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TiSn and Ti_2SnC Nanolaminates Prepared by Ion Beam Sputtering of Individual Phase Elements: Materials for Future Nuclear Application

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A family of ternary carbides and nitrides, known as MAX phases, combine attractive properties of both ceramics and metals, and has been suggested for potential nuclear reactor applications. The materials show high mechanical damage tolerance in terms of creep, thermal-mechanical fatigue and fracture resistance, and good chemical compatibility with select coolants such as molten lead and sodium [1].

Here, we report for the first time TiSn and Ti_2SnC MAX nanolaminates synthesized by an unconventional method utilizing sputtering of individual phase elements by low energy beam of noble gas prepared in an electrostatic accelerator (i.e., LEIF – Low Energy Ion Facility) equipped with a duoplasmatron-type ion source. 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 MAX nanolaminates was found to be about 40 nm (TiSn) and 50 nm (Ti_2SnC).

After sputter deposition, a set of the samples was subsequently annealed in vacuum in order to induce interphase chemical interaction and complete formation of the stoichiometrically-correct MAX systems. A part of the sample was annealed at lower temperature of 150°C for 24 hrs; another part of the sample was annealed at elevated temperature of 600°C for 2 hrs. The MAX nanolaminates prepared in such a way were further investigated for structural configuration and for controlled modification by energy ion beams. As-prepared samples were irradiated by 35 keV Ar^+ ions in order to analyze the radiation induce damage. The fluence of the ions was picked to 10^{14} ions cm^{-2}. Fig. 1a shows AFM images of as-deposited TiSn film on a Si substrate (no annealed and no irradiated) and TiSn films after annealing at 150°C and 600°C and irradiated by 35 keV Ar^+ ions (Fig. 1b and c). As can be seen that the surface of all films is built by particles organized within the granules (clusters) that are randomly distributed. The important feature is that clusters become bigger as films are annealed and irradiated. AFM technique is used to describe the surface roughness of nanolaminates. It can be observed that the height roughness and interface width values increased as the annealing temperature is increased. This behavior is due to the aggregation of the native grains into the larger clusters upon annealing. The 3D profile confirms that the surface roughness in post irradiated TiSn nanolaminates increases from 3.27 to 7.06 nm in comparison with as-deposited and not processed sample (Fig. 3b and c).

Young´s modulus (E/GPa) and hardness (H/GPa) of TiSc nanolaminates were measured by nanoidentation technique. It was found that the E value of nanolaminates decreases with temperature and after ion bombarding. The H value decreases with temperature and with ion bombarding also. SEM images of TiSn samples with results from nanoidentation as insets are presented in Fig. 2a-2f. The MAX nanolaminates should, however, exhibit an extraordinary radiation resistance. The experimental test based on analysis of the irradiated TiSn and Ti_2SnC nanolaminates using nanoindentation technique, SEM and AFM analyses confirmed that the intense bombardment of the samples by heavy Ar^+ ions induced only small structural change and negligible alteration of the associated physical properties which could predict their application in nuclear and extreme environmental conditions [2].

References:

[1] E.Hoffman *et al,* Nucl E Des **244** (2012), p. 17.
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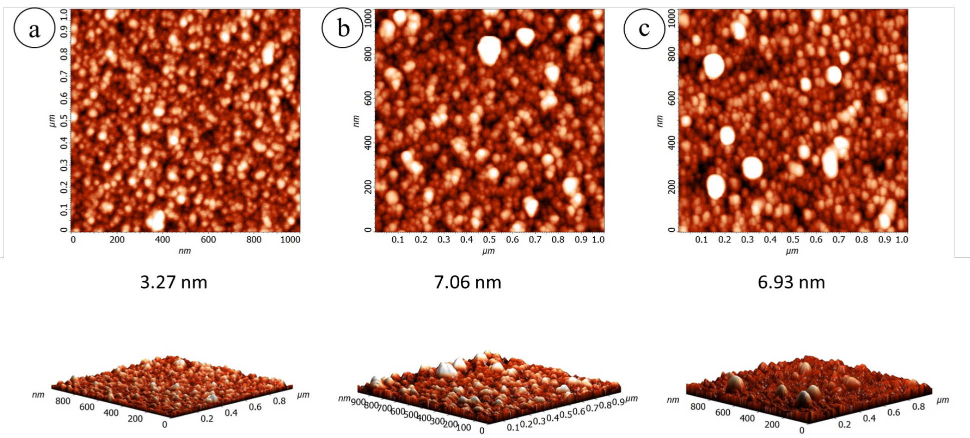

Figure 1. AFM images of TiSn films a) as-deposited film no annealed and no bombarded, b) annealed at 150C° and bombarded with 10^{14} Ar ions and c) annealed at 600°C and bombarded with 10^{14}Ar ions.

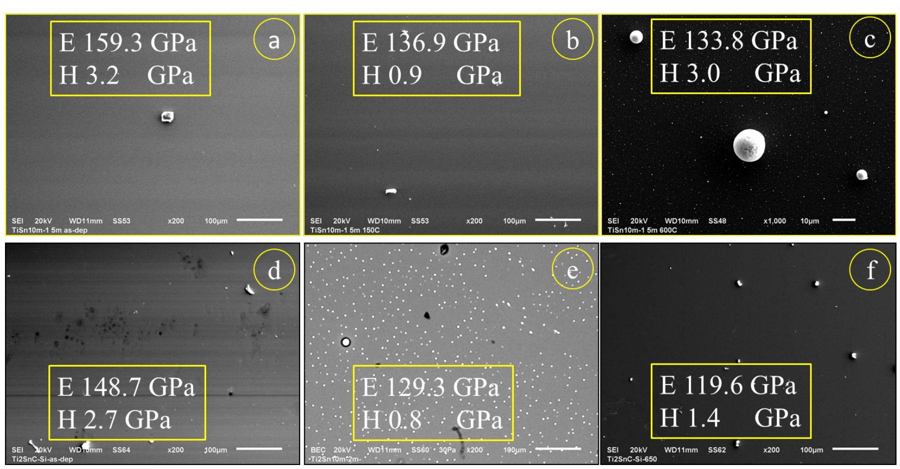

Figure 2. SEM images of TiSc nanolaminates: (a-c) before irradiation, an inset E and H data before irradiation, (d-f) after irradiation, an inset E and H data after irradiation.

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