State and Materials Sciences*, vol. 47, no. 5, pp. 736–771, Sep. 2022, doi: 10.1080/10408436.2021.1966384.

[3] S. Jin, Y. Guo, F. Wang, and A. Zhou, “The synthesis of MXenes,” *MRS Bull*, vol. 48, no. 3, pp. 245–252, 2023, doi: 10.1557/s43577-023-00491-x.

[4] R. Kumar, S. Sahoo, E. Joanni, and J.-J. Shim, “Cutting edge composite materials based on MXenes: Synthesis and electromagnetic interference shielding applications,” *Compos B Eng*, vol. 264, p. 110874, 2023, doi: https://doi.org/10.1016/j.compositesb.2023.110874.

[5] S. Aydinyan, “Synthesis of Ti2AlC MAX phase and Ti2C MXene by activated combustion,” *Ceram Int*, vol. 50, no. 7, Part B, pp. 12263–12269, 2024, doi: https://doi.org/10.1016/j.ceramint.2024.01.130.

[6] C. Ma, W. YU, Y. Ma, G. Ma, and H. Wang, “Atomic level out-diffusion and interfacial reactions of MAX phases in contact with metals and air,” *J Eur Ceram Soc*, vol. 44, no. 1, pp. 1–22, 2024, doi: https://doi.org/10.1016/j.jeurceramsoc.2023.08.014.

[7] G. Murali *et al.*, “A Review on MXene Synthesis, Stability, and Photocatalytic Applications,” *ACS Nano*, vol. 16, no. 9, pp. 13370–13429, Sep. 2022, doi: 10.1021/acsnano.2c04750.