A new in-situ cell has been designed and constructed to simulate the temperature, pressure and chemical conditions present in deep saline aquifers. The cell has been built to study reactions at the CO\textsubscript{2}-brine-solid interfaces produced during injection of supercritical CO\textsubscript{2} (sc-CO\textsubscript{2}) for storage and sequestration purposes. Much of the data relating to CO\textsubscript{2} storage relates to oil and gas wells. Little experimental data exists on the behavior of various rocks and engineering materials under these conditions, particularly with injection of impure CO\textsubscript{2} streams, and some of the existing data give significant cause for concern.

The cell has been designed for both reflection and transmission geometries so may be used both in laboratory and synchrotron environments. Although sc-CO\textsubscript{2}/water often reacts slowly under equilibrium, concerns have been raised about impurities such as SO\textsubscript{2} and H\textsubscript{2}S that will inevitably be present in industrial streams. These gases can react rapidly, so the slower laboratory and more rapid synchrotron measurements are highly complementary. Flow-through experiments may also be possible but haven’t been specifically targeted.

The cell has maximum working conditions of 300 bar pressure and 300 °C in pH3 brine conditions. These conditions are extremely demanding for any metals, sealing and window materials, and the restrictions imposed by the ASME Pressure Vessel and Boiler Code \cite{1} and Process Piping Code \cite{2} posed additional challenges. The materials certification requirements in the ASME code forced us into using structural grade beryllium windows as in our first generation cell \cite{3}. Beryllium is highly susceptible to chloride corrosion so a thin coating of tantalum is used to protect the windows under these conditions. Requirements of the pressure vessel certification process meant the cell was successfully tested hydrostatically to 450 bar pressure before delivery.

Some of the theoretical and practical considerations for the design and construction of the cell will be described. Although the cell may be used with a laboratory diffractometer, the silver X-ray tube source of tantalum is used to protect the windows under these conditions. The ternary intermetallic system R\textsubscript{2}T\textsubscript{4}X\textsubscript{12} (where R = rare earth; T= Mn, Cu, Ag, Au, Pd; X=Si, Ge, Sn) comprises an extensive series of isostructural compounds that crystallize in the orthorhombic Gd\textsubscript{3}Cu\textsubscript{4}Ge\textsubscript{2} -type structure (space group Imm\textsubscript{m}, \#71) \cite{1}. The exceptions to this generic orthorhombic crystalllography are Tm\textsubscript{3}Cu\textsubscript{4}Sn\textsubscript{4} and Lu\textsubscript{3}Cu\textsubscript{4}Sn\textsubscript{4} which have a monoclinic structure (space group C12/m1, \#121) at room temperature \cite{2}.

In the orthorhombic phase the R atoms occupy two crystallographically distinct sites \cite{3} and these compounds have attracted interest due to the general observation of independent magnetic ordering of these two R sublattices \cite{1}.

The present work reveals that a structural phase transition occurs in Er\textsubscript{3}Cu\textsubscript{4}Sn\textsubscript{4} and Ho\textsubscript{3}Cu\textsubscript{4}Sn\textsubscript{4} from orthorhombic Imm\textsubscript{m} to monoclinic C12/m1 upon cooling, at 262(2) K and 62(2) K respectively. We also found a structural phase transition at 458(2) K from monoclinic C12/m1 to orthorhombic Imm\textsubscript{m} in Tm\textsubscript{3}Cu\textsubscript{4}Sn\textsubscript{4} upon heating.

The results show a linear behaviour of the crystallographic transition temperatures with the ionic radii of the R\textsuperscript{3+} ions, and predict no transition for the Dy\textsubscript{3}Cu\textsubscript{4}Sn\textsubscript{4} compound, in accord with our measurements carried out at low temperatures for this compound.

Here, we present complete structural information from high resolution synchrotron X-ray powder diffraction studies of these R\textsubscript{3}Cu\textsubscript{4}Sn\textsubscript{4} compounds.

\textit{Keywords: \textit{ intermetallics, structural, transition}}

\section*{MS05.P25

Behaviour of actinide pyrophosphates $\text{AnP}_2\text{O}_7$ at high temperatures: $\text{An=Th,U,Np,Pu}$


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Actinide-bearing phosphate compounds can be encountered in mineralogy \cite{1}, biochemistry \cite{2} or issued from anthropogenic activities like nuclear fuel reprocessing. They have also been envisioned and investigated as potential host for actinide waste disposal. Because of their numerous oxidation states (e.g. III to VII for neptunium) and corresponding ionic radii, actinides can form various types of phosphate compounds \cite{3}, \cite{4}. In the present studies we focussed on the pyrophosphates family of general formula $\text{AnP}_2\text{O}_7$, in which actinides are tetravalent. Pyrophosphates have attracted a lot of interest over the last decade \cite{5} because the exhibit unusual high temperature properties such as negative thermal expansion above a certain temperature. In the present work we have investigated the structural behaviour of four

\section*{MS05.P24

A structural phase transition in the $\text{R}_2\text{Cu}_3\text{Sn}_4$ compounds ($\text{R=Ho, Er and Tm}$)

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The ternary intermetallic system $\text{R}_2\text{T}_4\text{X}_12$ (where $\text{R}$ = rare earth; $\text{T}$= Mn, Cu, Ag, Au, Pd; $\text{X}$=Si, Ge, Sn) comprises an extensive series of isostructural compounds that crystallize in the orthorhombic $\text{Gd}_3\text{Cu}_4\text{Ge}_2$ -type structure (space group Imm\textsubscript{m}, \#71) \cite{1}. The exceptions to this generic orthorhombic crystalllography are $\text{Tm}_3\text{Cu}_4\text{Sn}_4$ and $\text{Lu}_3\text{Cu}_4\text{Sn}_4$ which have a monoclinic structure (space group C12/m1, \#121) at room temperature \cite{2}.

