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Meeting-report

Structural and Morphological Characteristics of Rare Earth Element-based MAX Phase and MXene

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There has been a tremendous effort for the synthesis of crystalline MAX phases—a family of transition metal carbides and nitrides with a chemical formula of Mn+1AXn, where M is an early transition metal, A belongs to groups XIII – XVI in the periodic table, X is either C or N, and n can be in between 1 and 3 [1]. These materials have shown thermal and electrical conductance and have low density and high stiffness which open broad aspects of practical applications. Recently double transition metal MAX phases ($M'_{2/3}M''_{1/3})_2AX$ - out-of-plane ordered, labeled as *o*-MAX and in-plane ordered, labeled as *i*-MAX, were reported [1,2]. However, there are few reports on the synthesis and characterization of *i*-MAX and the corresponding MXenes. In this work, we synthesized Molybdenum-based rare earth containing *i*-MAX phase using arc melt technique. We then removed "A" element of *i*-MAX phase by acid treatment and sonication which resulted in two-dimensional (2D) transition metal carbides called MXenes [3]. These MXenes offer high conductivity, hydrophilicity, magnetism , good dispersion ability in solvents, mechanical stability, and structural diversity with at least 100 stoichiometric MXene compositions and are recognized as multifunctional materials [4-7].

The MAX phase with a nominal composition of $(Mo_{2/3}Er_{1/3})_2AlC$ was synthesized using conventional arc melting in an argon atmosphere with a water-cooled copper hearth. The starting materials are Mo (Thermo Scientific, powder, 99.95 %), C (Sigma Aldrich, powder, 99.99 %), Er (Ames Laboratory), and Al (Alfa Aesar, 99.999 %). First, Mo and C powder were mixed in an appropriate atomic weight ratio using a pestle and mortar to ensure a homogeneous powder and then mechanically pressed. Solid Al and Er were added on top of the pressed powder in the appropriate atomic weight ratio compositions and arc melted in an electric arc furnace. The alloy was flipped and remelted at least 4-5 times to ensure a homogeneous ingot. We then synthesized *i*-MAX $(Mo_{2/3}Er_{1/3})_2C$ MXene from $(Mo_{2/3}Er_{1/3})_2AlC$ *i*-MAX phase. $(Mo_{2/3}Er_{1/3})_2C$ MXene was synthesized by adding $(Mo_{2/3}Er_{1/3})_2AlC$ powders into the LiF/HCl solution and was etched for 7 days and washed using HCl and deionized water. The resulting product was vacuum dried at 70 ^{0}C to remove any solvent residue. It is noted that the surfaces of MXenes are usually terminated with a mixture of F, OH, and O groups during the etching process [6–8]. Structural, morphological, and elemental characterization was carried out using X-ray diffraction on Rigaku at 40 KV, 15 mA and scanning electron microscopy (SEM). For SEM imaging, both $(Mo_{2/3}Er_{1/3})_2AlC$ and $(Mo_{2/3}Er_{1/3})_2C$ samples were sputter coated with carbon and imaged in a JEOL field–emission JXA–8530F EPMA at 20 KV equipped with an SDD X-ray energy dispersive spectrometer (EDS).

Figure 1 shows the XRD patterns of $(Mo_{2/3}Er_{1/3})_2AlC$ MAX phase and $(Mo_{2/3}Er_{1/3})_2C$ MXene. The lowest Bragg peak of the $(Mo_{2/3}Er_{1/3})_2AlC$ powder occurs at 12.95°. After LiF/HCl etching at 70 ^{0}C temperature the peak broadens and shifts towards smaller angle (10.50°) compared to the unetched $(Mo_{2/3}Er_{1/3})_2AlC$, indicating the successful extraction of aluminum atoms from $(Mo_{2/3}Er_{1/3})_2AlC$ [8]. Some $(Mo_{2/3}Er_{1/3})_2AlC$ peaks are still observed in $(Mo_{2/3}\ Er_{1/3})_2C$ indicating the presence of some unreacted MAX phase in the bulk after the etching process. The 39° 2θ peak related to aluminum is relatively small as compared to $(Mo_{2/3}Er_{1/3})_2AlC$. These observations indicate that $(Mo_{2/3}Er_{1/3})_2AlC$ is transformed to $(Mo_{2/3}\ Er_{1/3})_2C$ MXene with some unreacted MAX phase.

Figure 2 shows the representative SEM images with EDS spectra of the $(Mo_{2/3}Er_{1/3})_2AlC$ MAX phase and $(Mo_{2/3}Er_{1/3})_2C$ MXene. We observed more slate/sheet like structures in $(Mo_{2/3}Er_{1/3})_2C$ MXene compared to its MAX counterpart. According to the EDS data, atomic mass percentage of aluminum in $(Mo_{2/3}Er_{1/3})_2AlC$ MAX phase is ~9% whereas it is below 1% after LiF/HCl Etching. This is consistent with the XRD results (Figure 1). Additionally, we observed the presence of fluorine in $(Mo_{2/3}Er_{1/3})_2C$ MXene (Figure 2d) as expected. Our results provide important information regarding the structural and morphological characteristics of rare earth element based *i*-MXenes which dictate the optoelectronic, magnetic, and electrochemical properties of these material [9].

