



CGE 2022 – Cristallographie et Grands Equipements

17 – 21 octobre 2022

Synchrotron SOLEIL, Saint-Aubin, France

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Programme

Lundi, 17 octobre

10:00 - 10:45 ACCUEIL

10:45 - 11:00 Bienvenue et Introduction

Amphithéâtre SOLEIL

Cours :

11:00 - 12:30 Cristallographie géométrique, symétries I
Delphine Cabaret

12:30 - 13:30 Déjeuner

Amphithéâtre SOLEIL

Cours :

13:30 - 15:30 Cristallographie géométrique, symétries II
Delphine Cabaret

15:30 - 16:00 Pause-café

Amphithéâtre SOLEIL

Atelier :

16:00 - 17:30 Cristal, réseau direct
Greg Cabailh

18:00 - 19:00 Session poster I

19:00 - 20:00 Diner

20:00 – 21:00 Session poster II



Mardi, 18 octobre

Amphithéâtre SOLEIL

Cours :

9:00 - 10:30 Diffusion Thomson, théorie cinématique I
Sylvain Ravy

10:30 - 11:00 Pause-café

Amphithéâtre SOLEIL

Cours :

11:00 - 12:30 Diffusion Thomson, théorie cinématique II
Sylvain Ravy

12:30 - 13:30 Déjeuner

Amphithéâtre SOLEIL

Cours :

13:30 - 14:30 SR, instrumentation
Jean-Paul Itié

Travaux dirigés :

Salle PHENIX

Groupe A: Groupes

Sébastien Pillet

14:30 - 15:30 Salle de formation T5
Groupe B: Facteurs de structure
El Eulmi Bendeif

15:30 - 16:00 Pause-café

Travaux dirigés :

Salle de formation T5

Groupe A: Facteurs de structure

Sébastien Pillet

16:00 - 17:00 Salle PHENIX
Groupe B: Groupes
El Eulmi Bendeif

17:30 - 19:00 Amphithéâtre SOLEIL - Conférence grand public – René Guinebretière

19:00 Diner



Mercredi 19 octobre

Amphithéâtre SOLEIL

Cours :

9:00 - 10:30 Analyse de données monocristaux

Sébastien Pillet

10:30 - 11:00 Pause-café

Amphithéâtre SOLEIL

Cours :

11:00 - 12:30 Analyse données poudres

Erik Elkaïm

12:30 - 13:30 Déjeuner

Travaux pratiques :

Salle PHENIX

Groupe A: Monocristaux

13:30 - 18:00 *Lise Chamoreau et El Eulmi Bendeif*

Salle de formation T5

Groupe B: Poudres

Erik Elkaïm et Hubert Chevreau

18:30 - 20:30 Visite de SOLEIL

20:30 Diner



jeudi 20 octobre

Amphithéâtre SOLEIL

Cours :

9:00 - 11:00 Neutrons et diffraction magnétique

Françoise Damay et Sylvain Petit

11:00 - 11:30 Pause-café

Amphithéâtre SOLEIL

Cours :

11:30 - 13:00 Pdf, neutrons et rayonnement X

Pierre Bordet

13:00 - 14:00 Déjeuner

Travaux pratiques :

Salle PHENIX

Groupe A: Poudres

14:00 - 18:30 *Erik Elkaim et Hubert Chevreau*

Salle du Pavillon d'accueil - A1.12

Groupe B: Monocristaux

Lise Chamoreau et El Eulmi Bendeif

19:00 Diner de Gala



vendredi 21 octobre

Amphithéâtre SOLEIL

Cours :

9:00 - 10:00 Surfaces, interfaces

Alessandro Coati

Amphithéâtre SOLEIL

Cours :

10:00 - 11:00 Désordre

Pascale Launois

11:00 - 11:30 Pause-café

Amphithéâtre SOLEIL

Cours :

11:30 - 12:30 Temps résolu

Claire Laulhé

12:30 - 13:30 Déjeuner

Amphithéâtre SOLEIL

Cours :

