samples which are important parameters to gain knowledge about the technological level of the Japanese weaponsmiths. A comparison with the typical characteristics of Japanese swords and helmets is also presented, showing that the quality of the metal composing the arrows is intermediate between the one used for swords (very high quality) and the one used for armour pieces (low quality).

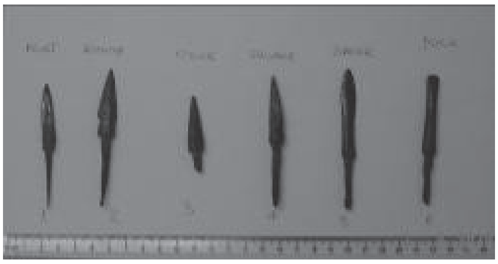

Fig.1 Picture of the six arrows measured through neutron diffraction.

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Keywords: japanese arrows, neutron diffraction, quantitative analysis.

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Importance of X-Ray Analyses in Slag Studies at Clintonville, New York, USA

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X-ray diffraction and x-ray fluorescence analyses of historical iron slags from Clintonville, NY, USA help describe the slags and provide insight to the uniformity and efficiency of the techniques used at the Clintonville Forge in the 1840s. Over 70 samples of slag, byproducts of the smelting process, were collected then divided based on macroscopic morphological characteristics. Samples were then analyzed with powder x-ray diffraction (XRD) in order to identify the mineral constituents within the slag. The wide range of morphologies does not govern mineralogical assemblages in Clintonville samples, as originally hypothesized. Modal abundances do, however, vary for some morphological groups. Clintonville slag commonly contains fayalite and magnetite, with minor occurrences of wüstite, forsterite, and glass. Although these phases are common in iron slag, the XRD is quintessential for definitive identification. This is especially important when these phases make up the fine-grained ground mass. Analyses using x-ray fluorescence spectroscopy (XRF) indicate chemical homogeneity for major oxides and trace elements throughout the morphological groups of slag. Consistent mineralogies and chemistries amongst slags reflect uniformity in the smelting processes at the forge. X-ray data further indicate that the Clintonville process was not efficient. Pure iron prills are nearly ubiquitous in the slags. Ore and cinders from this site have a total iron oxide content (FeO_t) ranging from 80-88 weight percent. Clintonville slag samples contained between 56 and 68 weight percent FeO_t. The smelting processes utilized at Clintonville consolidated only 12-20% of the iron into the final product.

Keywords: slag, fayalite, archaeometry

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The fluorescence life time of ruby at high pressures and temperatures

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The pressure determination in diamond anvil cell (DAC) experiments at high pressure and temperature (high-p,T) based on the ruby fluorescence method [1] is not straightforward, as both, pressure and temperature, have a significant influence on the frequency of the ruby fluorescence [2]. Here we sugest an alterative approach based on fluorescence life time measurements.

Fluorescence life time (FLT) thermometry is an established method at ambient pressure, where the ruby FLT decreases by three orders of magnitude between 300 and 900 K [3]. Up to now, only few reports concerning the pressure dependence of the FLT have been published. At 295 K the ruby FLT linearly increases between ambient pressure and 40 GPa by one order of magnitude [4].

Here we present the first study of the FLT of ruby at high-p,T. The FLT was measured in an externally and laser heated DAC. The fluorescence was excited by a ns pulse of a Nd:YAG laser at 532 nm. At constant temperatures the FLT increases linearly with pressure in the pressure range studied (< 30 GPa). At constant pressure the FLT decreases linearly with temperature. Our experiments show that FTL measurements may provide a reliable method for the determination of temperatures in a DAC at high pressure. A significant advantage will be, that FLT measurements on doped samples will unambiguously give bulk temperatures.

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Keywords: thermometry, fluorescence life time, diamond anvil cell

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Energy-domain SR ^{57}Fe-Mössbauer spectrometer for high-pressure mineral physics

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Iron is the most abundant element in the Earth. The multiple electronic configurations of iron (valence and spin state) give rise to complex physical and chemical properties of the Earth's mantle, resulting in the influence on the structure, dynamics, and evolution of the Earth's interior. The spin-state crossover and the valence state in $(Mg,Fe)(Si,Al)O_3$ silicate perovskite and post-perovskite, the most abundant mineral (~75 wt.%) in the lower mantle and the lowermost mantle, respectively, are essential for modeling the Earth's lower mantle. The degree to which the spin and valence states of iron in the lower mantle silicates influence the density and the elastic and seismic properties remain an open question.

