doi:10.1017/S1431927618000715
Microsc. Microanal. 24 (Suppl 1), 2018

Structural Determination of Gr_2GaC Nanolaminates by Electron Microscopy: Resistant Materials Under Thermal Damage

Snejana Bakardjieva[1], Jiri Plocek[1], Jaroslav Kupcik[1] and Jiri Vacik[2]

[1.] Institute of Inorganic Chemistry of the CAS, Husinec, Rez, Czech Republic.
[2.] Nuclear Physics Institute of the CAS, Husinec, Rez, Czech Republic.

The MAX phases are a new and exciting class of carbides and nitrides that bridge the gap between properties typical of metals and ceramics, while offering fundamentally new directions in tuning the structure and properties of ceramics for emerging applications [1]. The term "MAX phases" was coined in the late 1990s and applies to a family of 80+ ternary carbides and nitrides that share a layered structure. They are so called because of their chemical formula: $M_{n+1}AX_n$ where n = 1, 2, or 3, where M is an early transition metal, A is an A-group element (specifically, the subset of elements 13–16), and X is carbon and/or nitrogen. Nowotny and coworkers [2] discovered most of these phases in powder form roughly 50 years ago. However, Barsoum and El-Raghy's [3] report in 1996 on the synthesis of phase-pure bulk Ti_3SiC_2 samples and their unusual combination of properties catalyzed renewed interest in them. Since then, research on the MAX phases has exploded.

In this report, we investigate the microstructure of Gr_2GaC (M_2AX) phase by an electron microscopy. The Gr_2GaC was prepared from direct reaction between the constituent elements in quartz ampoule. After evacuation and sealing off, the sample was heated in encapsulated furnace at 1300°C for 32 hrs. The microstructure was understood by taking into account the growth processes of laminates or by the self – organization processes including segregation effects of the elements on a laminate surface. The effects of individual microstructural features like grain size, defect density, phase arrangements in a two - dimensional manner on the mechanical properties are evaluated. Because some MAX phases currently are being considered as a new class of ultra-durable materials, resistant against thermal damage, here we discussed the microstructural changes of Gr_2GaC during a thermal treatment. The analysis of post-sintered states was performed by HRTEM microscopy (JEOL JEM 3010 with laB_6 cathode). The stability of microstructures under severe condition was predicted.

HRTEM image in Fig. 1a show the size and shape of an individual Gr_2GaC particle. SAED pattern (inset in Fig.1b) reveals crystal with hexagonal symmetry (P63/mmc). High magnification image (Fig.1c) indicates that Gr_2GaC has multilayered structure. The bending of Gr_2GaC layers is attributed to the introduction of defects formed under annealing. The high temperature treatment leads to the wave–like undulations of Gr_2GaC planes with curvature, but no visible damage to layers is observed. Performed EDS analysis confirms Cr:Ga:C elements in ratio 2:1:1 (Fig.1d). The fringe spacing of 2.49Å corresponds to (100) lattice plane of Gr_2GaC (PDF ICDD No 65-3493). HRTEM image of second Cr_2GaC particle is shown in Fig. 2.a and confirms the layered morphology of Gr_2GaC. High resolution image in Fig. 2c shows crystallographic planes for the Cr_2GaC nanocrystals, which was deduced from the lattice fringes of a nanocrystallite shown in red circled part in Fig. 2b. We can conclude that as-obtained Gr_2GaC is a stable, homogeneous and defect-free material with layered morphology that can withstand the temperature up to at least 1300°C and can be used under high temperature conditions [4].

References:

[1] M Radovic and MW Barsoum, American Ceramic Society Bulletin **92** (2013), p. 20.
[2] H. Nowotny, Prog. Solid State Chem. **2** (1970), p. 27.
[3] MW Barsoum and TEl-Raghy, J. Am. Ceram. Soc. **79** (1996), p. 1953.
[4] The authors acknowledge funding from the from the Ministry of Education, Youth and Sport of the Czech Republic, Project Inter-Excellence/Inter-Action/LTAUSA 17128.

