

Condensed Matter Physics--X-ray Scattering

Description: The X-ray Scattering Group carries out basic studies of the structural, electronic and magnetic properties of condensed matter systems using x-ray synchrotron radiation. As members of various Participating Research Teams, the Group also develops instrumentation, maintains and operates three beamlines at the National Synchrotron Light Source (NSLS), and oversees the development of two insertion device beamlines at the Advanced Photon Source (APS). Particular emphasis is placed on investigation of surface and interfacial phenomena, including thin films, on electronic and magnetic structure and phase behavior, and on electronic excitations in solids. Recently, steps have been taken to move into materials synthesis, particularly polymer assisted synthesis and to move into soft condensed matter more generally.

Program Highlights

- Measured the dispersion of the Mott-Hubbard gap excitations in $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$ for the first time, using inelastic x-ray scattering. Observed orbital and Zhang-Rice exciton excitations in CuGeO_3 using the same technique. *v. Zimmermann et al., Phys. Rev. Lett. (submitted)*.
- Observed similar short-range correlations in $\text{La}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$ and $\text{Pr}_{0.67}\text{Ca}_{0.33}\text{MnO}_3$, two dissimilar perovskite manganites. Results suggest such correlations are robust and ubiquitous in these systems. *C.S. Nelson et al., Phys. Rev. B, 64 174405 (2001)*.
- First resonant x-ray reflectivity measurements from a liquid surface, revealing that binary alloys can break the Gibbs absorption rule. *E. DiMasi et al., Phys. Rev. Lett., 86 1538 (2001)*.
- Carried out first high-resolution in-plane diffraction from alkanes which undergo surface freezing demonstrating that in-plane domain sizes are in excess of 1 μm . *B.M. Ocko et al., Phys. Rev. E, 032602 (2001)*.
- First measurements of wetting phenomena on a nanostructured surface. *B. M. Ocko et al, (in preparation)*
- First structural measurements of electro-deposition processes on a msec time scale using high energy x-rays. *K. Tamura et al. (in preparation)*
- Discovered antiferromagnetic roughening at $\text{UO}_2(001)$ surface within 0.1 K of the bulk Neel temperature. *G. Watson et al., Phys. Rev. B, 61 8966 (2000)*.
- Discovered a simple stripe strain distribution with a 4.5 nanometer spacing for the complex reconstructions of Cu and Ag thin films grown on the $\text{Ru}(001)$ surface. *H. Zajonz et al., Phys. Rev. B., 62 10436 (2000)*

Impact: The Group's longstanding programs concerned with x-ray resonant phenomena and magnetism, with electrochemical interfaces and with liquid surfaces have been seminal in stimulating related efforts worldwide, and remain among the leading programs in these areas today. Recent inelastic x-ray scattering work has, with others, led to the creation of a Collaboration Access Team dedicated to inelastic x-ray scattering at the Advanced Photon Source (IXS-CAT).

Interactions: In a typical year, the Group collaborates with 40-50 PIs, together with an approximately equal number of students and post docs. A list of collaborators is given below. It includes significant internal BNL collaboration, both within Condensed Matter Physics and more widely (in particular, the NSLS, Chemistry and Materials Science Departments) together with external collaborations with universities, other national laboratories and foreign institutions.

Personnel: John Hill (Group Leader), Doon Gibbs (50%), Ben Ocko, Elaine DiMasi, Tianbo Liu (80%), Christie Nelson (30%, Research Associate), Young-June Kim (30%, Research Associate), Kazu Tamura (10% Research Associate)

Recognition: Group members have given 55 invited talks in the past 3 years at national and international meetings (including 8 plenary talks), and at universities. Awards: 2 Fellow of the American Physical Society, 1 Fellow of the American Association for the Advancement of Science, 1 Presidential Early Career/DOE Young Investigator Award, 1 Wohlfarth Award, 1 Goldhaber Distinguished Postdoctoral Award, 1 BNL Engineering Award.

Budget: 1030 K.

Current Staff and Facilities

John Hill, Group Leader	Strongly correlated electron systems, inelastic x-ray scattering, resonant x-ray scattering.
Doon Gibbs	Magnetism, X-ray resonance techniques, surfaces/interfaces.
Ben Ocko	Organic thin films, liquid interfaces, electrochemical interfaces.
Elaine DiMasi	X-ray scattering studies of biomineralization processes – the interaction of organic molecules with nucleating minerals.
Tianbo Liu	Use of chemically active polymers in nanofabrication of functional inorganic nanomaterials.
Christie Nelson	Orbital and charge ordering in transition metal oxides, including thin films.
Young-June Kim	Electronic excitations in 2D cuprates, x-ray and neutron scattering studies of transition metal oxides.
Kazu Tamura	<i>in-situ</i> electrochemical studies of electrolyte-metal interfaces.

