Why do we need Sirius? (Scientific examples)

Tuesday 23 Nov 2010 at 11:00 (01h00')

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1. Introduction



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2. Exploitation of the phase contrast: 3D Imaging at µm level



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2 Generation

















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2 Generation





Insertion devices

← NOTICE THE VERY SMALL DIVERGENCE OF THE BEAM



3 th Generation

Insertion devices



COHERENCE

- Light is coherent if it has:
 - Spatial (transverse)coherence→ divergence
 - Temporal (longitudinal) coherence→ monochromaticity

• The light emitted by an undulator has spatial coherence, temporal

coherence can be added by a monochromator

- Coherence length:
 - longitudinal: $L^{long} = \frac{1}{2} \frac{\lambda^2}{\Delta \lambda}$
 - transverse: $L^{tra} = \lambda R/(2\pi\sigma)$

 $(\lambda = wavelength, \sigma = beam size and R = distance source-sample)$

Ex: $L^{tra} = 25/100 \,\mu m$ in the vertical plane, 3/10 in the horizontal PUERTO VALLARTA 4

- Scientific research and technological development with synchrotron radiation have experienced an enormous growth all around the world over the past 20 years. In fact, there are today more than 50 operating synchrotron light sources all around the globe. Sixteen of them are 3th generation sources, characterized by low emmittance (small size and very small divergence of the beam) and the intensive use of magnetic insertion devices called undulators: they have been put in operation after 1994 and have allowed to gain 4 orders of magnitude in brightness. Some of the sources have come into operation quite recently, such as the British (Diamond), the French (SOLEIL), the Australian (BOOMERANG), the Canadian Light Source, the Chinese SSRF, the German (PETRA III) facilities and the Spanish one, ALBA, will come in operation next year.
- All of those laboratories represent the response of these various countries to the explosive growth in demand for synchrotron radiation, as a result of the wide range of scientific and technological applications which were made possible by the availability of these new sources. It is interesting to notice that the number of users of the DOE facilities in the USA (APS, ALS, NSLS, SSRL) has increased by 40% (6000→ 8400) between 2000 and 2008 while the number of users of the Éuropean Facility (ESRF) has increased by 46% during the period 2002/2009.

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- the exploitation of specific properties of the X-Ray beams of these new facilities (phase contrast and coherence) allowing to obtain three-dimensional images of any object with submicron resolution. This has brought to synchrotrons new communities: paleontology, cultural heritage, geophysics 5 environment... 5

2- EXPLOITATION OF THE PHASE CONTRAST IN X-RAY IMAGING

The behaviour of x-rays as they travel through an object can be described in terms of a complex index of refraction. In the x-ray region, it can be written as:

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• The real and imaginary parts have very different dependences on the photon energy; in the regime where the photoelectric effect dominates and far from absorption edges, $\beta \sim E^{-4}$ while $\delta \sim E^{-2}$. As a consequence, the values of δ can be orders of magnitude larger than β terms; for example, the values for nylon (C₂H₄) at 25 keV are $\delta = 3.50 \times 10^{-7}$ and $\beta = 8.12 \times 10^{-11}$.

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• X-rays passing through regions of different δ values are subjected to phase shifts that correspond to being refracted. These changes, which can originate from the purely geometrical effect of the shape of the object or, for instance, from local homogeneity defects of the object, cannot often be visualized using absorption imaging techniques.

Different techniques have been developed for detecting the phase variations:

- in-line holography (holotomography)
- interferometry
- diffractometry

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Principle of holotomography.



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8

Archaeopteryx: bird or dinosaur?

- Discovered in Germany in 1861.
- Missing link between birds and dinosaurs.
- Crow-sized, lived about 150 M years ago.
- Feathers identical to that of modern bird.
- Also had sharp teeth, a long bony tail, clawed fingers, and long feathers on its back and legs.
- This suggests that the first birds were four-winged gliders that coasted between treetops like flying squirrels.
 P.TAFFOREAU and E. BUFFETAUT





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The impossible fish brain revealed by synchrotron

holotomography A. Pradel (a,b), M. Langer (c), J.G. Maisey (d), D. Geffard-Kuriyama (a), P. Cloetens (c), P. Janvier (a) and P. Tafforeau (c). PNAS, 106, 5225-5228 (2009).
Animal fossils are generally remains of mineralised hard tissues (i.e. understanding of the evolution of life on our planet.





Fig. 125: 3D Rendering and virtual histological slice of a 145 million years old Cretaceous mammalian tooth from Cherves-de-Cognac (Charente, France). This minute fossil tooth was imaged using submicrometre resolution holotomography on ID19. The highly monochromatic source, in addition to short acquisition time, leads to unprecedented image quality for this kind of microfossil.

The detailed 3D morphology gives access to the internal histological information of dental microstructures including its developmental record. Study of the growth lines in such fossils should bring new evidence of the controversial physiological thermal regulation in our mammalian ancestors as well as in dinosaurs (Courtesy: J. Pouech, J.-M. Mazin and P.Tafforeau, unpublished).



