Wet-chemical assisted synthesis of nitride and carbonitride MAX phases as potential MXene precursors

N. Kubitza[1], U. Wiedwald[2], A. Regoutz[3], C. S. Birkel[1,4]

[1] *Department of Chemistry, Technische Universität Darmstadt, 64287 Darmstadt, Germany*
[2] *Faculty of Physics and Center for Nanointegration Duisburg-Essen, University of Duisburg-Essen, 47057 Duisburg, Germany*
[3] *Department of Chemistry, University College London, 20 Gordon Street, London, WC1H 0AJ, UK*
[4] *School of Molecular Sciences, Arizona State University, 85282 Tempe, USA*

Email address of corresponding author: christina.birkel@asu.edu

The vast majority of MAX phase and MXene chemistry is represented by solely carbide-based syntheses, whereas nitride and carbonitride phases are significantly underrepresented.[1] This can be attributed to the more demanding synthesis procedure due to the high stability and low diffusion rate of nitrogen-containing compounds. However, by combining wet-chemical based methods, such as the so-called "urea-glass method", [2] as well as the rare "liquid ammonia method", [3] with conventional solid-state preparation techniques, we have developed an alternative two-step process to further expand the hardly investigated field of MAX phase nitrides and carbonitrides by V_2GaN and the hitherto unknown carbonitride phases $V_2GaC_{1-x}N_x$ [4] and $Cr_2GaC_{1-x}N_x$. First, nanoparticular precursors are prepared. Here, the sol-gel based "urea-glass" approach results in nanoparticular ternary transition metal carbonitrides (e.g. $VC_{1-x}N_x$ or $CrC_{1-x}N_x$), whereas the liquid ammonia method provides carbon-free nanoparticular binary nitride precursors (e.g. VN). Afterwards, the nanoparticular precursors are thoroughly mixed with elemental precursors to ensure MAX phase stoichiometry. Subsequent heat treatment finalizes product formation. For the new carbonitride phases, the mixed carbon/nitrogen character was proven by means of X-ray powder diffraction, Electron energy loss spectroscopy, and X-ray photoelectron microscopy. SEM micrographs reveal the morphology of the samples, consisting of partly typical anisotropic layered MAX phase structures, as well as needle- and drop-like particles covering the surface. Furthermore, magnetic measurements of the $Cr_2GaC_{1-x}N_x$ were conducted to gain new insights of the influence of the mixed C/N character. This synthesis method has the potential to open the path to further hitherto unknown nitride and carbonitride MAX phases that can also be promising as MXene precursors.

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