Here we develop an energy-domain synchrotron radiation (SR) ^{57}Fe-Mössbauer spectrometer using the single-line Mossbauer source at beamline BL10XU of SPring-8 for enabling simultaneous X-ray diffraction (XRD) and Mössbauer spectroscopy (MS) experiments at high pressures and high/low temperatures in a diamond-anvil cell technique. The diffractometer for the energy-domain SR-MS consists of a high-resolution monochromator and a variable-frequency nuclear monochromator. The single-line Mössbauer sources with an energy width of neV can be extracted from SR using pure nuclear Bragg scattering. For MS measurements on iron-bearing materials, the incident X-rays are tuned to 14.4 keV. For XRD the energy of the incident X-ray beam from undulator source plan to be tuned the high-order harmonic of 14.4 keV. Many geological-relevant materials have no large hyperfine interactions, and exhibit the splitting collapses at high pressure. The energy-domain SR-MS method works in any operation mode for the storage ring. Analysis of MS spectra in energy domain is well established and is easier than that in time domain. Therefore, the energy-domain MS using SR would be suitable for high pressure studies of the magnetic and electronic properties of deep Earth materials, compared with nuclear resonance forward scattering of SR that is a time analogue of MS.

The combined system of XRD and MS can potentially offer the essential information for resolving outstanding issues in mineral physics. The MS method can be used with glass and fluid phase as well as crystalline materials. At present, we are at the stage where the device is started up. The device should be fully available in the second stage of 2011. In this talk, we describe this new facility and report on progress.

Keywords: mössbauer spectroscopy, nuclear bragg scattering, high pressure

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Setting up a home high pressure laboratory from scratch – when is it worth it?

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Although high pressure crystallography continues to expand in both scope and popularity [1], the specialized equipment and skills required to pursue such studies in an effective manner mean that it is still regarded as largely the preserve of large, well-established and highly-experienced groups. However, our experience of high pressure studies conducted at central facilities [2], where extensive expertise and support may be available, led us (and other researchers) to consider establishing high pressure facilities within our home laboratories. While these will obviously be modest in comparison to powerful central facilities, they offer the advantage of much greater access, as well as more flexibility and convenience to carry out high pressure experiments.

We have recently set up dedicated in-house high pressure facilities in Nottingham, within a crystallographic laboratory where no such facilities had previously been available [3], and we will describe our experience and the factors which had to be considered during the process. These factors include the nature and extent of the scientific program to be undertaken, grant support, other funding, accommodation and expert advice and support: their influence and interaction must be taken into account when deciding whether in-house high-pressure facilities are viable and, if they are, in designing, commissioning and installing them.

This contribution will also explore some of the different options available, taking into account local requirements and circumstances.

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Keywords: high pressure, instrumentation, techniques

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High-resolution single-crystal neutron diffraction to 10 GPa

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Single-crystal neutron diffraction is the technique of choice when trying to determine the nature of complex multi-site disorder and anisotropic atomic displacement parameters (ADPs). The absence of form factor effects in the neutron scattering process gives inherent advantages for measurement of disorder and ADPs and the access to three dimensional data provided by a single crystal gives greatly enhanced accuracy on directional dependence.

Now, with the development of new cell, anvil and gasket technologies, a significant breakthrough has been made, allowing us for the first time to take full structure refinement to ~10 GPa on the D9 instrument at ILL, Grenoble, and the Laue TOF instrument (SXD) at the ISIS Facility, UK.

We will describe the results from studies of squaric acid and KDP up to pressures of 10 GPa where we have been studying the proton distributions in nearly centred and centred hydrogen bonds. We will also present results from samples grown in situ at high pressure of phases of ice and ammonia water mixtures. We will describe the technical aspects of performing high-resolution single-crystal studies at high pressure both at neutron spallation and at reactor sources.

Details of analysis techniques will also be described. Finally we will also highlight developments in view to increase the range of both science and pressures that can be achieved.

Keywords: high-pressure, neutron diffraction, single-crystal, hydrogen bond

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Low-temperature high-pressure analysis utilizing a novel pressure cell design

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Inorganic compounds can undergo significant changes in their