P. Verboven et al., Plant Physiology 147, 518, 2008









ICE CREAM, CHOCOLATE....

PHASE CONTRAST WITHOUT A 3rd GENERATION SOURCE? a)Schematic of a Bonse–Hart interferometer b) a shearing interferometer.

The first grating splits the incoming wave into the +1st and -1st diffraction orders, which are then recombined by a second grating with half the pitch of the first grating. C. David et al., APL 81, 3287, 2002



X-Ray Talbot Interferometer

- Principle: the beam splitter grating (G1) splits the incident beam into essentially two diffraction orders, which form a periodic interference pattern in the plane of the analyzer grating. A phase object in the incident beam will cause slight refraction, which results in changes of the locally transmitted intensity through the analyzer.
- (Weitkamp ., Optics Express 13 (2005) 6296-6304.).



Example: mouse in formalin

Transmission tomogram





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tomography of a mouse in formalin – ESRF, ID19 9–14 April 2009 – 35.0 keV 30 um pixel size

I. Zanette – X-ray imaging with a grating interferometer

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I. Zanette – X-ray imaging with a grating interferometer

Grating-based X-ray tomography offers superior three dimensional images of the human cerebellum, which not only allow for the discrimination between grey and white matter but also between stratum moleculare and stratum granulosum. Additionally, the clear visualization of non-stained individual Purkinje cells is possible—a technique that is unrivalled so far.



-the stratum granulosum (blue),
- the stratum moleculare (green-yellow-red gradient)
-the white matter (mainly red).
G. SHULTZ et al., J. R. Soc.
Interface publied on line 21/07/2010

W.Ludwig, S. Schmidt, E. Mejdal Lauridsen and H.F. Poulsen-Appl. Crystallography 41,302,2008



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- The procedure is termed X-Ray diffraction contrast tomography (DCT), reflecting its similarities to conventional absorption contrast tomography. During acquisition of an optimized tomographic scan, undeformed grains embedded in the bulk of a polycrystalline sample give rise to distinct diffraction contrasts which can be observed in the transmitted beam each time a grain fulfils the Bragg diffraction condition.



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- By extracting and sorting these contrasts into groups belonging to individual grains, one is able to reconstruct the 3D grain shapes by means of parallel beam, algebraic construction techniques.



TRACKING CORROSION CRACKING

 \rightarrow During fabrication and operation, many stainless steel components are exposed to mechanical loads that create high strains inside the material, which results in mechanical failures at unexpectedly low loads.

→ Recently, King, Johnson, Engelberg, Ludwig &Marrow(SCIENCE 382, 321,2008) have shed light on the microscopic origin of environmental corrosion by studying crack formation in situ, such as an acidified solution of $K_2S_4O_6$ on the polycristalline grain structure of samples in an electrochemical cell.

 \rightarrow They combine grain reconstruction with an in situ localization of corrosion processes inside the sample.

 \rightarrow They use for that DCT(Diffraction Contrast Tomography) and CT (Computed Tomography)

 \rightarrow In these studies, the spatial resolution is 1µm. Next step: 50 nm

Fig. 1. Part of the 3D grain map obtained by DCT, including 169 grains (a total of 362 grains were mapped). The circumference of the sample is outlined, and the plane of the 2D section in Fig. 3 is also shown.

(A) Grains colored using a RBG scale, according to their crystallographic orientation.

(B) Low CSL grain boundaries are shown in color: low-angle Σ 1(orange), twins Σ 3 (red), Σ 9 (blue), other boundaries Σ < 29 (purple)



- → Combined use of Diffraction Contrast Tomography (DCT) and Computed Tomography (CT) data to identify crack bridging grain boundary structure
- (A) Cracks obtained from CT data are shown in black, at the final step before sample failure, and compared with DCT data of 3D grain shapes.
- (B) 2D section of the grain boundaries, identified by DCT, compared with the crack path identified by CT. The boundaries are colored as in Fig.1, and a crack bridge is shown.



Morphological clues to wet granular pile stability

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- When a granular material such as sand is mixed with a certain amount of liquid, the surface tension of the latter bestows considerable stiffness to the material, which enables sand castles to be sculpted
- The geometry of the liquid interface within the granular pile is of extraordinary complexity and strongly varies with the liquid content.
- Surprisingly, the mechanical properties of the pile are largely independent of the amount of liquid over a wide range.
- This puzzle has been resolved with the help of X-Ray microtomography, showing that the remarquable insensitivity of the mechanical properties to the liquid content is due to the particular organization of the liquid in the pile into open structures

Liquid bridge form at the contact between grains, as a result of surface tension a,Fluorescence microscopy image of liquid bridges between 375– µm–diameter glass beads. **b**, Schematic of a liquid bridge (blue) between spherical surfaces (yellow). θ is the liquid–solid contact angle, ϕ is the half–filling angle, defined as $\phi = \tan -1(r/R)$, where r is the radius of the liquid bridge, and R is the radius of the grain.