13:30 - 15:00 Cohérence

Vincent Jacques

15:00 - 16:00 Bilan de l'école



Liste des cours, travaux pratiques et travaux dirigés

Cours	Intervenant	N° Fichier
Cristallographie géométrique, symétries	D. Cabaret	01
Diffusion Thomson, théorie cinématique	S. Ravy	02
SR, instrumentation	J.P. Itié	03
Analyse de données monocristaux	S. Pillet	04
Analyse données poudres	E. Elkaïm	05
Neutrons et diffraction magnétique	F. Damay, S. Petit	06, 06b
Pdf, neutrons et rayonnement X	P. Bordet	07
Temps résolu	C. Laulhé	08
Désordre	P. Launois	09
Surfaces, interfaces	A. Coati	10
Cohérence	V. Jacques	11
Travaux pratiques	Intervenant	Dossier
Monocristaux	L. Chamoreau et E. Bendeif	TP-01
Poudres	E. Elkaïm et H. Chevreau	TP-02
Travaux dirigés	Intervenant	Dossier
Atelier : Cristal, réseau direct	G. Cabaillh	TD-01
Facteur de structure, mode de réseau et extinctions	S. Pillet	TD-02
Symétries et Groupes	E. Bendeif	TD-03



Abstracts des participants

Abstact-01

Solving Acetyltransferase structures: An insight into substrate selectivity
E. Alekseenko - CHRODYN team, IJPB, INRAE, Versailles, France

Abstact-02

Structural and magnetic properties of YNi_{3-x}F_x compounds and their hydrides
H. Bénet - Université Paris-Est, ICMPE, Thiais, France

Abstact-03

Effect of side chain end group in conducting polymer on ultra-thin film organization
H. Fernandez - CY Cergy and Synchrotron SOLEIL, Saint Aubin, France

Abstact-04

In situ diffraction study of the phase transformations occurring in the thermoelectric colusite Cu₂₆V₂Sn₆S₃₂
F. Guiot - Université de Rennes, CNRS, ISCR-UMR 6226, Rennes, France

Abstact-05

Exploration of the Fe(Nb_{1-x}W_x)O₄ solid solution for structural and magnetic properties
L. Spejchalová - University of Chemistry and Technology Prague, Czech Republic

Solving Acetyltransferase Structures: An Insight into Substrate Selectivity

Ekaterina Alekseenko, Jeffrey Leung, Valérie Gaudin

*CHRODYNO team, IJPB, INRAE Centre Ile de France de Versailles-Saclay,
Route de St-Cyr (RD10), 78026 Versailles Cedex, France*

ABSTRACT

NATA1, an *Arabidopsis thaliana* (thale cress) acetyltransferase, is involved in drought and biotic stress responses. With its' close homologue NATA2, it represents a novel family of the GCN5-related N-acetyltransferase (GNAT) superfamily. GNATs are present in all domains of life and are involved in many crucial processes, including gene expression regulation, stress response and metabolism. They catalyze the transfer of an acetyl group from Acetyl-CoA onto a primary amine, and, despite having a conserved GNAT catalytic domain, act on a variety of substrates, from large histones to small polyamines. Questions remain regarding how GNAT structure drives their substrate selectivity, particularly regarding small molecules. For example, why can some GNATs accommodate a variety of substrates while others only act on a certain type, or how even highly similar GNATs achieve distinct substrate selectivities. Despite the challenges of co-crystallizing proteins with small molecules, especially in the case of weaker binding, X-Ray crystallography remains one of the best methods to observe GNAT interaction with their substrates directly. NATA1 and NATA2, whose structures have not yet been solved, appear to be selective for small polyamines and have distinct substrate selectivities despite an 80% sequence identity. Thus, one goal of my project is to crystallize these acetyltransferases in the presence or absence of their preferred substrates, to determine how these selectivities are achieved on a structural level.

Structural and magnetic properties of $\text{YNi}_{3-x}\text{Fe}_x$ compounds and their hydrides

