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Nanolamellar Materials

Context

Max phases: a family of nano-lamellar materials with more than 100 known members

MAX phases combine the good properties of metals with that of ceramics

They can be exfoliated to produce MXenes, a novel family of 2D materials

Yet, due to a lack of single crystal availability, most anisotropic properties are not accurately known. In 2019 we still were the only group growing MAX single crystals of macroscopic size (cm)

Objectives

- To assess the intrinsic physical properties and anisotropies of nanolamellar phases from single crystals
- To exfoliate MAX phases in order to synthesize 2D MXenes and create new 2D, tunable electron systems
- To extend research to other nanolamellar phases, such as borides, with a focus on magnetic properties and electronic structure

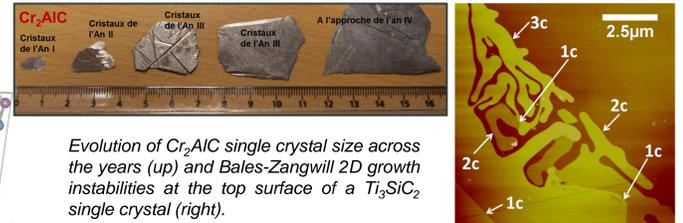
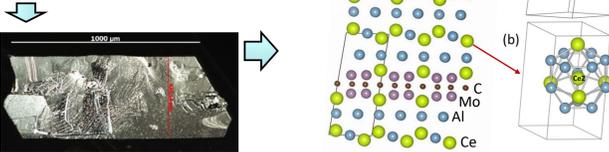
Skills & know-how

High temperature single crystal growth and characterization, DFT calculations, experiments on large scale facilities, magneto-transport...

Single Crystal Growth

- All MAX and nano-lamellar phases grown so far are non-congruent and require the use of high temperature solution growth. We grow more than 10 different nanolamellar phases.

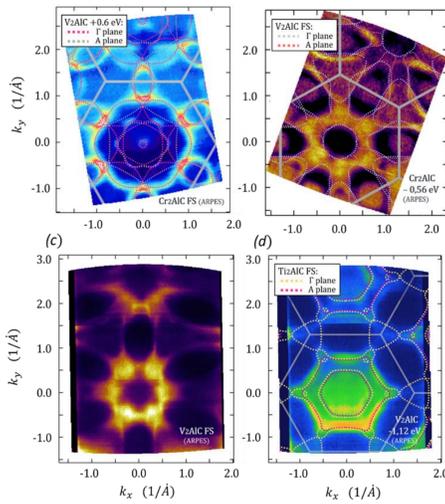
Ex.: In 2017, we successfully grew rare-earth-based magnetic i-MAX crystals.



Evolution of Cr₂AlC single crystal size across the years (up) and Bales-Zangwill 2D growth instabilities at the top surface of a Ti₃SiC₂ single crystal (right).

A. Champagne et al., *Phys. Rev. Mat.* 5, 053609 (2019)
Q. Tao et al., *Phys. Rev. Mat.* 2, 114401 (2018)

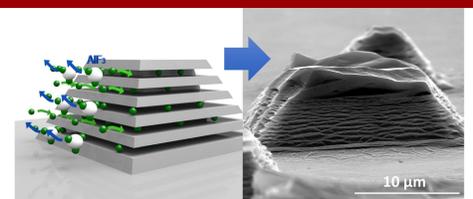
Electronic Structure of MAX Phases



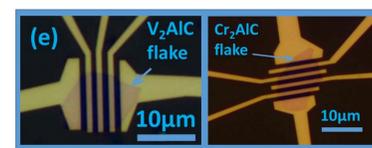
ARPES measurements and DFT computations of 211 MAX phase Fermi Surfaces. The latter can be deduced from one another by a simple shift in Fermi energy across a common rigid band model.

D. Pinek et al., *Phys. Rev. B* 100, 075144 (2019)
D. Pinek et al., *Phys. Rev. B* 98, 035120 (2018)
T. Ito et al., *Phys. Rev. B* 96, 195168 (2017)

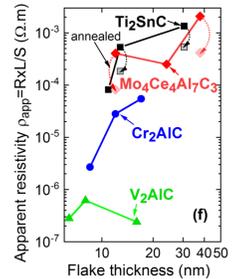
2D Materials Synthesis



Chemical exfoliation of pillars etched from MAX phase single crystals.