The curvature of the liquid interface leads to low pressure in the liquid causing a force of attraction between grains



~ ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	Dry grains — cohesion negligible
	Partially saturated — at small volume fractions, liquid bridges are formed between grains near points of contact. Liquid bridges induce cohesion between grains.
Trimer Tetrahedron Pentamer	At higher volume fractions, liquid bridges merge to give trimers, tetrahedra and pentamers.
	At still higher volume fractions, large contiguous wet clusters form.
	Slurry — the pore space is fully saturated with liquid. Cohesion becomes negligible again.

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(from Arshad Kudrolli Nature Materials 7, 175, 2008)

a, Top row: Capillary bridge (cb), trimer (tr), pentamer (pt) and filled tetrahedra (th) as obtained from X-ray tomography. Bottom row: As obtained numerically. . **b**, Fraction of a large percolating liquid cluster (X-ray tomography)



c, Cumulative plot of the total liquid surface area versus the volume of all clusters appearing at W (liquid content) =0.035, as obtained by X-ray microtomography (hp: heptamers). PUERTO VALLARTA

d, Distribution of angular distances between two neighbouring contact points (threshold separation 0.05 R). Inset: Schematic diagram of a capillary bridge at bead separation₂₅s, and two trimers.

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 In CDXI, the object is illuminated with coherent X-rays and its far-field diffraction pattern is recorded without any optic. From this diffraction pattern, the wave field behind the object is reconstructed by iteratively solving the phase problem. 3D imaging is possible by recording a (tomographic) series of diffraction patterns. Coherent illumination of the object is crucial to this this technique, and the coherent dose on the sample determines the spatial resolution. As the coherent flux at modern SR sources is limited, CDXI experiments require nanofocusing: a resolution of <u>5 nm</u> has been achieved with 100 nm focusing.
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• Coherent diffraction imaging emerged from the realization by Sayre (1952) that oversampled diffraction patterns can be inverted to obtain real-spaces images.

3. EXPLOITATION OF THE COHERENCE

There are different ways to do microscopy in the hard X-Ray:

 \rightarrow By using lenses (Fresnel, refractive..), mirrors or capillaries. For the moment the resolution is limited to <u>100-50 nm</u>.

 \rightarrow By coherent X-Ray diffraction imaging (CDXI).

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It was demonstrated by Miao, Charalambous, Kirz and Sayre in 1999 (Nature 400, 342).

Coherent X-Ray Diffraction Imaging: 3D mapping of a deformation field inside a nanocrystal. Pfeifer, Williams, Vartanyants, Harder&Robinson. Nature 442, 63, 2006



- Pb is evaporated (20 nm)/Si.

Aftermelting \rightarrow moltendroplets \rightarrow isolated hemispherical crystals. A CCD X-Ray detector is centred on the (111) Bragg peak of one of the crystals, to give the diffraction peak shown below ($\lambda = 1.38$ Å, APS)



\rightarrow The diameter of the nanocrystal is 750 nm and the resolution $\underline{40~nm}$

→ Diffraction also opens the new possibility of directly imaging the strain field within the crystal because it breaks the local symmetry of a diffraction pattern around a Bragg peak

→ The strain (yellow) is superposed on a translucent image of the nanocrystal itself (grey)



MICROSCOPY WITHOUT LENSES OR MIRRORS: Takahashi, Zettsu, Nishino, Tsutsumi, Matsubara, Ishikawa&Yamauchi NanoLetters 10, 1922, 2010

Synchrotron X-rays are focused on a 1-µmdiameter spot through KB mirrors, and a sample (Au/Ag nanobox) is placed on the spot. The intensity distribution of X-rays elastically scattered in the forward direction (coherent X-ray diffraction patterns) is measured by a charge-coupled device (CCD) X-ray detector. The obtained coherent X-ray diffraction patterns are processed by a computer to reconstruct a three-dimensional image of the sample structure. In the reconstruction, coherent X-ray diffraction patterns from different incident angles, obtained by rotating the sample, are used.



- (b) Reconstructed projection image of coherent X-ray diffraction data.
- (c) SEM image of same nanobox.
- (d) TEM image of different nanoboxes.



 A three-dimensional electron density distribution is obtained by performing a phase retrieval calculation with respect to the coherent X-ray diffraction patterns of a Au/ Ag nanobox observed with different X-ray incident angles. The detailed surface structure of the Au/Ag nanobox can be observed when an equivalent-electrondensity plane is displayed. Small pits and a large pit are confirmed at the positions indicated by the blue and green arrows, respectively.



- Cross-sectional views of a Au/Ag nanobox can be obtained by slicing the image of its 3D electron density distribution at arbitrary cross sections.
- The obtained crosssectional images can be displayed as 2D electron density distributions. The spatial resolution of the cross-sectional profiles was found to be higher than 10 nm by analyzing the cross section of the thinnest structure in the cross-



II. (C) COHERENT DIFFRACTION

→ A small gold particle (size < 100 nm) is illuminated with a hard X-Ray nanobeam (E=15.25 keV, beam dimensions \approx 100x100 nm²) and is reconstructed from its coherent pattern. A resolution of <u>5 nm</u> is achieved in 600 S exposure time.