2 Technical staff and (1/2) secretary.

Facilities

- NSLS Beamlines, X22A,B,C; Ben Ocko beamline spokesperson.
- (1/8) partner in Complex Materials Consortium CAT at the APS with Exxon, Princeton, Penn U. Tennessee, ORNL, LANL and UCSB. Doon Gibbs, CAT Director.
- Member Inelastic X-ray Scattering CAT at the APS, recently funded to build a dedicated inelastic x-ray scattering beamline. John Hill, CAT Director.
- Rotating Anode Facility.

Other Funding

LDRD Experimental and Theoretical Studies of Transition Metal Oxides, 1999-2001 \$60k, p.a. (Hill)

LDRD Biomineralization: a route to advanced materials, 2001-2002 \$100k p.a. (DiMasi)

LDRD Proteins on liquid surfaces 2000-2002 \$50 k p.a. (Ocko)

2% Initiative with SNL-Livermore and ORNL, \$80k p.a (Gibbs)

Charge Inhomogeneity in Correlated Electron Systems (2 post-docs)

Nanoscale Functional Materials (1/3 Tianbo Liu as of 10.1.01)

Charge Injection and transport in nanoscale materials (1/5 Tianbo Liu as of 10.1.01)

JSPS Fellowship (Kazu Tamura)

Artificial molecular films with evolving structural complexity:US-Israel Binational Sci. Foundation (Ocko)

Future Directions

- The x-ray group, as part of a BNL-wide effort is expanding its efforts in the area of soft condensed matter. In this regard, Ben Ocko has recently begun studies of wetting phenomena on nanostructured interfaces, building on his continuing work on thin organic films. Our new hire, Tianbo Liu, has initiated a program in polymer assisted synthesis. His future interests include self-assembly in polymer solutions with particular reference to biomaterials.
- Will play a prominent role in the proposed BNL Nanocenter, with significant effort in three of the initial thrust areas, nanomagnetism, strongly correlated nanoscale systems and thin organic films.
- A program has recently been started to study the interaction between organic molecules and nucleating minerals – biomineralization. If successful, it is hoped to grow this into a larger program studying biomimetics in general, in particular of nanoscale functional materials.

Recent Collaborators and Institutions (PIs only)

External Collaborators

Universities

R. J. Birgeneau University of Toronto
K. Blasie University of Pennsylvania
C. Burns W. Michigan University
P. Canfield Iowa State
B. Chu SUNY-SB
S.-W. Cheong Rutgers University
G. Cao Florida State University
A. Goldman Iowa State
L. B. Gower University of Florida
P. Heiney University of Pennsylvania
V. Kiryukhin Rutgers University
S. McLaughlin SUNY-SB
S. Moss University of Houston
P. Pershan Harvard University
M. Rajeswari Towson State
I. K. Robinson University of Illinois
D. Schwartz Tulane
P. Stephens SUNY-SB
H. Tostmann University of Florida
D. Vaknin Iowa State
C. Venkatesan University of Maryland
G. Watson University of Maryland, B.C.
B. Wells University of Connecticut

National Labs

A. Baddorf ORNL
D. Zehner ORNL
N. Bartelt Sandia
J. Brinker Sandia
M. Kent Sandia
R. H. Wang Sandia
P. Canfield Ames
A. Goldman Ames
G. Cao NHFML
D. Casa ANL
T. Gog ANL
G. Srajer ANL
C. Venkataraman ANL
S. Lee NIST
S. Satija NIST
J. Majewski LANL
C. Orme LLNL