Hugo Bénet, Junxian Zhang and Valérie Paul-Boncour

Université Paris-Est, ICMPE (UMR7182), CNRS, UPEC, F-94320 Thiais, France

ABSTRACT

Rare earth-transition metals alloys can store large amount of hydrogen (up to 2 wt% of hydrogen) in ambient conditions with a good volumetric capacity. They are also interesting for their magnetic properties which are associated with itinerant and localized magnetism¹. We are interested on $\text{YNi}_{3-x}\text{Fe}_x$ ($x = 0 ; 0.25 ; 0.5 ; 1.5$) compounds and their hydrides, there are few studies on their hydrogenation and magnetic properties and those studies are relatively old^{2,3}. The Rietveld analysis of powder X-ray diffraction (XRD) patterns has shown that those compounds have a majority phase with a rhomboedral structure (PuNi₃ type) and few percent of a secondary phase YNi_2 with a cubic structure (for $x = 0.25$ and $x = 0.5$). An increase of the lattice parameters is observed as a function of iron concentration. The hydrogenation of those compounds reveals that YNi_3 can absorb 1.42 wt % of hydrogen and the PCI of YNi_3 at 25°C exhibits two plateau pressure. Compounds with iron absorb hydrogen with a slower kinetic than YNi_3 . Structural properties of YNi_3 and $\text{YNi}_{2.75}\text{Fe}_{0.25}$ hydrides have been studied by XRD. Both hydrides have the same structure than their parent compounds but hydrogenation leads to an increase of 22% of the cell volume of YNi_3 and $\text{YNi}_{2.75}\text{Fe}_{0.25}$. Magnetic measurements show that all the intermetallic compounds are ferromagnetic with a non-linear increase of the Curie temperature as well as a non-linear increase of their spontaneous magnetization as a function of iron concentration. This has been attributed to Fe substitution in different Ni sites depending on Fe concentration. ZFC-FC measurements below 1 kOe have shown some reentrant behavior linked to magnetic irreversibilities for low iron concentrations ($x = 0.25$ and $x = 0.5$).

REFERENCES

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Effect Of Side Chain End Group In Conducting Polymer On Ultra-Thin Film Organization

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Regioregular poly(3-alkylthiophenes) are rigid conducting polymers widely used in different devices such as field effect transistors or photovoltaic cells, but also as surface coatings to tune wetting properties or for biological applications^{1,2}. The device performances strongly depend on the polymer organization. π - π interactions between thiophene units lead to the formation of semicrystalline thin films at room temperature, in which the polymer tends to adopt a nanofibril organization. Nevertheless, the organization degree significantly depends on the processing conditions (deposition method, solvent, concentration...), showing the importance of the film elaboration technique to control the polymer organization³. As it leads to highly ordered layers, we propose to use the Langmuir film technique to provide an alternative way to the deposition by spin-coating or drop-casting generally used for device elaboration.

We are thus studying regioregular electronically conducting polymers at the air-water interface and onto solid substrate, namely poly(3-hexylthiophene) (P3HT) and poly[3-(potassium-7-heptanoate) thiophene-2,5-diyl] (P3HTCOOK) (**Figure 1.A**). The objective is to investigate the effect of the dipole moment (CH_3 or COOK) of the side chain on the film structure. We used a wide variety of experimental techniques to characterize the films at the air-water interface. X-Ray Reflectometry and Total Reflection X-Ray Fluorescence experiments show that P3HT forms a bilayer (edge-on structure) at the air-water interface while P3HTCOOK has a monolayer organization (**Figure 1.B**). The results are in agreement with the topography of the films transferred onto solid substrate, as determined by Atomic Force Microscopy. Grazing Incidence X-ray Diffraction (GIXD) experiments show a long-range in-plane organization of the P3HT film unlike the P3HTCOOK film having a shorter-range positional order (**Figure 1.C**). P3HT Langmuir films are organized according to a rectangular lattice with $a=0.778 \text{ nm}$ and $b=0.768 \text{ nm}$ (**Figure 1.D**). The a direction corresponds to the polymer backbone while the b one is associated to the π - π stacking of the thiophene units on adjacent polymer chains. Structure factor calculation enabled us to determine fine details of the P3HT organization in the unit cell.