 \rightarrow What is next? By improving the refractive optics in term of image quality and transmission and by otimally matching the lateral coherence length of the incident beam to the aperture of the optic, the resolution could be pushed below 1 nm?



C.G.Schroer et al., PRL101,090801,2009

resolution by measuring oversampled diffraction patterns. The need for **isolated objects**, however, prevent a widespread and easy-to-use application of the method. Recently, two approaches have been demonstrated that enable CDI investigation of non isolated objects. [J.Miao, P. Charalambous, J.Kirz and D.Sayre (Nature 400, 342, 1999), M. A. Pfeifer et al.,(Nature 442, 63, 2006)]

•Ptychography uses multiple diffraction patterns from overlapping sample areas, that are reconstructed jointly by applying a complex iterative phase retrieval algorithm. [P. Thibault et al., [Science 321, 379, 2008]]

•Keyhole CDI uses a single diffraction pattern from a sharply defined, divergent beam to reconstruct a section of an extended object.[B. Abbey et al., [Nature Physics 4, 394, 2008]

• Fourier Transform Holography: much simpler is the image formation process using Fourier transform holography (FTH)-pattern yielding an unambiguous image of the object. As the phases are encoded in the hologram, several numerical contrast enhancing procedures, as e.g., Zernike or Schlieren phase contrast, can be applied to the image. The spatial resolution in FTH-based methods is limited by the size of the reference aperture-today FTH masks can be routinely produced with reference holes of 30 nm size. However, the image obtained by reverse Fourier transform provides an excellent starting point for a further phase retrieval treatment. In such a way the resolution limitation of FTH can be overcome [D Stickler et al., Applied Physics Letters 96, 042501, 2010]]



 X-ray magnetic circular dichroism (XMCD) is a difference spectrum of two xray absorption spectra (XAS) taken in a magnetic field, one taken with left circularly polarized light, and one with right circularly polarized light. By closely analyzing the difference in the XMCD spectrum, information can be obtained on the magnetic properties of the atom, such as its spin and orbital magnetic moment.



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- In the case of transition metals such as iron, cobalt, and nickel, the absorption spectra for XMCD are usually measured at the L-edge. This corresponds to the process in the iron case: with iron, a 2p electron is excited to a 3d state by an x-ray of about 700 eV. Because the 3d electron states are the origin of the magnetic properties of the elements, the spectra contain information on the magnetic properties.



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- We will see that this allows to measure the spin and the orbital momentum



 For an isolated electron, the orbital (L) and spin (S) magnetic moments can change provided that the total angular momentum of the particle is conserved. In condensed matter, an efficient transfer between L and S can occur owing to <u>the spin—orbit interaction</u>, <u>which originates in the relativistic</u> <u>motion of electrons</u>. Disentangling the absolute contributions of the orbital and spin angular momenta is challenging, however, as any transfer between the two occurs on femtosecond timescales. For an isolated electron, the orbital (L) and spin (S) magnetic moments can change provided that the total angular momentum of the particle is conserved. In condensed matter, an efficient transfer between L and S can occur owing to <u>the spin—orbit interaction</u>, <u>which originates in the relativistic</u> <u>motion of electrons</u>. Disentangling the absolute contributions of the orbital and spin angular momenta is challenging, however, as any transfer between the two occurs on femtosecond timescales.

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ferromagnetic films with a magnetization perpendicular to the plane of the film. Such materials are used for large storage densities in computer hard drives. The perpendicular magnetic anisotropy (PMA) responsible for the spin orientation along the disk normal has to be artificially induced by tailoring spin–orbit coupling in the material.

How to do that? By electronic hybridization of 3d transition metal valence levels (for example, of Fe or Co carrying large magnetic moments but relatively small spin–orbit coupling) with valence levels of 4d, 5d transition metals (for example, of Pd or Pt) with small magnetic moments but a large spin–orbit coupling. A layered sample structure can then induce a preferential spin orientation perpendicular to the layers. At the microscopic level, the change in ground state energy due to orienting the spin moment S is given by E_{PMA}=-ξ L.S, where ξ is the spin–orbit coupling parameter.

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- As the orbital and spin angular momentum can vary separately providing that the total angular momentum is conserved, a fundamental question is then: how do the orbital and spin magnetic moments change after an ultrafast laser excitation? On such ultrashort timescales (t,1 ps), the 34 way the electronic subsystem may exchange angular momentum is still debated.



• Geometry of the pump-probe experiment at the femtoslicing synchrotron beam line at BESSY. Time resolved XMCD allows measurement of the ultrafast dynamics of spin and orbital momenta along the quantification axis z parallel to the applied magnetic field.



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• Optical pulses with a central wavelength of $\lambda_{pump=}$ 579 nm and a duration of τ_{pump} =60±20 fs excite the ferromagnetic films perpendicularly, aligning the electric vector **E** in the film plane. The density of absorbed laser energy is E_{abs}=12 mJ cm⁻².



