# Novel experimental technique of synthesis two-dimensional nanoparticles of autointercalated Niobium Diselenide

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Two-dimensional inorganic nanostructures as two-dimensional or graphene-like nanoparticles of d-transition metals dichalcogenides ("inorganic graphene-like nanostructures"; "2D nanostructures"; "ultrathin nanolayers") including 2H-$MCh_2$ nanostructures (M = Nb, Ta; Ch = S, Se; 2H-$TaS_2$ structural type; metallic type of conductivity) and their intercalated nanophases have been receiving great attention in recent years because they show unusual physical properties which are the result of a quantum size effect associated to their ultra-thin structure. These 2D nanosheets are now considered to be excellent candidates for future electronic applications. As demonstrated by groundbreaking advances such as superconductors and magnetic superlattices, 2D nanostructures play a pivotal role to realizing electronic, magnetic and optical properties.

The family of layered 2H dichalcogenides represents an interesting system in which charge-density-wave (CDW) order coexists with superconductivity. The CDW transition temperature decreases, while the superconducting critical temperature ($T_C$) increases from 2H-$TaS_2$ and 2H-$NbSe_2$ to 2H-$NbS_2$, suggesting that these two parameters compete. Indeed, as became known, in 2H-$TaS_2$ and 2H-$NbSe_2$, $T_C$ increases under pressure while $T_{CDW}$ decreases. After CDW order disappears, $T_C$ remains approximately constant. All of anomalies, including an apparent anisotropy of the superconducting gap, is very important in physical research two-dimentional nanostructured systems.

Various techniques for nanostructured 2H-$MCh_2$ synthesis have been developed in the last decade or so. However, this methods are limited to the fabrication of a small amount of single-layer nanosheets materials with low reproducibility, which is disadvantageous for their application in electronic devices.

The nanosynthesis was carried out by "up-bottom" activated processes of intercalation ($Li^+/H_2O$) of autointercalated 2H-$Nb_{1.02(1)\div1.29(1)}Se_2$ micron powders. We studied the timing data of galvanostatic processes of intercalation with the potentiostat (PI-50-1, reference electrode – AgCl). The structural properties of dispersed powders were investigated by X-ray studies, SEM.

It was synthesized the homogeneous, anisotropic graphene-like 2H-$Nb_{1.02(1)}Se_2$ nanoparticles (2D, 2H-$TaS_2$ structural type) with average sizes of 22.7(7)–46.4(1.4) nm for [013] crystallographic direction, 61.9(1.7)–144(7) nm for [110] direction. Unit cell parameters (*a*, *c*) of 2H-$Nb_{1.02(1)}Se_2$ nanostructures correlate with average sizes of nanoparticles.

As a result of scanning electron microscopy flat (2D) 2H-$Nb_{1,02(1)}Se_2$ nanoparticles have correct hexagonal form with considerable anisotropy sizes of length and thickness. It form conglomerates and does not include other types of particles.

Graphen-like intercalated 2H-$MCh_2$ nanoparticles with wide structural-sensitive physical properties set are perspective for mentioned 2D nanomaterials design. 2D crystals can also be assembled in 3D heterostructures that do not exist in nature and have tailored properties, opening an entirely new chapter in condensed matter research.