In the orthorhombic phase the R atoms occupy two crystallographically distinct sites \cite{3} and these compounds have attracted interest due to the general observation of independent magnetic ordering of these two R sublattices \cite{1}.

The present work reveals that a structural phase transition occurs in $\text{Er}_3\text{Cu}_4\text{Sn}_4$ and $\text{Ho}_3\text{Cu}_4\text{Sn}_4$ from orthorhombic Imm\textsubscript{m} to monoclinic C12/m1 upon cooling, at 262(2) K and 62(2) K respectively. We also found a structural phase transition at 458(2) K from monoclinic C12/m1 to orthorhombic Imm\textsubscript{m} in $\text{Tm}_3\text{Cu}_4\text{Sn}_4$ upon heating.

The results show a linear behaviour of the crystallographic transition temperatures with the ionic radii of the R\textsuperscript{3+} ions, and predict no transition for the $\text{Dy}_3\text{Cu}_4\text{Sn}_4$ compound, in accord with our measurements carried out at low temperatures for this compound.

Here, we present complete structural information from high resolution synchrotron X-ray powder diffraction studies of these $\text{R}_2\text{Cu}_3\text{Sn}_4$ compounds.

\textit{Keywords: \textit{powder diffraction, gas, pressure}}
 actinide-bearing pyrophosphates and found that all of them exhibit this unusual high temperature behaviour above 673K. Besides we also found that their room temperature structure is not cubic as previously reported [6] but triclinic (distorted cubic) and transform into a cubic form around 150°C. The phase transition has been characterized by various experimental techniques, X-ray diffraction, solid-state NMR and Raman spectroscopy. The room temperature structure has been determined by from synchrotron radiation data obtained at the ESRF. Our presentation will report the experimental results and discussed the structural aspect related with the contraction of the materials at high temperatures.


Keywords: actinides, phosphates

Crystal Structure and phase transitions of mechanoactivated manganese oxides
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One of the features of the Mn-O system is the formation therein of MnO₂, MnO₂, MnO₂, and MnO₁ below high-energy ball milling (g=60). The as-prepared mechanically activated manganese oxides were characterized as single-phased, nanoscaled or ultra-fine-grained compounds. Ball milling gave rise to a direct phase transition from amorphous manganese oxides to MnO₂ during heating. This result was obtained both for the as-prepared nanoscaled MnO₂ oxide and for the coarse-grained powder after multiple thermal recycling. The same effects were observed upon thermal study of the other activated manganese oxides. A significant increasing in the temperature of the phase transition from MnO₂ to MnO₂ during heating and an easy reversibility of the MnO₂ to MnO₂ phase transition upon cooling were also detected. It was proposed structural defects due to the mechanical activation are responsible for the anomaly in phase transition behaviour of mehanoactivated manganese oxides. Therefore the structure and microstructure of the phases involved were refined depending on treatment conditions and temperature.

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Keywords: oxide, temperature, transition

Negative thermal expansion in trioxane silica sodalite (TRSS)
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The thermal dehydration and template burning process of a silica sodalite (SS), synthesized in the presence of boric acid and trioxane (TR), Na₈[BO₃Si₄O₁₂]1.8TR, were studied in situ by synchrotron powder diffraction up to 900°C. Time-resolved diffraction data were collected at the GILDA beamline at ESRF (Grenoble). Thermal analyses (TG, DTA and DTG) of the as-synthesized samples were carried out in the same temperature range under a constant flux of air (heating rate 5°C/min).

The cell volume of TRSS does not change remarkably until the cell volume of TRSS does not change remarkably until the

Keywords: neutron scattering, materials, in situ

Hybrid instrument for in situ studies at the ESS
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The decision to build the European Spallation Source (ESS) came in the spring of 2009. The ESS will be build in Lund, Sweden and become the most powerful neutron source in the world. The ESS will give unprecedented possibilities for neutron scattering in crystallography and materials science. Investigations of materials at multiple length scales are getting increasingly important for understanding and improving materials performance such as hydrogen storage, solid oxide fuel cells and lithium ion batteries.

A new concept for a neutron scattering instrument will be presented. The new idea combines powder neutron diffraction (PND), small angle neutron scattering (SANS), and neutron imaging (NI) in the same instrument. The PND potentially allows extraction of pair distribution functions for looking at amorphous systems. By combining these techniques it will be possible to look at length scales 0.01-5 nm using diffraction, 1-1000 nm using SANS, and 0.01-100 mm using NI. In addition to the different techniques a high time resolution is envisioned to study processes at the subsecond level. In this way it is possible to study materials in situ or in operando.

Through the presentation it is intended to start discussions with the potential user community of the suggested hybrid instrument. The ESS is soon to determine the instrument suite which will be built in the first round. Since this instrument presents new concept there is no existing user community. The IUCr is therefore a great opportunity for engaging with the potential users.

Keywords: neutron scattering, materials, in situ