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• The ellipsoidal shape of -E_{PMA} illustrates the perpendicular anisotropy of the film. The easy magnetization direction is defined by the largest value of the z-axis projected value of L, (Lz). <u>On</u> applying the external magnetic field **H**_{ext}, the spin magnetic moment **S** aligns parallel to the orbital magnetic moment **L** along the z axis. A variable





Zholents et al, ALS, Berkeley



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- C is a constant related to the number of unoccupied 3d states that can be determined from the X-ray absorption spectra measured with linearly polarized radiation.



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•Static energy resolved X-ray absorption spectra of CoPd film using circularly polarized light. Two XAS spectra (red and black) and the normalized difference spectrum XMCD (line in blue) at the Co $L_{2,3}$ edges are displayed for the 15-nm Co_{0.5} Pd_{0.5} film in normal incidence geometry with a magnetic field of ± 4 kOe, collinear with the incident circularly polarized X-rays.



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 Integration of the energy resolved XMCD spectrum (green curve) allows a quantitative determination of the static values (without pump) of the spin and orbital magnetic moments at t<0: PUERTO VALLARTA




a, Sum rule extracted effective spin and orbital magnetic moments $S_z(t)$ and $L_z(t)$ as a function of the delay time between the laser pump and the X-ray probe. The continuous lines are fits obtained by using a 130-fs FWHM Gaussian function accounting for the time resolution of the experiment (including the X-ray probe and the fs laser pump). The blue dashed line represents the fit to $L_z(t)$ scaled to the value of $S_z(t)$ before laser excitation.



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b, The ratio $(L_z/S_z)(t)$ obtained as a function of the delay time shows that the orbital magnetic moment reduces more than the effective spin magnetic moment during the ultra fast demagnetization process. The black continuous line is the ratio between the two simulations of $L_{z}(t)$ and $S_{z}(t)$, showing a relative variation of $-29\pm5\%$. The red line is the ratio obtained when we take two identical values $\tau_{th}(L_z) = \tau_{th}$ $(S_7)=260$ fs. The error bars for $L_7(t)$, $S_7(t)$ and $(L_7/S_7)(t)$ are obtained from the error bars of the time resolved XMCD at the Co L_2 and Co L_3 edges.



CONCLUSIONS

- These measurements enable to disentagle the spin and orbital components of the magnetic moment, revealing different dynamics for L and S
- They highlight the important role played by the spinorbit interaction in the ultrafast laser-induced demagnetization of ferromagnetic films.
- They show that the magneto crystalline anisotropy energy is an important quantity to consider in such processes.

5.GRAPHENE



- a, The honeycomb lattice pattern of graphene explains its strength and good conductivity. Each carbon atom (green dot) uses three of its outer valence electrons to form strong covalent bonds, leaving one left over that is available for conduction.
- **b**, The quadratic, newtonian energy–momentum relation, $E=p^2/2m^*$ (*E*, energy; *p*, momentum; m^* , reduced mass) is obeyed by electrons in a semiconductor.
- **c**, **The energy–momentum** relation of electrons in graphene is quite different, *E*=*v*|*p*| (*v* is the electron velocity), allowing them to be modelled as massless, relativistic particles according to the Dirac formulation of quantum mechanics

GRAPHENE BILAYER: TUNABLE BANDGAP





The electronic structure near the E_F of an AB-stacked graphene bilayer features two nearly parallel conduction bands above two nearly parallel valence bands. In the absence of gating, the lowest conduction band and highest valence band touch each other with a zero bandgap. Upon electrical gating, the top and bottom electrical displacement fields D_t and D_b (Fig. 1c) produce two effects(Fig. 1d):





- The difference of the two, $\delta D = D_b - D_t \rightarrow \text{net carrier doping}(a \text{ shift of } (E_F))$. - The average of the two, $\check{D} = (D_b + D_t)/2$, breaks the inversion symmetry of the bilayer and generates a non-zero bandgap. The electronic structure near the E_F of an AB-stacked graphene bilayer features two nearly parallel conduction bands above two nearly parallel valence bands. In the absence of gating, the lowest conduction band and highest valence band touch each other with a zero bandgap. Upon electrical gating, the top and bottom electrical displacement fields D_t and D_b (Fig. 1c) produce two effects(Fig. 1d):





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- Left: Allowed transitions between different sub-bands of a graphene bilayer.
- Center: Gate-induced absorption spectra for different applied displacement fields. Absorption peaks due to transition I are apparent (dashed black lines are guides to the eye). The sharp asymmetric resonance observed near 200 meV is due to phonon resonances with continuum electronic transitions. The broad feature around 400 meV is due to electronic transitions II, III, IV and V.
- Right: Theoretical prediction of the gate-induced absorption spectra. The fit provides an accurate determination of the gate-tunable bandgap.
- IN CONCLUSION, THE BANDGAP GOER FOR AREA SELY CONTROLED FROM

6. MATERIALS UNDER EXTREME TEMPERATURE AND PRESSURE

THE NEW PICTURE A More Complex Planet

The earth is structured like an onion, with different materials appearing in each concentric layer. The discovery of a new, high-density material, called postporovskite, implies the existence of a new layer of that eater and explains pezziling behavior by seismic waves traveling through the planet.

