a diffraction scan has been performed dividing the blade into three different sections: the edge, the core and the ridge, thus determining the inner phase distribution and confirming the highly differentiate specialization of the single parts of this kind of swords. The shape of the ferrite peak has also been studied in order to semiquantitatively determine the texture level, the strain level and the domain size of the grains to gain knowledge about the several forging methods used by the different schools and traditions.

An energy resolved neutron imaging study has also been performed using the ICON beamline at the spallation neutron source SINQ in Switzerland [3]. A sword fragment has been analysed in order to map the ferrite density by exploiting the enhancement of contrast induced by the selection of two different neutron wavelengths to be used for imaging, thanks to the Bragg edges [4]. We have performed two tomographic reconstruction using two different neutron wavelengths at values immediately before and after the 110 ferrite Bragg edge. The two tomographic reconstructions have been combined together in order to maximize the ferrite phase contrast compared with all the others and evidencing into the tomography the distribution of the ferrite phase inside the sample.

[1] K. Nagayama, *The Connoisseur's Book of Japanese Swords*, Kodansha International Press **1997**. [2] F. Grazzi, M. Celli, S. Siano, M. Zoppi, *Nuovo Cimento C* **2007,** *30*, 59. [3] E. Lehmann, L. Josic, G. Frei, *Neutron News* **2009,** *20*, 20. [4] L. Josic, A. Steuwer, E. Lehmann, *Appl. Phys. A* **2010,** *99*, 515.

Keywords: japanese swords, neutron diffraction, bragg edge neutron imaging

MS.46.4

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Estimation of iron valencies of Prussian blue pigment by anomalous X-ray diffraction

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Prussian blue (PB, iron(III)hexacyanoferrate(II) [1]) is a pigment that has been widely used in Europe in the 18th and 19th centuries. Exposed to light or to anoxic treatments, some PB-containing artifacts discolor due to a photoreduction of iron(III) into iron(II). Although several experiments on light induced degradation of PB have been done in the past, the oxidoreduction process related to the fading and particularly the role of the substrate remains poorly understood.

Anomalous diffraction experiments at the Fe K-edge have been performed on different synthesized PB powders and PB laid on paper. Following the previous literature [2-3], the Fe(III) / Fe(II) ratio could be quantified and related to the state of discoloration of the pigment. In addition to XANES spectra and X-ray diffraction data, the present study aims at better understanding the chemical and structural variations observed on faded PB artefacts.

[1] H.J. Buser, D. Schwarzenbach, et al. *Inorganic Chemistry* **1977,** *16*, 2704-2710. [2] J. Lorimier, F. Bernard, J.-C. Niepce, N. Guigue-Millot, O. Isnard, J.-F. Bérar, *Journal of Applied Crystallography* **2003,** *36*, 301-307. [3] F. Ferreira, P.R. Bueno, G.O. Setti, D. Giménez-Romero, J.J. Garcia-Jareño, F. Vicente, *Applied Physics Letters* **2008,** *92*, 264103.

Keywords: anomalous diffraction, iron, prussian blue

MS.46.5

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XRPD studies of the objects of cultural heritage made of copper or its alloys

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Bronze disease is a dangerous phenomenon which can totally destroy an archeological or art object in a short time. Archaeological metallic objects are susceptible to quick corrosion reactions when excavated and exposed to environmental conditions. Archaeological artifacts can also be contaminated (e.g. chloride contamination) with salts from the burial environment. Bronze disease happens when an artifact containing copper is exposed to humidity and acidity - a condition in which cuprous chloride can be formed. When it is exposed to water, hydrochloric acid forms along with cuprous oxide according to the reaction:

$CuCl_2 + H_2O = 2\ HCl + CuO$

HCl in oxidizing atmosphere reacts with metallic copper giving CuCl, which in contact with moisture, oxygen from air or cuprous oxide turns into $CuCl_2$, which subsequently reacts with water producing dangerous HCl. The information provided above indicates that a systematic description of individual copper phases in artworks and explanation of their origin and transformations are very desirable and the knowledge concerning the solution of these problems in the best museums will be valuable for our research.

In the presented study we have investigated a painting executed on a copper plate which was partially corroded. The samples were taken from both sides of the painting. The identified compounds are presented in the table below:

Name of identified compound	Chemical Formula	PDF number
cuprite	Cu_2O	04-006-6514
tenorite	CuO	00-003-0884
cerussite	$Pb(CO_3)$	04-002-0438
hydrocerussite	$2PbCO_3Pb(OH)_2$	00-001-0687
gypsum	$CaSO_4{\cdot}2H_2O$	00-036-0432
brochantite	$CuSO_4{\cdot}3Cu(OH)_2$	00-013-0398
Quartz	SiO_2	01-085-0865
copper	Cu	00-004-0836

As it is shown above we have not detected the signs of bronze disease. Details of this study as well as the usefulness of powder diffraction technique in the investigations of the deterioration processes of metal objects will be presented.

Keywords: corrosion, XRPD technique, cultural heritage

MS.47.1

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Structure and elasticity of single-crystals by phonon imaging at high pressure

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The wavefront of vibrational energy emanating from a point disturbance within the crystal is far from spherical, as classically observed in fluids. Although the mathematics of elastic-wave propagation in an anisotropic medium has been known for decades, experimental observation of a vibrational wavefront in a small (micro- or nano-sized) crystal has only been here achieved.