Industry

C. Black IBM
S.-W. Cheong Lucent
R. Kolb ExxonMobil
E. Sirota ExxonMobil
P. Stevens ExxonMobil

Foreign

J. Aarts Free U. Amsterdam, NETHERLANDS
N. Andersen Ris ?, DENMARK
A. Boothroyd U. Oxford, ENGLAND
R. Cowley U. Oxford, ENGLAND
M. Deutsch Bar-Illan University, ISRAEL
J. Fossum Norwegian U. of Science & Technology., NORWAY
A. Frenkel Yashiva University
A. Gibaud Univ of Maine, FRANCE
C. Giles State U. Campinas, BRAZIL
J. Goff U. Liverpool, ENGLAND
S. Ishihara U. Tokyo, JAPAN
N. Jiswari Birzeit University, Palestine
B. Keimer MPI-Stuttgart, GERMANY
G. Lander EITU, GERMANY
P. LoNosto Firenze U.
O. Magnussen U. Ulm, GERMANY
D. McMorrow Ris o, DENMARK
M. Mueller U. Ulm, GERMANY
A. Mueller University of Bielefeld
Y. Murakami KEK/Photon Factory, JAPAN
J. Sagiv Weizmann Inst. Sci., ISRAEL
J. Schneider HASYLAB, GERMANY
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C. Vettier ESRF, FRANCE
M. v.Zimmermann HASYLAB, GERMANY
F. Yakhou ESRF, FRANCE
H. Zajonz MPI-Stuttgart, GERMANY

Internal BNL Collaborators

L. Berman (NSLS)
G. Gu (Physics)
J. Hrbek (Chemistry)

C.-C. Kao (NSLS)
S. Shapiro (Physics)
G. Shirane, B. Noheda (Physics)
J. Tranquada (Physics)
T. Vogt (Physics)
J. Wang, R. Adzic (Materials Science)
Y. Zhu (Materials science), C. Creutz, (Chemistry)

Liquid metal and electrochemical interfaces
Single crystal growth
Sulfur induced nanophase structures in Cu/Ru bilayers
Electronic excitations in strongly correlated systems
Perovskite Titanates
in-situ studies of Ferroelectric Materials
Charge ordering in transition metal oxides
Powder diffraction studies of various compounds
in-situ studies of electrochemical interfaces
Charge transfer and TEM studies of polyoxomolybdate clusters

Recent Research Highlights

Excitons in charge transfer insulators

[Hill, Kim, Kao, v. Zimmermann (BNL), Birgeneau (Univ. of Toronto), Wakimoto (MIT), and Uchinohura (Univ. of Tokyo)].

Understanding the electronic excitation spectrum is crucial for a complete understanding of strongly correlated electron systems. We have carried out detailed studies of the energy and momentum dependence of the excitation spectrum in the 1D and 2D cuprates, CuGeO_3 and $\text{La}_{2-x}\text{Sr}_x\text{CuO}_4$, respectively. This work, utilizing inelastic x-ray scattering, was performed at CMC-CAT at the Advanced Photon Source, following earlier work at X21 at the NSLS. In CuGeO_3 , three excitations were observed (figure 1). The feature at 1.7 eV is ascribed to an orbital $d-d$ electron, that at 3.8 eV to a Zhang-Rice-like exciton (defining the optical gap) and that at 6.4 eV to a charge transfer excitation. The lower two are dispersion-less while the 6.4 eV feature exhibits measurable dispersion. In the 2D cuprate, the gap excitation consists of two modes which exhibit considerable dispersion. These data are unique in probing the dispersion of these excitations and challenge current theoretical understanding of the materials.

Refs: v. Zimmermann, et al. (PRL submitted), Kim, et al. (in preparation)

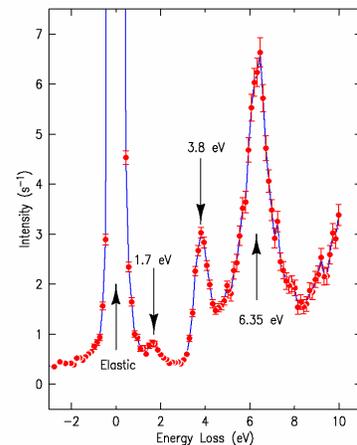


Fig. 1. Excitation spectrum of CuGeO_3 as measured using resonant inelastic x-ray scattering.

Breaking the Gibbs Adsorption Rule: Resonant X-ray Reflectivity from a Liquid Alloy

[DiMasi, Ocko, Berman (BNL), Tostmann, Huber, Shpyrko, Pershan (Harvard), and Deutsch (Bar-Ilan University, Israel)]

The surface of binary liquids are supposed to follow a simple rule, formulated by Gibbs in 1878: the species having the lowest surface tension should segregate into a separate monolayer at the surface. However, this picture assumes that interactions between atoms can be disregarded. What happens in alloy-forming mixtures with attractive interactions between atoms?