CRUST (UP TO 35 KILOMETERS OF DEPTH)

The continents, which are in part submerged by the oceans, are made of overse rock that is up to several billion years old and estatively light. Thus, they had on the denser mantie undermostin. The heavy bualdic rock that forms the built of the oceanic crust originates from mantie magma that erupts at underwater ridges and eventually sinks back into the manife, typically within 100 million years.

ALC: NOT THE

Mantie rock consists primarily of oxygen, silicon and magnesium. Despite being mostly solid, it does deform on peologic timescales. In fact, the rock slowly flows as convec-tive currents stir the entire mantle. That flow dissipates the earth's inner heat and propets continental drift.

UPPER MANTLE (35-660 KM)

Ar greater depite tring higher pressures and temperatures, the martle's elemental components arrange into different crystal structures (minerals), for ming layer, three minerals — of him, modified spinal and apriled — give the layer of the upper mantle their respective names.

LOWER MANTLE (660–2, 900 KM) The lower martle was for decades thought to be relatively uniform in structure. Ind setmenting cal data suggested that something different was happening at the potters.

 Perovskite layer The most provalent mineral hero (76 percent by weight) is a magnesium silicate (MgSIO) belonging to the family of crystal structures called perovikites. In this densety packed persentation, in this densely packed structure, magnetistum iters (wellow) are sumpainded by octahedral silicon-integrity drags tokar double-governed shaped. Units incoming, scientifists thought that no dense crystal arrangement of these ele-ments could excit.

Postperovskite layer

At the pressures and temperatures of the bottom 300 km of the mantle, perovskite transforms into a new structure: the magnesium ions and the silicon-oxygen groups arrange themselves into separate layers. The transition releases heat and reduces. dramatic effects on the entire planet Date Allustrations on pages 82-81





The despect part of the earth consists predominantly of iron, which is liquid in the outer core and solid in the inner core. Convection stin the outer core just an II stin the mantle, but because the core is much dense. If the midal goccars between the mantle and the core. Core convection is thought to produce the planet's magnetic field.



GEOPHYSICS AND MATERIALS: EXTREME CONDITIONS

- Crust 0–35 km <1 Gpa, 200–600
- Upper mantle 35-660 km 1-25 Gpa, 600-1600
- Lower mantle 660–2890km, 25–136 Gpa, 1600–4000
- Outer core 2890-5150 km, 136-330 Gpa, 4400-6100
- Inner core 5150–6360km, 330–360 Gpa, 6100K (±500K)



INTERFACE MANTLE-FLUID CORE 5200 K, 140 GPa

SOLID CORE: P= 360 GPa, T= 6000 K

HOW TO REPRODUCE THESE TEMPERATURES AND PRESSURES? Diamond Anvill-Cell+Laser X-Ray beam: few µm diameter

THE DEEP EARTH AT SYNCHROTRONS



Beam size for the X-Ray: 1-2 µm Beam size for the laser: 20 μ m \rightarrow to avoid T gradients

The Structure of Iron in Earth's Inner Core

Shigehiko Tateno, Kei Hirose, Yasuo Ohishi, Yoshiyuki Tatsumi SCIENCE 330, 359, 2010

Earth's solid inner core is mainly composed of iron (Fe). Because the relevant ultrahigh pressure and temperature conditions are difficult to produce experimentally, the preferred crystal structure of Fe at the inner core remains uncertain. Static compression experiments showed that the hexagonal closepacked (hcp) structure of Fe is stable up to 377 gigapascals and 5700 K(melting), corresponding to inner core conditions. The observed weak temperature dependence of the c/a axial ratio suggests that hcp Fe is elastically anisotropic at core temperatures. Preferred orientation of the hcp phase may explain previously observed inner core seismic anisotropy.



Representative XRD patterns of hcp Fe at : (A) 332 GPa and 4820 K and (B) 356 GPa and 5520 K. The peak positions of the bcc and fcc phases were calculated for volumes larger by 0 to 1% than that for the observed hcp phase.

hcp, hcp Fe; py, pyrite-type SiO_2 (pressure medium); C, Fe_3C



Phase diagram of Fe and the inferred temperature profile inside Earth.

Open symbols indicate the present results (different symbols indicate different runs), and solid diamonds indicate data from previous experimental work . The lowpressure solid-solid phase transition

boundaries and melting curve are from Boehler . Liq., liquid.

(Inset) Sample photograph at 335 GPa in the DAC.



