

Sintering and Nanoindentation of Ti_2SnC (M_2AX) Ceramics – Attractive Materials in the Topic of Nuclear Engineering

Snejana Bakardjieva¹, Jiri Vacik², Jiri Plocek¹, Pavel Horak², Jiri Nemecek³ and Robert Klie⁴

¹. Institute of Inorganic Chemistry of the CAS, Husinec - Rez 1001, 250 68 Rez, Czech Republic.

². Nuclear Physics Institute of the CAS, Husinec – Rez 130, 250 68 Rez, Czech Republic.

³. Czech Technical University in Prague, Faculty of Civil Engineering, 166 29 Prague, Czech Republic

⁴. University of Illinois at Chicago, Department of Physics, 845 W Taylor Str., 60 607 Chicago, USA.

MAX phases are a group of ternary carbide or nitride phases with a nano-layered microstructure. Their general formula is $M_{n+1}AX_n$ with $n=1$ to 3, where M is a transition metal, A is an A group element (from IIIA to VIA), and X is either carbon or nitrogen [1]. The microstructure of alternating A- and XM_6 polyhedral layers gives rise for a unique combination of properties of metals and ceramics. Like a ceramic, MAX materials exhibit a high stiffness at room temperature, a high strength at high temperature, a good resistance to thermal shock and oxidation as well as crack healing ability. The goal of this study is to investigate the microstructure and mechanical properties of the Ti_2SnC with layered crystal structure as one of the most fascinating “211” member of MAX phases.

Ti_2SnC MAX phase was synthesized by Ti (powder), Sn (thin foil), and TiC (powder) mixed and grinded in agate mortar in molar ratio 1:0.8:0.9. The material was pressed to pellet with diameter 1.3 cm with force 100 kN. The pellet was annealed in vacuum at 1200°C for 15 min; as-obtained pellet was milled and heated again with the same regime [2]. Microstructural data were obtained by XRD, SEM and HRTEM/SAED/EDS analyses. The nanoindentation was provided by means of Hysitron Tribolab Ti-700 instrument. Young's moduli were calculated from unloading parts of the penetration curves.

Powder XRD revealed that Ti_2SnC is the major phase and some residuals from Sn and $TiC_{0.5}$ are occurred also. The crystallite dimension was calculated using Scherer equation and shape anisotropy was observed; the crystallite size of 120 x 250 nm was determined. Detailed crystallite size analysis was performed by HRTEM measurement which confirms an average size dimensions deduced by Scherrer formula. Fig.1a shows a TEM micrograph where heterogeneity in size and shape can be observed. A HRTEM micrograph of an irregular particle, with dimension higher than 200 nm, is shown in Fig.1b. High magnification of red boxed part confirmed the presence of small Sn nanoislands and amorphous layer with thickness of 10 nm (Fig.1c). A HRTEM micrograph of blue boxed part revealed ceramic material with layered structure (Fig.1d) and highly ordered periodical lattice fringes with $d_{(012)}$ spacing of 0.32 nm, which matches well with the hexagonal Ti_2SnC phase (Fig. 1e) [3]. The EDS spectrum of the Ti_2SnC material sintered at 1200°C (Fig.1/a₁) confirmed Ti, Sn and C elements. Fig. 2a shows SEM image of Ti_2SnC with fully dense microstructure. The cyclic nanoindentation verified that the material is very compact and the occurring inelastic hysteresis leads to the influence of the elastic and hardness parameters. The excellent mechanical properties of Ti_2SnC material could be attributed to solid solution strengthening effect, grain size effect and formation large amount of dimples and kink (Fig.2b). The composite with nanoscale microstructure exhibits a HV (Vickers) of 492.16, EIT of 133.45 and HIT of 5.31 (GPa), respectively (Fig.2c). We can conclude that as prepared Ti_2SnC material offered a high microstructural potential allowing its application at extreme and nuclear conditions.

References:

- [1] M.W. Barsoum, Prog. Solid State Chem. 28 (2000), p.201.
 [2] Shi Bo Li, G.P.Bei, H.X. Zhai, Y. Zhou, Mater. Letters, 60 (2006), p.3530.
 [3] C.Hu, H.Zhang, F.Li, Q. HuAng, Z. Bao, Inter J refract Met and Hard Mater, 36 (2013), p.300
 [4] The authors acknowledge funding from the Czech Science Foundation (Project/GACR 18-21677S).