To answer this question, we performed resonant x-ray reflectivity measurements from a liquid Bismuth-Indium mixture having 22 at% Bi in the bulk. By tuning the incident x-ray energy through the Bi LIII absorption edge, we can determine the ratio of Bi to In at the surface. The Gibbs rule predicts segregation of 70% Bismuth at the surface. However, we find a Bi enrichment of 35 at% in the surface layer, compared to the 22 at% in the bulk. This is considerably less Bi than would be expected in the absence of attractive Bi-In interactions, which we find must be on the order of 10 $k_B T$ to explain our data. These are the only resonant x-ray reflectivity measurements ever performed on a liquid surface, and have allowed us to quantify, for the first time, the extent to which attractive interactions can compete with Gibbs adsorption.

Ref: E. DiMasi et al, Phys. Rev. Lett. 86 (2001) 1538.

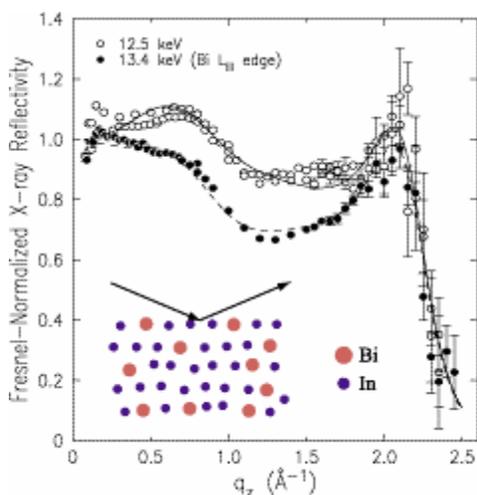


Fig. 2. X-ray Reflectivity from In-Bi alloy. Data taken at the Bi LIII edge (closed circles) and away from the edge (open circles).

Growth and Hydration Effects in Multilayer Silane Films

[Ocko, DiMasi (BNL), Sagiv (Weitzman Inst.)]

Silane derivitized, thin organic films hold great promise in electronics, non-linear optics, tribology, and in the development of advanced methods of nano-fabrication technologies. Remarkably, a process has been discovered whereby silane films grow in a geometric manner, doubling in thickness during each chemical cycle. This is in contrast to ordinary multilayer films that grow in a layer-by-layer process. The cyclic process by which these films grow involves a hydration step followed by dipping into a solution containing octadecyl-trichlorosilane (OTS; $\text{SiCl}_3\text{C}_{18}\text{H}_{37}$). Our x-ray reflectivity and grazing incident angle diffraction studies of the structure of these films indicate that these films do not order in the same simple bilayer configuration (see figure) as does the bulk. Further, the expansion of the layers, which accompanies hydration, is consistent with one water molecule per silane molecule. This preadsorbed water is precisely what is required to hydrolyze one additional silane molecule, i.e., the amount needed to double the film thickness during each cycle.

Refs. R. Maoz, J. Sagiv, S. Matlis, B.M. Ocko and E. DiMasi, et al., Nature 384, 150 (1996) and B. M. Ocko and et. al., Unpublished Results.

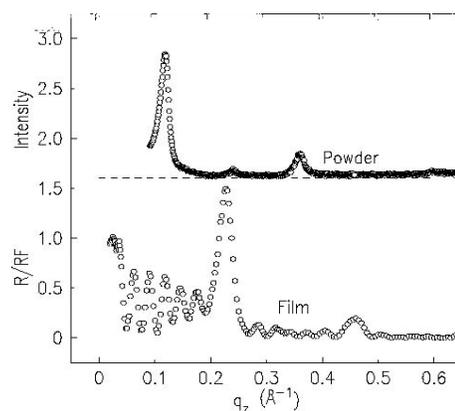


Fig. 3. Comparison of the x-ray scattering from a bulk OTS powder sample and an 8 layer OTS film

Positional order and thermal expansion of surface crystalline *N*-alkane monolayers

[Ocko, DiMasi (BNL), Sirota (ExxonMobil), Deutsch (Bar Ilan), Gog, and Venkataraman (CMC-CAT)]

Classical statistical mechanics predicts the phenomena of surface melting. However, the opposite effect, surface freezing, was believed to be impossible prior to our findings which showed that an ordered monolayer of *n*-alkanes (and derivatives) forms at the surface of the liquid above its bulk freezing temperature. The structure of this monolayer has been determined previously with x-ray reflectivity and grazing incidence diffraction. However, it was not possible to measure the scattering lineshape or the lattice constants. At the Advanced Photon Source, we have carried out studies of *n*-eicosane, ($C_{20}H_{42}$), just above its melting temperature with a factor of twenty improvement in the resolution. The monolayer diffraction peak was found to be resolution limited, implying positional correlations in excess of $\sim 1 \mu\text{m}$. The 2D thermal expansion was $(dA/dT)/A = 1.8(\pm 0.1) \times 10^{-3} \text{ } ^\circ\text{C}^{-1}$, which is comparable to the bulk value. Our data are consistent with the power-law lineshape expected from quasi-long range order in 2D.

