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Radiation Stability of Ti_2InC (M_2AX) Nanolaminates Under He Ions Irradiation – Evaluation Through STEM microscopy

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In the 1960s, H. Nowotny and co-workers discovered a family of layered carbides and nitrides, which they called the 'H' phases, now known as the '211' MAX phases (i.e. n = 1) [1]. These carbides and nitrides possess unusual combination of chemical, physical, electrical, and mechanical properties, exhibiting both metallic and ceramic characteristics under various conditions which can be well explained by their anisotropic lamellar microstructure [2]. The hexagonal-layered Ti_2InC compound is a member of the lamellar M_2AX family that crystallizes in the hexagonal structure with space group $P6_3/mmc$.

Here, we report for the first time Ti_2InC (M_2AX) nanolaminates synthesized by an unconventional method utilizing sputtering of individual elements by LEIF (Low Energy Ion Facility) as shown in Fig. 1A. The LEIF facility was utilized without a separation magnet, which means that the MAX phase targets were bombarded with a mixture of Ar^+ and Ar^{2+} ions. the Ar^+ ions were accelerated to the energy 25 kV, the beam current was kept on a high level of 400 μA. The target holder was designed in a specific way in order (i) to keep the MAX phase elements separated and (ii) to rotate the holder with a variable rotation speed based on a sputtering yield of each MAX phase element. The thickness of the Ti_2InC nanolaminate was found to be about 65 nm by EELS. After sputter deposition, the samples were subsequently annealed in vacuum at 120°C for 24 hrs in order to induce interphase chemical interaction and complete formation of the stoichiometrically-correct M_2AX compounds. The as obtained Ti_2InC samples were further investigated for structural and controlled modification by energy ion beams. In order to analyze the radiation stability in the terms of damage profile, the He ions irradiation was performed. A set of as-prepared samples was irradiated with He^+ ions with energy 100 keV up to the fluence 10^{15} cm^{-2} with the beam current 0.5 μA. Another set of the samples was irradiated with He^+ ions with energy 100 keV up to the fluence 10^{17} cm^{-2} with the beam current 3 μA.

Figure 1 (a-f) shows a series of BF, LAADF and HAADF images of no irradiated Ti_2InC nanolamintes at low (Fig.1 a-c) and higher (Fig. 1 d-f) magnifications. As can be seen that the surface of the sample is built by particles organized within the clusters with anisotropic growth. Irradiation with He^+ ions (100 keV, fluence 10^{15} cm^{-2}, beam current 0.5 μA) does not induce significant changes of roughness (Fig. 2a) and hexagonal structure of Ti_2InC (PDF 54-0505) is preserved (Fig. 2b and FFT analysis as inset). Nevertheless, irradiation defects are clearly observed as compared with the untreated sample: lamellar twinning in Ti_2InC crystals (Fig. 2c) and formation of crystalline Ti_2InC core with concentrated point defects and amorphous shell structure (Fig. 2d). The roughness of the Ti_2InC arises when we used with He^+ ions with energy 100 keV, fluence 10^{17} cm^{-2} and beam current 3 μA. Figure 2e demonstrates reduced grain size and structural alteration includes deformed nanocrystals and amorphous segments in the Ti_2InC framework. The formation of amorphous thick layer (over 5 nm) have been produced (Fig. 2g); the achievable thickness was increased in comparison with He^+ ion bombardment with lower fluence. It is important to note that the crystalline nature of Ti_2InC is significantly intact as shown in Fig. 2h. The present results demonstrate suitability of Ti_2InC nanolaminates for radiation and harsh environmental applications.

References:

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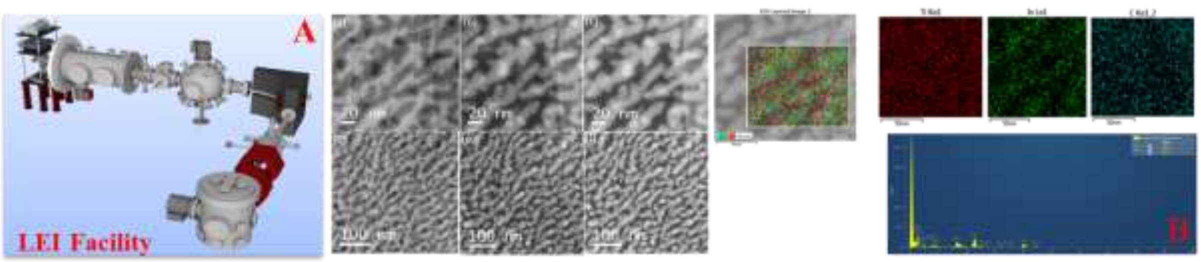

Figure 1. (A) LEI facility for M_2AX preparation, (a-f) a series of STEM – BF, LAADF and HAADF images showing variation of contrast from the same region at different magnifications (B) EDS analysis and elemental mapping confirming Ti, In and C.

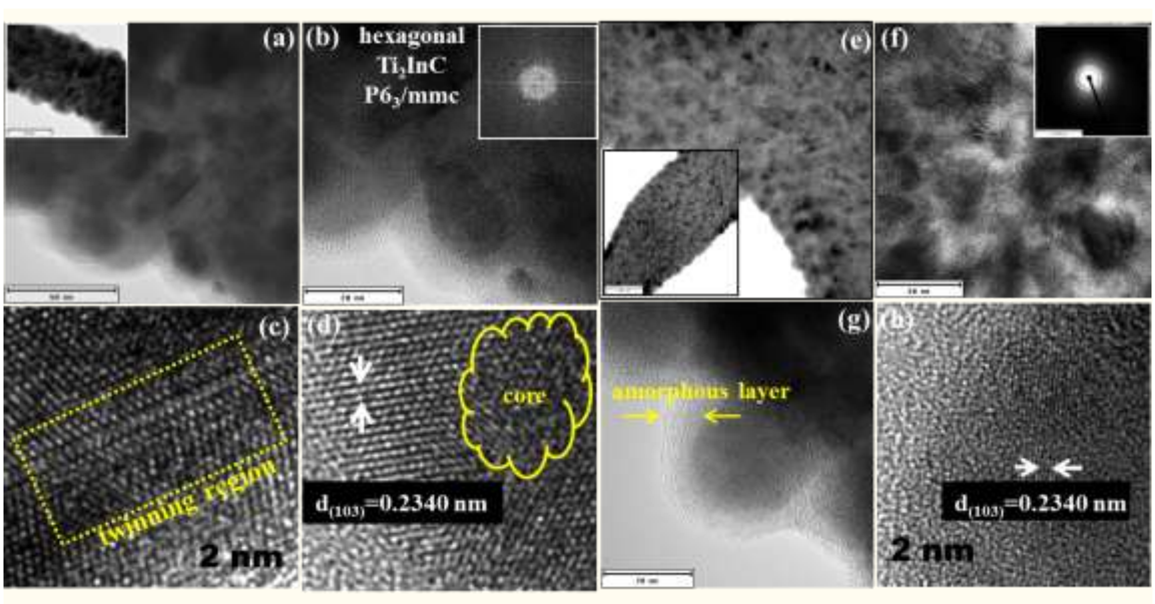

Figure 2. Microstructural evolution of Ti_2InC nanolaminate during irradiation by (a-d) He^+ ions with energy 100 keV up to the fluence 10^{15} cm^{-2} and beam current 0.5 μA (e-h) He^+ ions with energy 100 keV up to the fluence 10^{17} cm^{-2} and beam current 3 μA.