IUCr participation to CODATA-VAMAS Working Group on the Description of Nanomaterials

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Creating a Framework for Uniform Description System for Materials on the Nanoscale: define a complete set of information categories of descriptors usable by all communities interested in nanomaterials

- Participants: ISO (TC229), Norway Ac Sci, OECD, IUPAC, IUCr, VAMAS, CODATA, ICSU, ARKEMA, ...

- Synthesis, formulation, properties, risks, production, purchase, legislation, regulation, standards, reference materials, databases …
Crystallography and related techniques provide physically sound information sets for nanomaterial descriptions:

- Structure, Microstructure and defects (0D to 3D), Phase content, Residual Stresses, Roughness, Thickness, Magnetism, Texture, Porosity …

Crystallography already developed a large open and adaptable nomenclature: CIF

- CIF dictionaries (existing Core, Restraints, Powder, Modulated-Composite, Electron density, Twinning, Macromolecular, Images, Symmetry … and to come reflectivity, properties, MAUD … ): Provide a formal taxonomy of terms and ideas, structured, machine-readable data
- databases: PDB, COD, NDB, AMCSD, Bilbao, IZA, crystal shapes, Raman, MPOD, TCOD, PCOD …
Microstructure, 3D to 0D, iso-anisotropic: size, distributions, twin faults, antiphase boundaries, stacking faults, turbostratism, interstitials, microdistortions …

\[
\langle R_h \rangle = R_0 + R_1 P_2^0(x) + R_2 P_2^1(x) \cos \phi + R_3 P_2^1(x) \sin \phi + R_4 P_2^2(x) \cos 2\phi + R_5 P_2^2(x) \sin 2\phi + \\
\langle \varepsilon_h \rangle^4 = E_1 h^4 + E_2 k^4 + E_3 l^4 + 2E_4 h^2 k^2 + 2E_5 l^2 k^2 + 2E_6 h^2 l^2 + 4E_7 h^3 k + 4E_8 h^3 l + 4E_9 k^3 h + \\
4E_{10} k^3 \ell + 4E_{11} l^3 h + 4E_{12} l^3 k + 4E_{13} h^2 k \ell + 4E_{14} k^2 h \ell + 4E_{15} \ell^2 kh
\]
EMT nanocrystalline zeolite

Ng, Chateigner, Valtchev, Mintova: Science 335 (2012) 70
#subordinateObject_Popa rules

riet_sizestrain_sym_model 'Popa rules'

rita_harmonic_expansion_degree 4

中国队Par anisocryst size
3129.8965(67.47402)
-339.8306(43.31268)
455.05365(37.83163)
1003.05383(27.05627)

中国队Par aniso microstrain
2.627043E-4(2.0511094E-5)
-3.466392E-5(3.9479637E-5)
3.5241275E-5(5.5889054E-6)
2.1924837E-4(4.90759E-6)

#end_subordinateObject_Popa rules
Crystallography probes down to nanoscale

From crystalline to amorphous states

Single crystals, Textured, Powders

Thin architectures and bulks

Structure
Microstructure
Phase content
Residual Stresses
Roughness
Thickness
Magnetism
Texture
Porosity
Crystallographic Techniques

Diffraction (scattering + interferences): X, $\gamma$, n, e$^-$

Reflectivity (specular, off-specular): x-rays and neutrons

Small-Angles Scattering: x-rays (SAXS), neutrons (SANS)

Tomography (absorption or phase contrasts): x-rays, neutrons, electrons

Spectroscopy:
- X (XRF, XANES, EXAFS, DAFS)
- e$^-$ (EDS)
- $\mu^+$ ($\mu$SR)
CIF dictionaries provide a formal taxonomy of crystallographic terms and ideas. Dictionary entries are constructed in a structured machine-readable manner that facilitates validation and structuring of data: http://www.iucr.org/resources/cif/dictionaries

Dictionaries: Core, Restraints, Powder, Modulated-Composite, Electron density, Twinning, Macromolecular, Images, Symmetry

Local dictionaries: reflectivity (to come), MPOD (Properties), MAUD
Structure (unit-cell metric, atomic positions, thermal vibrations)

_loop_
_atom_site_aniso_label
_atom_site_aniso_B_11
_atom_site_aniso_B_22
_atom_site_aniso_B_33
_atom_site_aniso_B_12
_atom_site_aniso_B_13
_atom_site_aniso_B_23
_atom_site_aniso_type_symbol
O1 .071(1) .076(1) .0342(9) .008(1) .0051(9) -.0030(9) O
C2 .060(2) .072(2) .047(1) .002(2) .013(1) -.009(1) C
C3 .038(1) .060(2) .044(1) .007(1) .001(1) -.005(1) C
N4 .037(1) .048(1) .0325(9) .0025(9) .0011(9) -.0011(9) N
C5 .043(1) .060(1) .032(1) .001(1) -.001(1) .001(1) C
# - - - - data truncated for brevity - - - -
O21 .094(2) .109(2) .068(1) .023(2) .038(1) -.010(1) O
C51 .048(2) .059(2) .049(1) .002(1) -.000(1) .007(1) C
C511 .048(2) .071(2) .097(3) -.008(2) -.003(2) .010(2) C
C512 .078(2) .083(2) .075(2) .009(2) -.005(2) .033(2) C
C513 .074(2) .055(2) .075(2) .004(2) .001(2) -.010(2) C
Tensor properties: $c_{ij}$, $d_{ij}$, $\pi_{ijkl}$ …