Devices processed from mechanically exfoliated M-Xenes and their electrical resistivity.



Advanced Characterization

Context

Team NanoMAT leading activity focus on the chemical synthesis and crystal growth of low dimensional materials. An outstanding problem is understanding how to reproducibly synthesize nano-objects with the desired structural and physical properties. Achieving this requires precise understanding of the mechanisms that take place during growth and annealing process.

Objectives

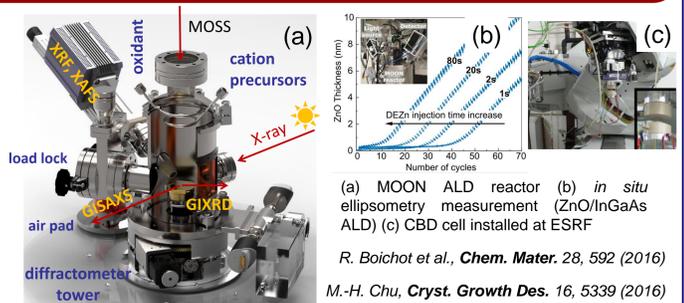
- To study/monitor the growth and annealing in controlled atmosphere, *in situ*, by multi-probe characterization methods (which includes optical and chemical probes and synchrotron radiation based techniques)
- To build-up custom equipments and develop specific characterization methods
- To tailor thin films and heterostructures
- Targeted materials: oxides, chalcogenides, lamellar di-chalcogenides

Skills & know-how

Structural and chemical characterization (XRD, XAFS, Raman scattering, TEM, ellipsometry)
Chemical Vapor Deposition (ALD, MOCVD)
Synchrotron radiation experiments

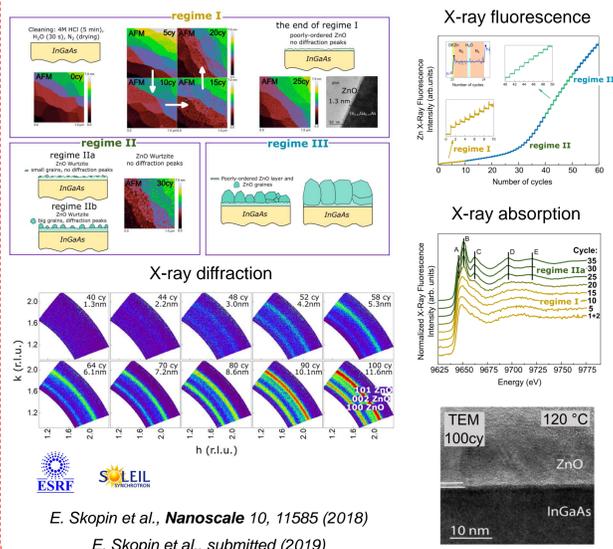
In situ Structural and Chemical Characterization During Synthesis

- We build-up instruments which can be moved to large scale facilities (i.e. synchrotron centers) and characterization platforms to perform *in situ* studies
- We implement probes for *in situ* and real time monitoring of growth and annealing in LMGP



(a) MOON ALD reactor (b) *in situ* ellipsometry measurement (ZnO/InGaAs ALD) (c) CBD cell installed at ESRF
R. Boichot et al., *Chem. Mater.* 28, 592 (2016)
M.-H. Chu, *Cryst. Growth Des.* 16, 5339 (2016)

In situ Study of the Early Stage of ZnO ALD on InGaAs



E. Skopin et al., *Nanoscale* 10, 11585 (2018)
E. Skopin et al., submitted (2019)

2D Materials Synthesis

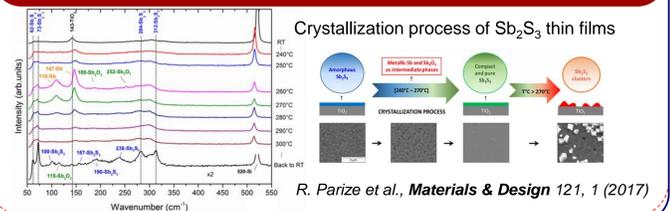
□ Lamellar chalcogenides

- Oxide thin film sulfurization
- Atomic Layer Deposition

ALD/MLD cycle

Hybrid inorganic-organic thin film deposition (ALD/MLD), en route toward TiS₂

In situ Raman Scattering Studies



R. Parize et al., *Materials & Design* 121, 1 (2017)