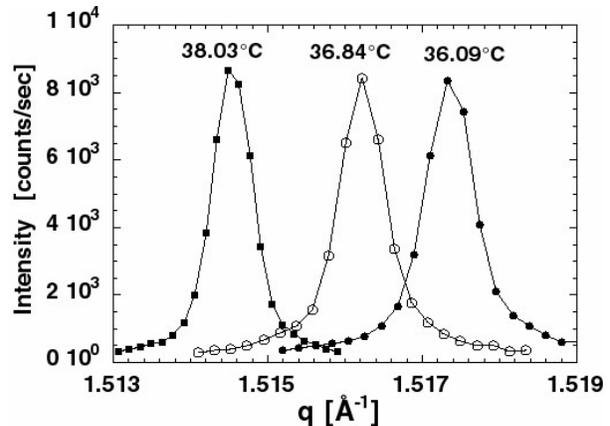


Fig. 4. High-Resolution in-plane diffraction from a frozen monolayer of *n*-eicosane.

Ref. B. M. Ocko *et al.*, Phys. Rev. E **63**, 032602 (2001)

Antiferromagnetic Roughening

[Gibbs (BNL), Lander (EITE), Langridge (ISIS), Watson (U. Maryland, Baltimore County) Vettier (ESRF).]

In the last several years, there have been continuing efforts to probe long range magnetic order at surfaces. Basic questions include: Does the magnetization profile at the surface follow that of the electronic charge density? Does the magnetic critical behavior near a surface differ from that of the bulk? What is the relationship between the chemical and magnetic interfacial roughness? In these studies we have used x-ray resonant scattering techniques to measure the magnetic reflectivity of a UO_2 (001) surface versus temperature. Previously, we have shown that in contrast to the bulk, the surface layers order continuously. We suggested a model in which a magnetically disordered region exists at the surface, separated from the ordered bulk by a diffuse magnetic interface. Here, we suggest that this magnetic interface roughens as the bulk magnetic order transition is approached from below. To our knowledge, this is the first measurement of magnetic roughening of an antiferromagnet. As the bulk Neel temperature is approached, the lineshape of the transverse profile takes on a power-law form, consistent with antiferromagnetic roughening, figure 5. These results are qualitatively consistent with the theory of surface induced disorder. A detailed analysis of the lineshapes is currently underway.

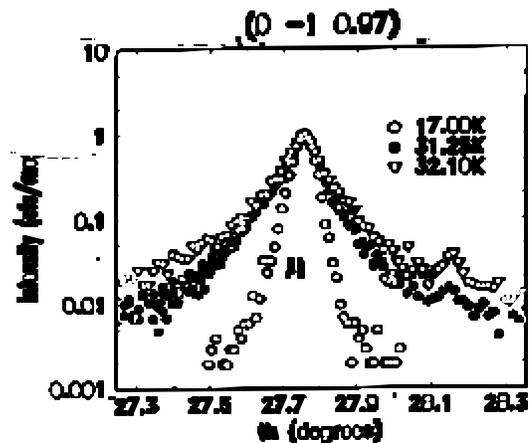


Fig. 5. Transverse scans through magnetic truncation rod as transition is approached. Diffuse foils indicate a roughening of the magnetic interface.

Refs: G. Watson *et al.*, Phys Rev B **61**, 8966 (2000), S. Langridge *et al.*, unpublished.