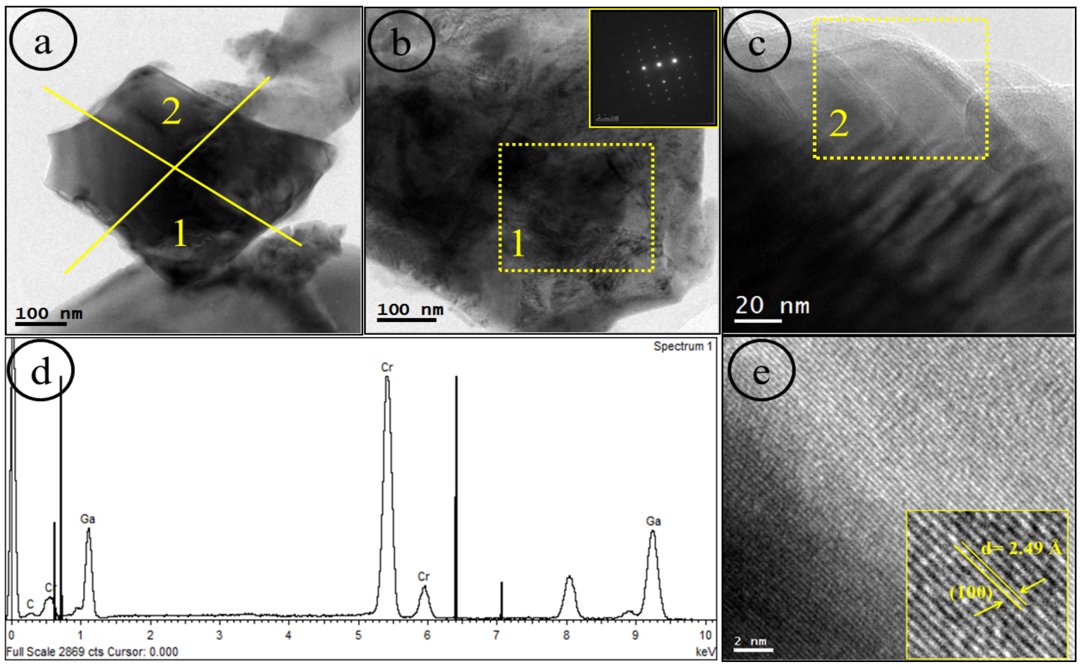

Figure 1. HRTEM determination of Cr_2GaC sample: a) Low magnification of single particle b) High magnification of part 1 with SAED as an inset confirming single Cr_2GaC nanocrystal c) High magnification of part 2 confirming stacked nanolayers with Cr_2GaC structure (PDF ICDD 65-3493).

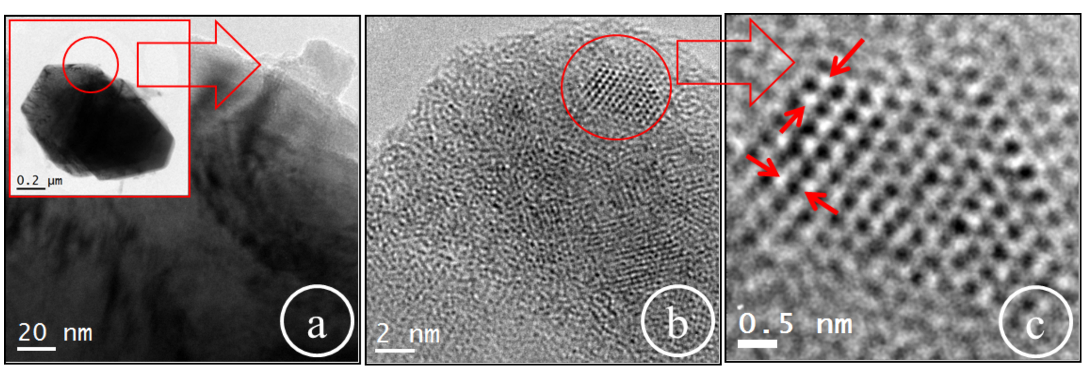

Figure 2. HRTEM image of a) an individual Cr_2GaC layer from the red circled part in the inset b) High resolution image of the Cr_2GaC layer c) HRTEM image of nanograin with Cr_2GaC structure (PDF ICDD 65-3493).

https://doi.org/10.1017/S1431927618000715 Published online by Cambridge University Press