Radiation Resistant Layered Ti$_3$AlC$_2$ Ceramics Prepared by LEIF

Snejana Bakardjieva$^1$, Jiri Vacik$^2$, Robert Klie$^3$ and Xue Rui$^3$

1. Institute of Inorganic Chemistry of the Czech Academy of Sciences, 250 69 Rez, Czech Republic.
2. Nuclear Physics Institute of the Czech Academy of Sciences, Rez 205 68, Czech Republic.
3. University of Illinois at Chicago, Department of Physics, 845 W Taylor Str., 60 607 Chicago, USA.

* Corresponding author: snejana@iic.cas.cz

M$_{n+1}$AX$_n$, where M is the transition metal, A is the preferentially subgroup IIIA or IVA element of the periodic table, and X is carbon or nitrogen are known as oxygen-free compounds possessing a layered structure bridging the gap between metals and ceramics. Analogously to their corresponding binary carbides and nitrides (MX), the MAX phases are elastically stiff, good thermal and electrical conductors, resistant to chemical attack, and have relatively low thermal expansion coefficients. Like ceramics, they have a high melting point, and they are sufficiently stable at elevated temperatures up to 2000 °C [1].

MAX phase with composition TiAlC attracted attention due to their excellent thermal shock resistance and damage tolerance [2]. However, TiAlC was most complicated phase to study due to the difficulty of synthesis - long synthesis time, high pressure, impurity owing to reactants and stops of satellite phases. In this research, single-phase Ti$_3$AlC$_2$ compound was prepared by an unconventional method utilizing sputtering of individual elements by LEIF (Low Energy Ion Facility). The LEIF facility was utilized without a separation magnet, which means that the M$_3$AX$_2$ 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 to (i) keep the Ti$_3$AlC$_2$ phase elements separated and (ii) rotate the holder with a variable rotation speed based on a sputtering yield of each Ti-Al-C phase element. After sputter deposition, the samples were subsequently annealed in vacuum at 200 °C for 20 hrs in order to induce interphase chemical interaction and complete formation of the stoichiometric corrected Ti$_3$AlC$_2$ compound.

The as obtained Ti$_3$AlC$_2$ material was 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 Ar$^+$ ions irradiation was performed. As-prepared samples were irradiated by 35 keV Ar$^+$ ions in order to analyze the radiation induce structural deformation and radiation stability in the terms of damage profile. The fluence of the ions was picked to $10^{14}$ ions cm$^{-2}$.

Figure 1 (left panel) illustrated the morphology of the pristine Ti$_3$AlC$_2$. According HRTEM and FFT observations as prepared material is amorphous. In addition, small spherical particles with size of approx. 20 nm were unhomogenously distributed on its surface. The right panel of Fig 1 presented effects of Ar$^+$ irradiation on Ti$_3$AlC$_2$ structure. Precipitation process was occurred and irregular shaped grains are generated. One can observe small matrix precipitates with highly irregular morphology (Fig.1a and Fig 1d) formed within the material. HRTEM image (Fig. 1b and Fig. 1e) and corresponding FFT analyses (Fig. 1c and Fig. 1f) revealed that precipitates are in the form of hexagonal Ti$_3$AlC$_2$. Radiation induced number of changes in Ti$_3$AlC$_2$ microstructure - integrated nanocavities (Fig. 1g) and curved dislocation lines are clearly visible (Fig. 1h a and Fig.1j).

The present LEIF method with Ti, Al and C target as starting materials demonstrates advantages of shorter processing time, lower temperature and higher product purity (EDS analysis of sample before
and after Ar\(^+\) irradiation established the same stoichiometry. The modern view about the features of the three-component Ti\(_3\)AlC\(_2\) layered system are considered in this work. The Ti\(_3\)AlC\(_2\) possesses promising combination of properties to be used in harsh environmental conditions.

References:

[3] The authors acknowledge funding from the Czech Science Foundation (Project GACR 18-21677S).

**Figure 1.** Morphological evolution of Ti\(_3\)AlC\(_2\) before Ar\(^+\) ion irradiation (right panel) and after Ar\(^+\) ion irradiation (left panel). The EDS spectra are also included.