Nanoscale Stripe Patterns in Strain Relief of Heteroepitaxial Growth

[Zajonz , Gibbs (BNL),Baddorf and Zehner (ORNL)]

We have studied the *in-situ* growth and phase behavior of thin Cu and Ag alloy films deposited on Ru(001) surfaces. These interfaces are related to important bimetallic catalysts, yet surprising gaps persist in our knowledge of their structure. Relative to the Ru(001) surface, the bulk spacing of Cu is compressed by ~ 5.8%, while that of Ag is expanded by ~ 6.3%. This leads to complex stripe reconstructions driven by the relief of strain arising from the substrate-film lattice mismatch. Utilizing simulated annealing techniques for what we believe is the first time in surface crystallography, we have found a diverse range of structures of Cu and Ag films grown on Ru(001). For Cu, these include a fully strained, pseudomorphic monolayer. For Ag, a yet more complicated-looking reconstruction was found, consisting of a distorted, hexagonal dislocation network. However, we discovered a surprising connection between the two structures when the in-plane strain distribution is calculated relative to the bulk. In each case, a simple stripe structure with a ~ 4.5 nm spacing emerges (fig.6). This fascinating result reflects the fact that the hcp and fcc patches for Ag/Ru tend to align within the unit cell, whereas for Cu/Ru the domains themselves comprise the stripes. The remarkable similarity points to the importance of long wavelength elastic interactions of the film with the substrate, though a detailed understanding is still lacking.

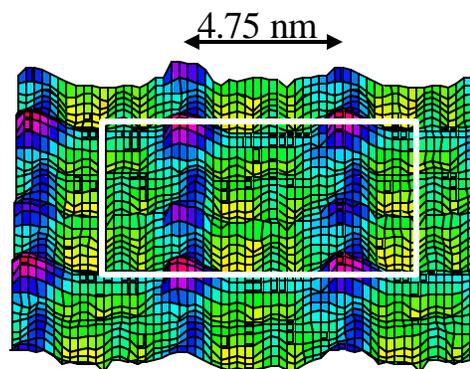


Fig. 6. In-plane strain distribution of Ag monolayer on Ru. Stripes of highly strained regions are 4.75 nm apart.

Refs: H. Zajonz et al., Surf. Sci. Lett. **447**, L141-146 (2000), H. Zajonz et al., Phys. Rev. B **62**(15), 10436-10444 (2000). A.P. Baddorf, et al., Surf. Sci. (in press), A.P. Baddorf et al., Phys. Rev. B (submitted), H. Zajonz, et al., Phys. Rev. B (submitted).

Synthesis and characterization of giant polyoxomolybdate “nanomolecules”: The largest inorganic molecules observed to date.

[Liu (BNL), Chu (SUNY-SB)]

In the past year, we have been actively characterizing the recently discovered giant polyoxomolybdate (POM) clusters, which are synthesized via a unique polymer-assisted process. They are regarded as the largest inorganic molecules discovered to date and are analogous to the putative C_{240} molecules, the so-called “second generation” C_{60} molecule. We have found that these 5.1 nm hollow spherical POM clusters pack into a zeolite-like cubic structure. SAXS and TEM (figure 7) work demonstrated that this packing is highly ordered. Raman scattering, EXAFS and electrochemistry work demonstrated that there are 12 pentagons on the nanosphere with 60 Mo(V) and 432 Mo(VI) ions. Further, surfactants have been used to create monodispersed solutions of single nanospheres. Future work will characterize the novel catalytic, electronic and magnetic properties of these unique molecules, together with attempts to synthesize similar molecules with other metals, e.g., tungsten.

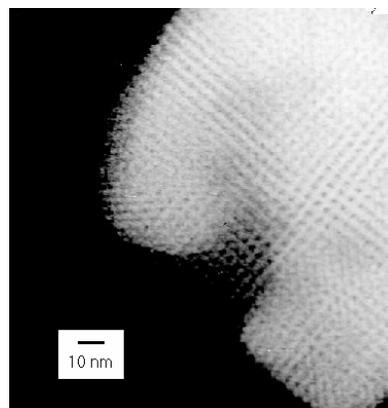


Fig. 7. TEM of clusters of polyoxomolybdate nanomolecules. The regular packing of these structures is clearly evident.

Refs: Liu, T.; Wan, Q.; Xie, Y.; Burger, C.; Liu, L.-Z.; Chu, B. J. Am. Chem. Soc., 2001, web-released on Oct., 11th, (in press), Liu, T.; Xie, Y.; Chu, B. Langmuir, 2000, 16, 9015.

Publications

2001

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Baptiste, A., Gibaud, A., Bardeau, J.F., Wen, K., Maoz, R., Sagiv, J., and Ocko, B.M. X-ray micro-Raman and infrared structural characterization of self-assembled multilayer silane films and variable numbers of stacked layers. *Langmuir* (submitted).

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