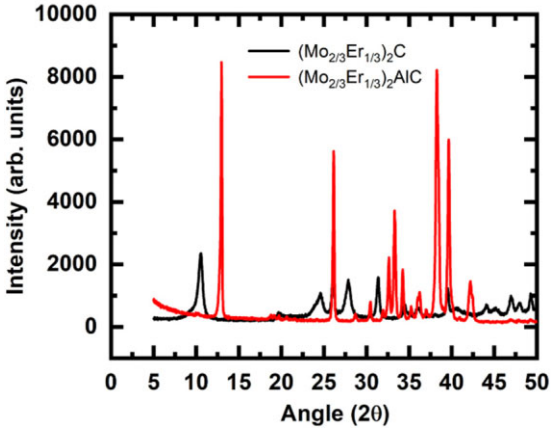

Figure 1. XRD patterns of $(Mo_{2/3}Er_{1/3})_2AlC$ MAX phase and $(Mo_{2/3}Er_{1/3})_2C$ MXene.

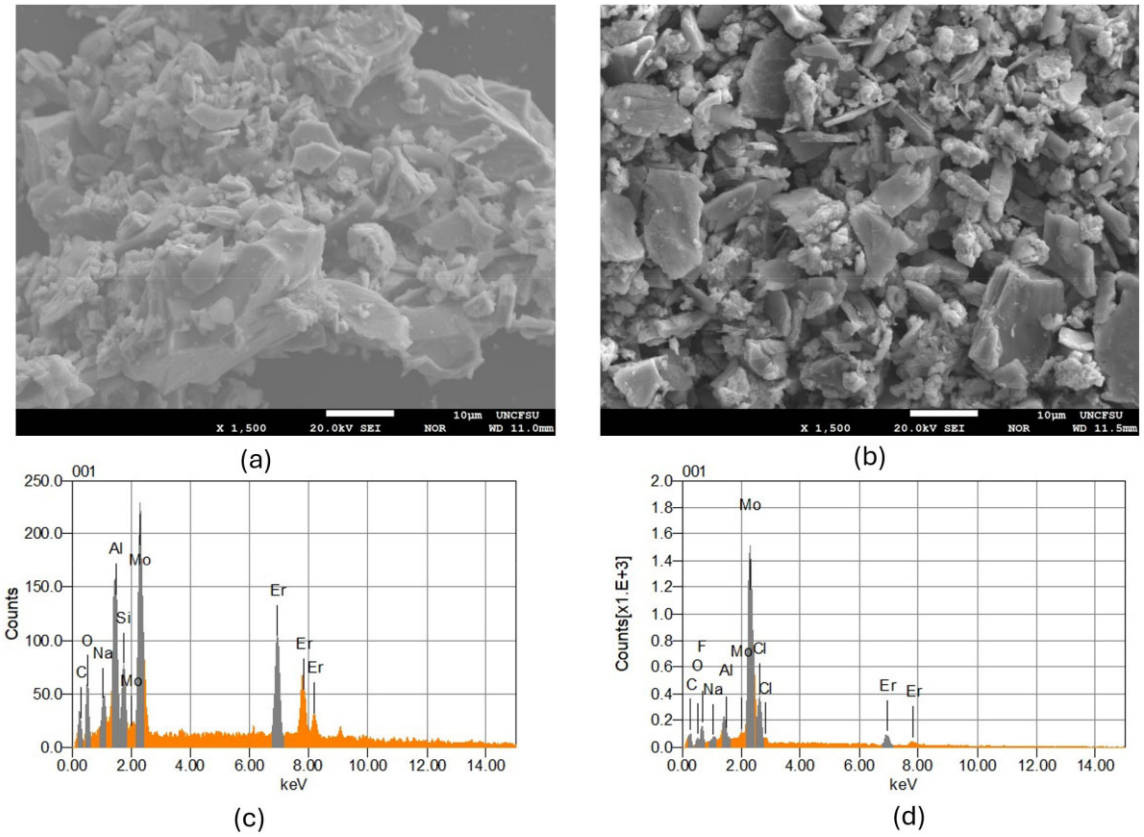

Figure 2. SEM images and EDS of $(Mo_{2/3}Er_{1/3})_2$ AlC (a) and (c) and $(Mo_{2/3}Er_{1/3})_2C$ (b) and (d).

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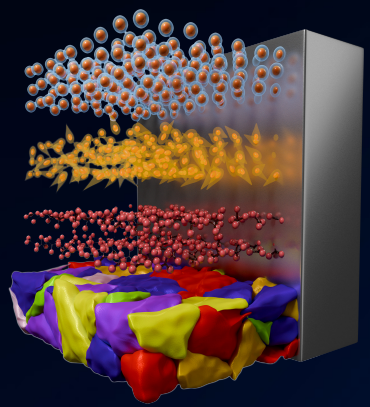

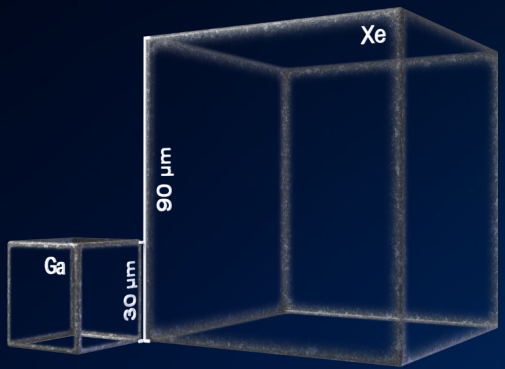

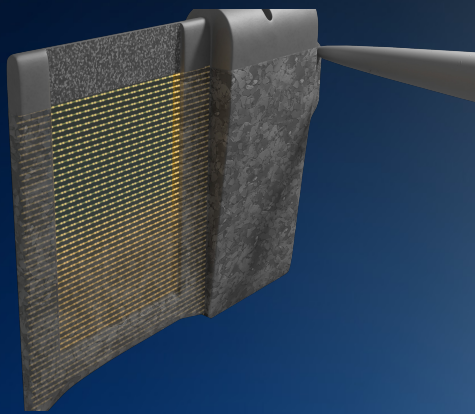

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