Peridotite at 5200 K and 140 GPa (SiO₂, 44.3 %; MgO, 41.6;FeO, 7.3; MnO, 0.1, TiO₂, 0.2; Al₂O₃, 2.4; CaO, 1.6; Cr₂O₃, 0.1)





G. Fiquet, A. L. Auzende, J. Siebert, A. Corgne, H. Bureau, H. Ozawa, G. Garbarino SCIENCE 329, 1561, 2010

Scanning electron microscopy image of a "mantle" sample after transformation, stuck on a copper grille and thinned down by a focused ion beam (FIB). It allows to detect the different synthesized minerals and liquids during these experiments: a matrix, consisting of a phase of a perovskite structure ((Mg,Fe)SiO₃), - the most abundant mineral in the Earth because it is the most stable in the inferior mantle), is shown in light grey. The veins and liquid pockets enriched in iron and calcium are visible (in dark grey). Scale of the horizontal bar is 2 µm. Credits: G.Fiquet.
Hot spot: 20 µm, X-Ray beamsize: 1.7x2.3 µm at 33. 3 keV

At high temperature, pressures were measured from cell parameters of the magnesium perovskite (Mg,Fe)SiO₃ by using a thermal equation of state recently reported for the same KLB-1 peridotitic starting material as used in our study (18). We used in situ x-ray diffraction as primary criterion for melting) and to determine the order in which crystalline phases melt.

Diffraction patterns collected at 61 GPa after normalized reference background subtraction:subsolidus at 2715 K (bottom) and above solidus at 3750 K (top). The diffuse scattering liquid contribution is outlined by the shaded area as a guide; it does not correspond to a physical structural model of the liquid. HKL indexes are given for remaining diffraction peaks that can be assigned to magnesium silicate perovskite, observed above the solidus temperature at this pressure (top). Stars denote diffraction peaks of Ca-perovskite and ferropericlase



 Promoting freezing in a liquid is straightforward : you simply add suitable templates. The templates can be either 'seeds' of the crystalline phase that would form from the liquid, or small crystals of another material whose atomic-level surface structure in some way matches that of such seeds.

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- What is more difficult to conceive of is a solid surface that inhibits freezing by acting as a template for the liquid. However, recently, Schulli, Daudin, Renaud, Vaysset, Geaymond & Pasturel [Nature 464, 1192, 2010] describe evidence suggesting that such a template is possible. Their results have wide implications not only for fundamental studies of freezing, but also for the practical control of this phase transition.

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- The cooling of a liquid to below the ideal freezing temperature, known as supercooling is of great interest in diverse areas ranging from the control of microstructure in metallic welds and castings to the inhibition (or promotion) of ice formation necessary for the survival of living systems.


For liquids in contact with solids, crystalline surfaces induce layering of the adjacent atoms in the liquid and may prevent or lower supercooling. This seed effect is supposed to depend on the local lateral order adopted in the last atomic layers of the liquid in contact with the crystal. Although it has been suggested that there might be a direct coupling between surface-induced lateral order and supercooling, no experimental observation of such lateral ordering at interfaces is available.

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In situ X-ray scattering and ab initio molecular dynamics reveal that <u>pentagonal</u> <u>atomic arrangements of Au atoms at this interface [Si(111) 6x6] favour a lateral-ordering stabilization process of the liquid phase</u>. This interface-enhanced stabilization of the liquid state shows the importance of the solid–liquid interaction for the structure of the adjacent liquid layers. Such processes are important for present and future technologies, as fluidity and crystallization play a key part in soldering and casting, as well as in processing and controlling chemical reactions for microfluidic

11/29/10

ADVANTAGES OF THE X-RAY

You can probe simultaneously:

- the bulk
- the surface layer (reconstruction)
- the liquid

LIQUID	
Bulk Si	



Surface layers

T. U. Schülli, R. Daudin, G. Renaud, A. Vaysset, O. Geaymond & A. Pastrurel Nature 464, 1174, 2010



Step 1: Seven monolayers of Au are deposited at room temperature.

Step 2: On annealing they transform into crystalline Au islands.

<u>Step 3</u>: At T_E =636 K, melting sets in and AuSi droplets with the eutectic composition (Au_{81.4}Si_{18.6}) are formed.

<u>Step 4</u>: Heating up to 673K before cooling induces a (6x6) reconstruction, and leads to a preservation of the liquid phase down to 513K(<u>step 5</u>), where phase separation and solidification occur (step 5). Above T_E , on heating or cooling, the liquid composition is expected to follow the Si liquidus. Below T_E , it follows the (extrapolated; dotted line) metastable Si liquidus. The degree of supercooling (red arrow) has to be measured between this latter and the Au liquidus above T_E for the corresponding composition of ~15 at.% Si. It amounts to ~ 3 60 K because as the liquid alloy droplets cool, Si comes out of solution and redeposits on the substrate. The observed freezing point of 513 K represents a supercooling of 360 K below the liquidus of the resulting composition.

Figure 2 | Reciprocal space mapping of liquid AuSi islands on (6x6) reconstructed Si(111).



a, Reciprocal space map of the liquid in its supercooled state on a (6x6) reconstructed Si(111) surface. Blue colour corresponds to low intensity, and red to high intensity, yellow being intermediate. Three bulk Bragg peaks are visible, together with a mesh of smaller peaks arising from the (6x6) surface/interface periodic superstructure. The three diffuse rings correspond to liquid-like scattering.

b, Anisotropy of the first order maximum of the liquid structure factor: In the vicinity of strong (6x6) reconstruction peaks the signal from the liquid is enhanced, underlining morphological similarities between the crystalline surface and the adjacent liquid layers.

c, Right: scans across the first ordermaximum of the liquid structure factor in the plane (along section S marked in a and b) and parallel to it for several values of out-of-plane momentum transfer, Q_z . Left: the sketch indicates in orange the position of the first maximum of the isotropic liquid. The green poercovals to the first distribution stemming from preferential in-plane order.

