At IUCr2008, we proposed a new rapid exhaustive search powder indexing algorithm that was obtained by developing the Ito algorithm from various aspects. Several amendments have been added to the algorithm after that, and it was implemented into a powder indexing software *Conograph*. At IUCr2011, the algorithm and its performance are presented. The name of the software is taken from a mathematician J. H. Conway and a graph he observed in [1]. The graph is utilized to detect zones having more possibilty to correspond to a sublattice of the true crystal lattice (*cf.* [2]) and to describe a theorem about the distribution of unobserved reflections due to extinction rule. The theorem is found and confirmed by the first author and gives a theoretical foundation of our algorithm.

The main function of Conograph consists of premitive unit-cell determination, lattice symmetry determination and lattice paramter refinement. In these procedures, observed errors of peak-positions are utilized to check the equality of equations including the Ito equation $2(q_1+q_2) = q_3 + q_4$[3], to calculate propagated errors of lattice constants for lattice symmetry determination, and as weights in least squares method to refine lattce constants.

Conograph is already equipped with a graphic user interface to help users find the most plausible candidates easily. Several figures of merit(FOM) are prepared to sort the candidates: the de Wolff FOM [4], a symmetric FOM we propose as a natural extension of the de Wolff FOM, the number of lattice candidates detected in the neighbor of each candidate, and the unit-cell volume. For further improvements in the usability of the software, Conograph is planned to be distributed free including among users of Japan Proton Accelerator Research Complex (J-PARC).

[1] J. H. Conway **1997** *"The Sensual (Quadratic) Form"*, **1997**, The mathematical association of America [2] R. Oishi, M. Yonemura, T. Ishigaki, A. Hoshikawa, K. Mori, T. Morishima, S. Torii, T. Kamiyama *Zeitschrift für Kristallographie Supplements* **2009**, *30*, 15-20 [3] P.M. de Wolff *J. Appl. Cryst.* **1957**, *10*, 590-595 [4] P.M. de Wolff *J. Appl.Cryst.* **1968**, *1*, 108-113.

Keywords: powder indexing, algorithm, extinction rule

MS.95.5

Chemical selectivity in structure determination by modulation enhanced X-ray diffraction

Marco Milanesio,[a] Wouter van Beek,[a,b] Luca Palin,[a] Rocco Caliandro,[c] Davide Viterbo,[a] Atsushi Urakawa,[d] Dmitry Chernyshov,[b] Hermann Emerich,[b] *[a] Dipartimento di Scienze e Tecnologie Avanzate and Nano Sistemi IC, Università del Piemonte Orientale, Viale T. Michel 11, Alessandria, 15121, (Italy). [b] Swiss-Norwegian Beamlines at ESRF, BP 220, Grenoble, 38043, (France). [c] Institute of Crystallography – CNR, Via G. Amendola 122/O, 70126 Bari, (Italy). [d] Institute of Chemical Research of Catalonia (ICIQ), AV. Països Catalans, 16, E-43007, Tarragona, (Spain).* E-mail: marco.milanesio@mfn.unipmn.it

X-ray diffraction methods in general allow only a limited chemical selectivity. In this contribution we show that structural information on a subset of atoms can be selectively obtained in a diffraction experiment, where the sample is subjected to a periodically varying external stimulus (temperature, pressure, concentration, light etc). This technique is called modulation enhanced diffraction (MED) [1]. In MED, the periodically varying data are averaged and post-treated by phase sensitive detection (PSD). PSD is a demodulation technique exploited in modulation excitation spectroscopy (MES) to obtain information on active species diluted into a spectator inactive matrix. In analogy to MES, MED extracts crystallographic information on a subset of atoms responding to an external stimulus, thus introducing

selectivity in diffraction. Simulated and experimental MED data were produced by using a TS-1 zeolite sample as spectator, in which Xe (acting as active species) is adsorbed and desorbed by a periodic temperature change. For both simulated and experimental data, MED yielded the powder diffraction pattern of the active subset, called the "demodulated pattern". The demodulated pattern was used to solve the crystal structure of the active species exclusively, demonstrating the chemical selectivity of MED and envisaging wide applications in the structure analysis of periodically changing materials (magnetic information storage systems, electrochemical systems such as batteries, solar cells, etc).

[1] D. Chernyshov, W. van Beek, H. Emerich, M. Milanesio, A. Urakawa, D. Viterbo, L. Palin, R. Caliandro, *Acta Crystallographica Section A67* **2011**, *in press*.

Keywords: structure solution, modulation enhanced diffraction, chemical selectivity.