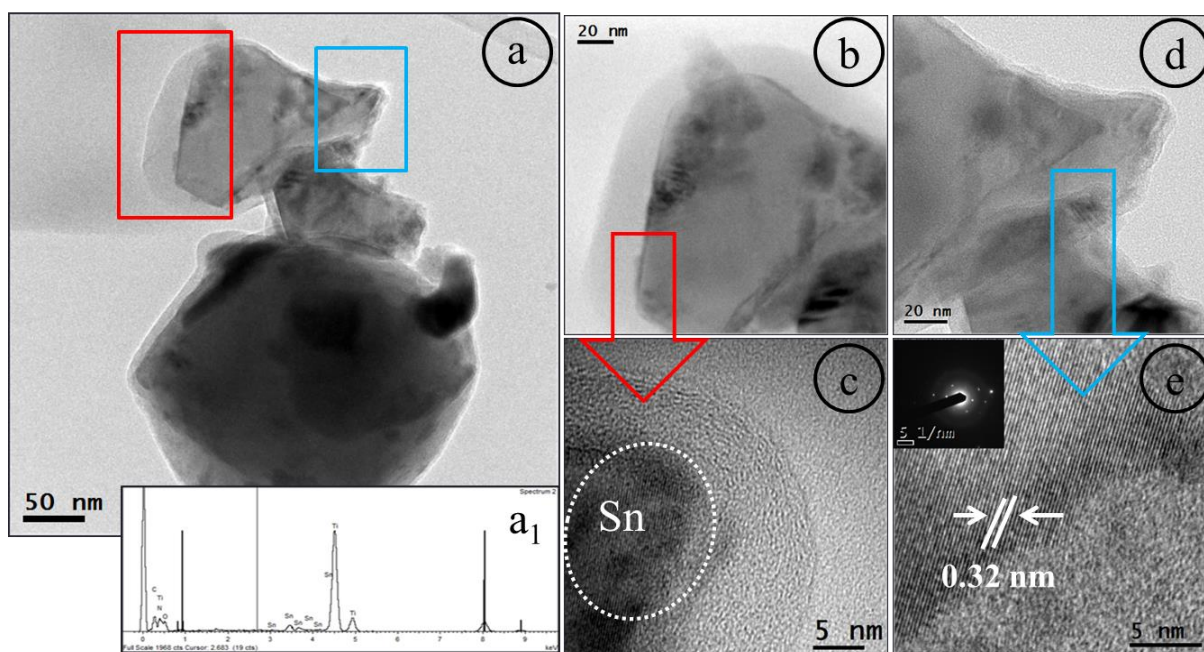


Figure 1. HRTEM study of Ti_2SnC sample: a) Low magnification, b) and c) High magnification of red boxed part, d) and e) High magnification of blue boxed part, a₁) EDS spectrum of Ti_2SnC .

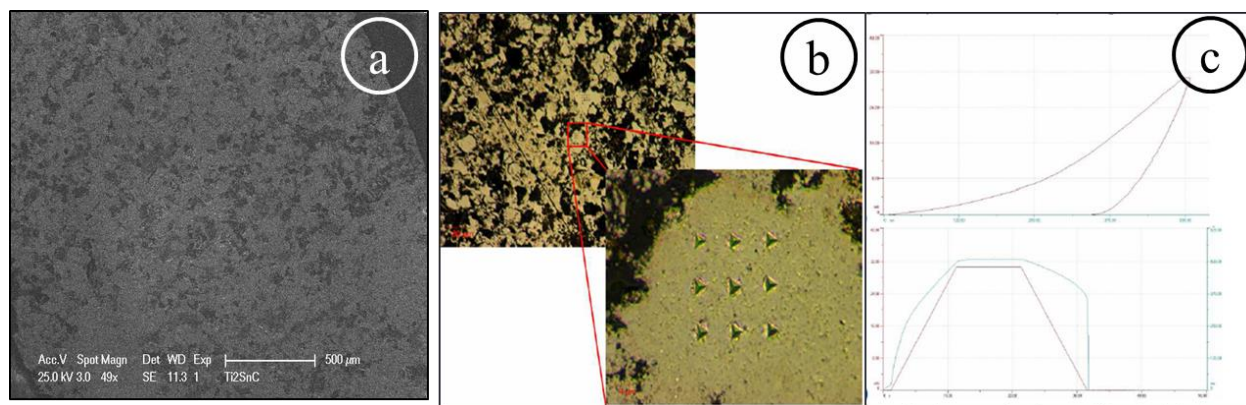


Figure 2. Morphology and microstructure of Ti_2SnC sample obtained by a) SEM microscopy and b-c) Nanoidentation experiment.