Figure 3 | Evolution of the liquid structure factor during cooling and solidification.



a, Angular average of the experimental structure factor S(Q) of liquid AuSi at 563K (black line) together with the theoretical structure factor extracted from MDS at 600K(red line). The blue line corresponds to the mean structure factor of the (6x6) reconstruction.

b, Zoom (logarithmic scale) on the low-Q spectrum of the structure factor, showing the Bragg peaks from two-dimensional crystallites floating on the surface of liquid AuSi.

c, Hysteresis loops of the integrated intensity of the Au(220) Bragg peak during the solid–liquid–solid transition of Au islands on Si(001) (black open circles), on an Si(111)-($\sqrt{3}x\sqrt{3}$)R 30° reconstruction (blue filled circles) and on an Si(111)-(6x6) reconstruction (red squares).

d, Liquid structure factor (logarithmic scale) along the <110>. crystallographic direction of the Si(111) surface. The strong influence of the appearance of the (6x6) reconstruction on the structure of the liquid is visible.

PUERTO VALLARTA

• Evolution of the first maximum of the liquid structure factor in the supercooling regime

Figure 4 | Au-induced Si(111)-(6x6) surface leading to enhanced supercooling. Unit cell (black lozenge) of the complex (6x6) reconstruction (only the Au atoms are shown) formed at T<673K after annealing temperatures T>673 K. A pentagonal cluster (see inset three-dimensional structure) present in the simulated liquid has similar topology and bond length (2.84 Å) as the surface structure (2.86 Å) smaller than in the Au f.c.c. lattice (2.90 Å). <u>Out of 45 atoms in the unit cell, 30 are in a pentagonal environment (interconnected by blue lines).</u>



However, when the alloy droplets freeze on the 6 × 6 silicon surface, the resulting gold crystals form in random orientations. This suggests that the substrate has no orienting role in freezing; the actual site and mechanism of crystal nucleation remain undetermined.

8. Conclusions

- There are already beamlines with beam size of 100x100nm
- 30x30 are in preparation but this needs new technology, with AFM on the BL, otherwise you will never align the sample and the beam. I addition thermal noise, electrical noise, vibrations have to be tackled.



ESPCA ESCOLA SÃO PAULO DE CIÊNCIA AVANÇADA

THE NEW DEVELOPMENTS IN THE FIELD OF SYNCHROTRON RADIATION

The State of São Paulo would like to increase the number of foreign pos-docs. For that, it is creating "Advanced schools" on different topics. The goal is to have 100 students, during their PhD: 50% from Latin America and 50% from other parts of the world. The school will not deal with the techniques, but with the scientific domains: structural biology, 3D imaging, magnetism, nanosciences, catalysis, etc.

DATES

From January 17th till 25th 2011, followed by 2 days visiting universities in Sao Carlos, Campinas and Sao Paulo.

SUBMISSION OF APPLICATIONS

Application period from July 01st till October 15th, 2010. The acceptance of the school will be notified by e-mail on October 18th, 2010.

LOCATION

Brazilian Synchrotron Light Laboratory (LNLS) Rua Giuseppe Máximo Scolfaro, 10.000 - Pólo II de Alta Tecnologia Campinas - SP - Brazil



STRUCTURAL BIOLOGY

Ada Yonath (Weizmann Institute of Science, Israel) J.A. Manguez (EMRL, Grenoble, France) José Nelson Onuchic (USCD, USA) Frederic Leroy (L'Oréal, Paris) Igor Polikarpov (USP, São Paulo, Brazil)

3D X-RAY IMAGING

Franz Pfeiffer (T.U. Munich) Paul Tafforeau (ESRF, Grenoble) Wolgang Ludwig (CNRS, Lyon) Jin Wang (APS, USA)

CATALYSIS

M. Salmeron (UC, Berkeley, USA) Gemma Guilera (ALBA, Barcelona) Daniela Zanchet (LNLS, Brazil)

MAGNETISM AND SUPERCONDUCTIVITY

Albert Fert (Orsay, France) Sebattian Stepanow (Max Planck Institut, Germany) S.V. Borisenko (FW, Dreiden, Germany) S. Louie (UC, Berkeley, USA)

NANOSCIENCES

S. Louie (UC, Berkeley, USA) D. Ugarte (Unicamp, Campinas, Brazil) R. Panniago (UFMG, Minas Gerais, Brazil) Galo Soler Illia (CNEA, Argentina)

ENVIRONMENT

P. Eduardo Artaxo Netto (IFUSP, São Paulo, Brazil)

ORGANIZATION

Lodging, transportation and air tickets will be covered and organized by the School



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