_loop_

_loop_data_label
_loop_data_tensorial_index
_loop_data_value
_loop_measurement_method

$\text{prop_elastic_stiffness}_{cij} \ 'cij'$
$\text{prop_piezoelectric}_{dij} \ 'dij'$
$\text{prop_conditions}_{temperature} \ 257.0(5)$

c_11  c_12  c_13  c_14  c_15  c_16
 c_22  c_23  c_24  c_25  c_26
 c_33  c_34  c_35  c_36
 c_44  c_45  c_46
 c_55  c_56
 c_66

d_{11}  d_{12}  d_{13}  d_{14}  d_{15}  d_{16}
 d_{21}  d_{22}  d_{23}  d_{24}  d_{25}  d_{26}
 d_{31}  d_{32}  d_{33}  d_{34}  d_{35}  d_{36}
 d_{41}  d_{42}  d_{43}  d_{44}  d_{45}  d_{46}
 d_{51}  d_{52}  d_{53}  d_{54}  d_{55}  d_{56}
 d_{61}  d_{62}  d_{63}  d_{64}  d_{65}  d_{66}

d_{15}  d_{24}  d_{32}  d_{33}  d_{44}  d_{55}  d_{66}

c_{ij}$

$c_{11}$  29.1(1) BS
$c_{12}$  1.8(3) BS
$c_{13}$  1.4(3) BS
$c_{14}$
$c_{15}$
$c_{16}$
$c_{22}$  33.5(2) BS
$c_{23}$  19.8(3) BS
$c_{24}$
$c_{25}$
$c_{26}$
$c_{33}$  26.0(1) BS
$c_{34}$
$c_{35}$
$c_{36}$
$c_{44}$
$c_{45}$
$c_{46}$
$c_{55}$
$c_{56}$
$c_{66}$

d_{ij}$

$d_{15}$  3100 RUS
$d_{24}$  2435 RUS
$d_{32}$  -302 RUS
$d_{33}$  267 RUS
$d_{44}$
$d_{55}$
$d_{66}$

$d_{11}$  15 3100 RUS
$d_{22}$  24 2435 RUS
$d_{33}$  32 -302 RUS
$d_{44}$
$d_{55}$
$d_{66}$
Specular Reflectivity

\[ \alpha \]

\[ \alpha_i \]

\[ t \]

\[ 0.0 \]

\[ 0.5 \]

\[ 1.0 \]

\[ 1.5 \]

\[ 2.0 \]

\[ 2.5 \]

\[ 3.0 \]

\[ 3.5 \]

\[ 4.0 \]

\[ 1E-6 \]

\[ 1E-5 \]

\[ 1E-4 \]

\[ 1E-3 \]

\[ 0.01 \]

\[ 0.1 \]

\[ 1 \]

\[ 10 \]

\[ \alpha_c^2 \]

\[ \lambda/2h \]

\[ p^2 \]

#subordinateObject_Layer_x1

_riet_spec_layer_id 'Layer_x1'

_riet_par_spec_layer_thickness 142.3256(1.5491493)

_reflectivity_layer_critical_qc 0.03169255(5.7467838E-5)

_reflectivity_layer_absorption 5.452058E-8(1.28723885E-8)

_reflectivity_layer_roughness 0.33851644(0.08996007)

#end_subordinateObject_Layer_x1
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<th>multiplicity</th>
<th>D-spacing</th>
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<th>Microstrain</th>
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<td>55.1405712350...</td>
<td>0.007826284017...</td>
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<tr>
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<td>1</td>
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<td>81.9223706294...</td>
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<td>0.011068037</td>
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</tr>
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</tbody>
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riet_par_spec_layer_thickness 1.0E8

loop_
pd_phase_atom_%
0.9201359(0.004872608)
0.07986411(0.0069217416)

@end_subordinateObject_layer1
Free and Fee Databases and Tools: use CIF intensively!

COD (crystal structures): www.crystallography.net
PCOD (predicted crystal structures): www.crystallography.net/pcod
TCOD (theoretical crystal structures): www.crystallography.net/tcod
MPOD (properties): www.materialproperties.org
PDB (proteins): http://www.wwpdb.org
NDB (nucleic acids): http://ndbserver.rutgers.edu/
AMCSD (minerals): http://rruff.geo.arizona.edu/AMS/amcsd.php
IZA (zeolites): http://www.iza-structure.org/databases
Raman (minerals): http://minerals.gps.caltech.edu/files/raman
Bilbao Server (aperiodic): http://www.cryst.ehu.es
Crystal-Eye (merged 2014): http://wwmm.ch.cam.ac.uk/crystaleye/
PubChem
AtomWork
FPDM (Full-Pattern Search-Match): http://cod.iutcaen.unicaen.fr

CSD, ICSD, CrystMet, PDF, Pauling, Pearson
Highscore (Panalytical), Eva (Bruker), Search-Match (Crystal Impact),
PDXL (Rigaku), 3DSystems, PolyXtal (Jordan Valley Semiconductors)