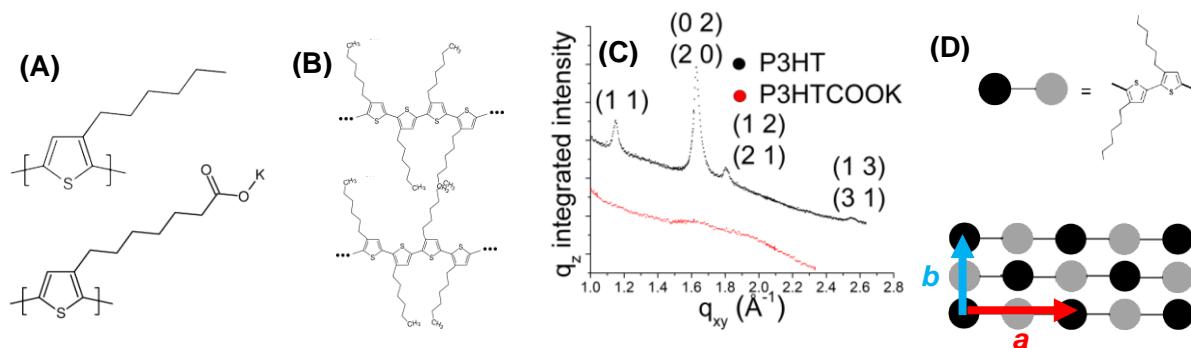


Figure 1: Chemical structure of P3HT and P3HTCOOK (**A**). Vertical structure for the P3HT Langmuir film (**B**). GIXD spectra of P3HT and P3HTCOOK Langmuir films at 5mN/m (**C**). Rectangular in-plane cell for the P3HT Langmuir film (**D**).

REFERENCES

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3. Kim Y., Jung H., Ahn C., Jeon H.; Adv. Mater. Interfaces, 4, 1700342 (2017).

In Situ Diffraction Study of the Phase Transformations Occurring in the Thermoelectric Colusite Cu₂₆V₂Sn₆S₃₂

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ABSTRACT

Interest in thermoelectric (TE) technology has been continuously growing in the last decade driven to the necessity to limiting waste heat during energy transformation. Among the most promising TE materials at medium temperature, complex copper-based sulphides are of double interests as they are usually made of eco-friendly and low cost elements[1] and exhibit intrinsically low thermal conductivity.[2,3] Derivatives of the natural mineral colusite, with general formula Cu₂₆A₂E₆S₃₂, (A = V, Nb, Ta, Cr, Mo, W; E = Ge, Sn, As, Sb), are an emerging class of excellent thermoelectric materials.[4] As example, the ZT value of the colusite Cu₂₆V₂Sn₆S₃₂ rises to near unity at 675K, making this material one of the best p-type TE in this temperature region.[5] Its performances are mainly related to the coexistence of an ordered (*P*-43*n*) and a disordered (*F*-43*m*) forms obtained after sintering at 1023 K (i.e. sample H), leading to a very low thermal conductivity.[5][6] In addition, colusite Cu₂₆V₂Sn₆S₃₂ is known to exhibit an intrinsic exsolution phenomenon supposed to be related to the coexistence of Sn-rich and Sn-poor colusites.[7,8]

In this study, we investigated by *in-situ* synchrotron powder diffraction the solid-state phase equilibrium as function of the temperature between the ordered and disordered forms of colusite Cu₂₆V₂Sn₆S₃₂. The use of high-resolution setup revealed a complex behavior with several phase transformations, probably related to a mutual interaction and kinetic effects.

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Exploration of the $\text{Fe}(\text{Nb}_{1-x}\text{W}_x)\text{O}_4$ Solid Solution for Structural and Magnetic Properties

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ABSTRACT

FeNbO_4 and FeWO_4 are both part of a large family of compounds, which have promising applications in photocatalysis^{1, 2}, batteries^{3, 4} and sensors^{5, 6}. FeNbO_4 has been reported to crystallize in three polymorphic forms (tetragonal, orthorhombic, and monoclinic $P2/c$)⁷, whereas FeWO_4 crystallizes only in the monoclinic symmetry $P2/c$.⁸

The solid solution $\text{Fe}(\text{Nb}_{1-x}\text{W}_x)\text{O}_4$ has been first reported in a preliminary study done by Noda *et al.*⁹ in regard to its electric and magnetic properties, as well as the initial work on the effects of replacing niobium for tungsten on the change in cell parameters. The solid solution combines both end-members (FeNbO_4 and FeWO_4) in the monoclinic symmetry $P2/c$.

Here, we also report solid state synthesis of the solutions in the range $0 \leq x \leq 1$ (including the end members FeNbO_4 and FeWO_4), discuss the change of cell parameters and how it affects the unit cell based on a Rietveld refinement of XRD diffractograms. The magnetic properties (using SQUID measurements) have been also studied in more detail to further investigate the structural changes in the materials. Future work is focused on combining the experimental results with theoretical DFT calculations.

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