MS.96.1

Polarized Neutron and Light Scattering from Magnetic Nano-structures under AC-field

Boris P. Toperverg, *Ruhr-University-Bochum (Germany). Petersburg Nuclear Physics Institute (Russia).* E-mail: Boris.Toperverg@rub.de

Complementarity of Polarized Neutron and Soft X-rays Reflectometry (PNR and SXRR) has experimentally been demonstrated in a number of studies in which these methods were combined to refine static magnetic structures in thin films, super-lattices and lateral nano-patterns [2]. Both methods use similar principles [1] of the data acquisition, theoretical description and analysis providing a comparable resolution in layer-by-layer vector magnetometry. The effect of spin birefringence in PNR is dual to the X-ray magnetic circular dichroism (XMCD) so that the depth sensitivity of PNR is well complemented by the element specificity of SXR. Furthermore, off-specular PNR probes lateral magnetic structures over micrometer scale reciprocal to that accessed with the direct space photoemission electron microscopy (PEEM) with resonant circular polarized light (RCPL). Smaller scales probed by polarized neutron grazing incidence small angle scattering (PN GISAS) can also be approached with RCPL GISAS.

Both methods, PNR and SXRR, have recently shown potentials to probe not only static properties of magnetic nano-structures, but also their evolution under time-dependent external field. Nearly instant X-ray response for a magnetization variation allows to access fast spin kinetics in individual nano-elements. In contrast, PNR can, as was recently demonstrated [3], records collective response of magnetic nano-structures to AC field varied over a broad range of amplitudes and frequencies (up to a few MHz). This, as will be thoroughly discussed, opens up new perspectives to experimentally distinguish between various re-magnetization scenarios in ensembles of lateral nano-patterns. First results on domain kinetics in continuous and periodically patterned magnetic films and heterostructures deduced from AC PNR experiments are reported. Further developments, including application of the time modulation of the incident neutron beam synchronized with AC field at the sample position will be outlined. Feasibility of magnon spectrometry with inelastic PNR under condition of ferromagnetic resonance is theoretically examined.

[1] H. Zabel, K. Theis-Bröhl, B.P. Toperverg, *Handbook of Magnetism and Advanced Magnetic Materials, ed.* H. Kronmüller and S. Parkin, (New-York: Wiley) **2007**, *12*, 1237-1308. [2] H. He, K. Zhernenkov, M. Vadala, N. Akdogan, D. Gorkov, R.M. Arbudan, B.P. Toperverg, H. Zabel, H. Kubota,

S. Yuasa *J. Appl. Phys.* **2010,** *108*, 5063922-1. [3] K. Zhernenkov, S. Klimko, B.P. Toperverg, H. Zabel, *Jornal of Physics: Conference Series* **2010,** *211*, 012016-1.

MS.96.2

Understanding the Interface Properties of Magnetic Heterostructures

Yaohua Liu, Suzanne G.E. te Velthuis *Materials Science Division, Argonne National Laboratory, Argonne, Illinois 60439, (USA).* E-mail: yhliu@anl.gov

Engineered interfaces in heterostructures with preferred functionality is frequently tied to a modified behavior at the interfaces and therefore requires detailed knowledge on the chemical, electronic, and magnetic properties as a function of position within the structure. As X-ray reflectometry (XRR) and polarized neutron reflectometry (PNR) provide access to the depth-dependent chemical structure and the magnetization vector, they can provide insights on the relationship between the electronic and magnetic properties and the microstructures, especially at the interfaces. Two examples of our recent work using primarily XRR and PNR will be presented. In the first work, the intrinsic magnetic properties in an Fe/Sm-Co bilayer fabricated under nearly optimal spring-magnet conditions was determined.[1] We found that at the Fe/Sm-Co interface the chemical compositions and magnetic properties change gradually at the length scale of 8 nm via complementary studies of XRR, PNR and micromagnetic simulations. In this intermixed interfacial region, the saturation magnetization and magnetic anisotropy are lower and the exchange stiffness is higher than values estimated from the model based on a mixture of Fe and Sm-Co phases. Therefore, the intermixed interface yields superior exchange coupling between the Fe and Sm-Co layers, but at the cost of average magnetization. In the second work, we investigated LaSrMnO/PrCaMnO superlattices designed as a prototype of magnetically tunable metal–insulator devices by utilizing the competition between the ferromagnetism (FM) and the charge-orbital (CO) ordering [2] . Our XRR studies reveal that the chemical intermixing at the interfaces depends on the deposition order. The preliminary PNR results provide further evidence for the FM/CO phase separation in the real space has been realized with controllable boundaries.