Based on the weird behaviour of acoustic phonons in anisotropic media like crystals, we here present the first development of phonon imaging at high pressure by means of picoseconds acoustics in diamond anvil cell, an advanced and unconventional technique to probe (with a pulsed laser) the structure and elastic behaviour of thin solids under extreme conditions.

Our method gives snapshots that portray the acoustic ray which provides an immediate indication on the complete elastic properties of thin compounds and its evolution under extreme mechanical condition.

The example of single-crystalline silicon up to 10 GPa is presented as a case study [1].

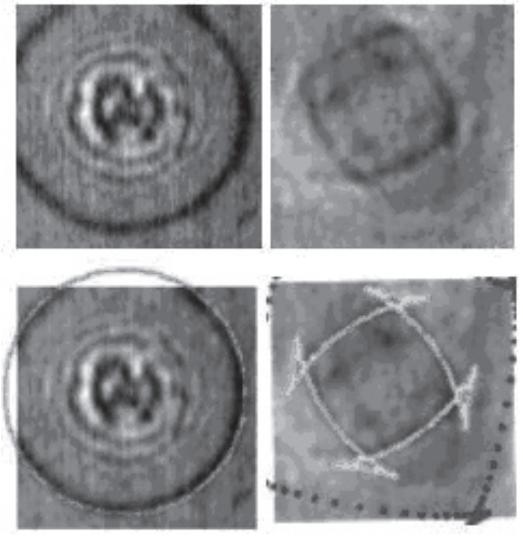

Figure : Top : experimental phonon imaging patterns in the (100) plane of silicon at 7.75 GPa at two different pump-probe delays. Bottom : same as top with superimposed calculation curves for longitudinal, fast and slow transversal group velocities (red, blue and green dashed lines respectively) using C11= 196.9 GPa, C_{12}= 104 GPa and C_{44}= 80 GPa.

[1] F. Decremps, L. Belliard, M. Gauthier, B. Perrin, *Phys. Rev. B* **2010,** *82,* 104119.

Keywords: phonon, pressure, elasticity

MS.47.2

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High-pressure neutron diffraction at the SNS

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To date, the role of neutron diffraction in high pressure research has been relatively peripheral, with the vast majority of structural studies using synchrotron-based x-ray diffraction. This is despite well known (and not so well known) advantages of neutrons for crystallography, including: sensitivity to positions of light atoms (in particular H^2, Be, B^{11}, C, N and O), exceptional real-space resolution and the ability to measure long-range magnetic order.

The principle reason for the lack of neutron science under extreme pressure conditions is the intrinsic weakness of available sources and the corresponding need for large sample sizes. To put this in context, current typical sample volumes for neutron powder diffraction are of order 25mm^3 compared with 0.0025mm^3 for diamond-anvil cell synchrotron diffraction. The realisation of such small volumes for neutron diffraction would greatly expand the capabilities of this unique probe.

Recently, the neutron landscape changed with the opening of the 'next generation' 1.4 MW Spallation Neutron Source (SNS) at Oak Ridge National Laboratory. Since then, a collaboration between the Geophysical Laboratory (GL) and the SNS has sought to exploit the SNS's unrivalled, pulsed flux of neutrons for high-pressure crystallography. A key component has been the incorporation of many aspects of GL's experience in high-pressure synchrotron beamlines onto the high-pressure neutron diffractometer, SNAP. This has been coupled with a new generation of diamond anvil cell (DAC) technology tailored to optimize sample volume, and minimise background scattering.

We will present the most recent achievements of this collaboration: diffraction measurements from powder samples, at pressure, inside DAC's with volumes below 0.1mm^3. We will also show the latest attempts to exploit these small volumes to achieve unprecedented pressures for neutron crystallography.

Keywords: neutron, pressure, diffraction

MS.47.3

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Towards the use of Laue microdiffraction intensities for structural studies at extreme conditions

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Single crystal X-ray diffraction is a most powerful technique to decipher the atomic structure of crystalline material. In its most mature and most precise flavor, monochromatic single crystal X-ray diffraction, the reciprocal lattice of a mounted crystal is rotated through a single Ewald sphere, representing one narrow band pass energy. The drawbacks of monochromatic single crystal X-ray diffraction are the need to rotate the sample through the Ewald sphere and - linked to this - the relatively time consuming data acquisition. The need for a mobile sample makes this technique ill suited for in situ studies such as e.g. laser heated diamond anvil experiments. One possible alternative for such situations is Laue diffraction. By using a broad band-pass X-ray source such as provided for example by a Synchrotron bending magnet, a large number of reciprocal lattice points can be simultaneously imaged in a single exposure.

The potential of Laue diffraction for time resolved in situ experiments has been recognized and exploited by the protein community. In their approach, the Laue specific problems were addressed by the large data redundancy stemming from the large unit cells of typical protein crystals. This approach is not applicable to inorganic substances with relatively small unit cells. We devise ways to extract the integrated intensities from a Laue pattern in cases where high reflection redundancy cannot be achieved. In order to properly interpret intensities, various specific issues such as energy dependent correction factors (absorption, Lorentz