[1] Y. Liu, S.G.E. te Velthuis, J.S. Jiang, Y. Choi, S.D. Bader, A.A. Parizzi, H. Ambaye, V. Lauter, *Phys. Rev. B*, in press **2011**. [2] M. Nakamura1, D. Okuyama, J.S. Lee, T.-h. Arima, Y. Wakabayashi, R. Kumai, M. Kawasaki1, Y. Tokura1, *Advanced Materials*, **2010** *22*, 500.

MS.96.3

Structural and Polarized Neutron Reflectrometry Characterization of $Fe_{16}N_2$ Thin Films with Giant Saturation Magnetization

Jian-Ping Wang,[a] Nian Ji,[a] and Valeria Lauter,[b]   *[a]Departament of Electrical and Computer Engineering, University of Minnesota, Twin Cities, Minnesota, (USA), 55414. [b]Spallation Neutron Source, Oak Ridge National Laboratory, Tennessee, (USA), 37831.* E-mail: jpwang@umn.edu

It is well known that the greatest saturation magnetization value that has been achieved so far is $M_s=2.45T$ with $Fe_{65}Co_{35}$ alloy [1]. Recently Wang's group reported the discovery of the origin of the giant saturation magnetization in $Fe_{16}N_2$[2]. More importantly, we have developed a theory to well explain the ordering effect on the giant Ms [3]. This laid out a foundation for further experiment work. T. K. Kim and M. Takahashi [4] firstly reported a material $Fe_{16}N_2$, with a saturation magnetization about 18% higher than $Fe_{65}Co_{35}$ alloy, which was confirmed 20 years later, by Sugita's group [5]. However this topic has been dropped by the magnetic materials community since 1996 and has been viewed as a controversial topic because there is no theory to support the existence of giant M_s and the traditional of methods to test M_s is volume dependence and typically has a large error bar. More importantly, there is lack of advanced experimental technique to probe the magnetization in a direct and independent approach. Furthermore, Wang's group has developed a repeatable and reliable process to fabricate high quality $Fe_{16}N_2$ thin film samples using a facing target sputtering technique [6].

Here we present a careful X-ray diffraction studies with scattering vector both in-plane and out-plane (A typical out of plane x-ray diffraction pattern on one of these samples is shown in Fig.1) confirms the formation of $Fe_{16}N_2$ phase. A rocking curve measurement reveals mosaic spreading of ~0.5°, which is comparable to monocrystaline films.

By using polarized neutron reflectometry at ORNL, for the first time, we directly confirmed the exsistence of giant saturation magnetization in our films. These data resolve a 40 years controversy among magnetic researchers regarding whether $Fe_{16}N_2$ possess giant saturation magnetization. PNR results will be reported at the conference in details.
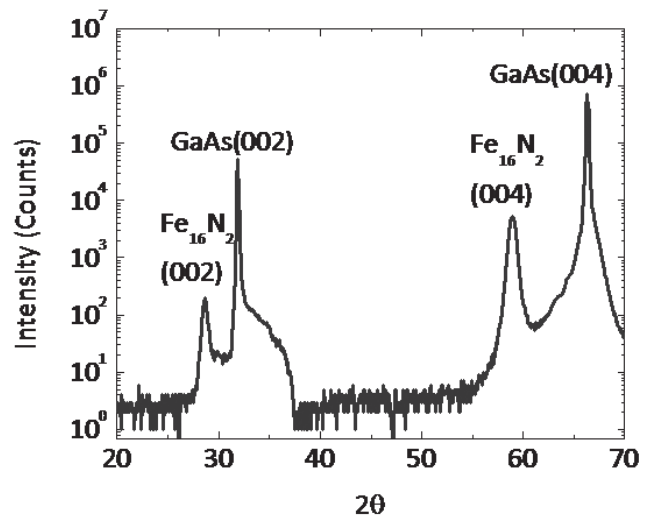

FIG..1 X-ray diffraction on sample with structure GaAs/Fe(2nm)/Fe-N(50nm) (unpublished data). The film peaks can only be indexed to the $Fe_{16}N_2$

[1] R.C. O'Handley, *Modlern Magnetic Materials Principles and Applications* (John Wiley & Sons, Inc., USA, **2000.** [2] N. Ji, X. Q. Liu, J.P. Wang, *New J. Phys.* **2010,** *12*, 063032. [3] N. Ji, F. Allard, E. Lara-Cuzio, J. –P. Wang, *Appl. Phys. Lett.* **2011,** *98*, 092506. [4] T.K. Kim, M. Takahashi, *Appl. Phys. Lett.,* **1972,** *20,* 492. [5] M. Komuro, Y. Kozono, M. Hanazono, Y. Sugita, *J. Appl. Phys.* **1990,** *67,* 5126. [6] N. Ji, Y. M. Wu. J. –P. Wang, *J. Appl. Phys.* **2011,** *109*, 07B767.

MS