ESRF	**Experiment title**: Structure and dissolution of sol-gel derived bioactive glass scaffolds for tissue engineering applications	**Experiment number**: SC-1584
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Report:

Overview

All present day orthopaedic implants lack some critical characteristics of living tissues, and current research is shifting emphasis from replacement of tissues to regeneration. Inorganic scaffolds will be used that guide tissue growth and dissolve at a rate to match tissue regeneration leaving little trace of a defect, i.e. promoting tissue engineering. The basis of this approach is a synthetic scaffold which should reabsorb into the body as non-toxic degradation products at the same rate that the cells produce their own extracellular matrix. Bioactive glasses bond to connective tissue such as cartilage and are reabsorbable in the body. Their bone bonding ability has been attributed to the formation of a hydroxycarbonate apatite (HCA) surface layer on contact with body fluid. The composition and structure of the HCA layer is similar to that of bone mineral but its detail is still relatively poorly understood. Sol-gel derived bioactive glasses tend to be more bioactive and reabsorb quicker than melt-derived glasses of similar compositions. The compositions of gel-glasses can also contain fewer components while maintaining bioactivity. This project examined sol-gel calcium silicate materials, focussed on the optimum bioactive composition $(CaO)_{0.3}(SiO_2)_{0.7}$ and aimed to follow the subsequent modification of the structure and the formation of the surface HCA layer of the standard sol-gel sample, over the key timescale (the first few hours).

Experimental method and results obtained

This project built on the methodology developed for time-resolved X-ray diffraction data collected on ID15B through ME-673 of the kinetics and structural evolution of the initial gelation stages of TiO_2-SiO_2 sol-gel samples. Two approaches were considered. First was to have the sol-gel powder dispersed in simulated body fluid (SBF) and then pumped via a peristaltic pump through a cell. However the second strategy developed, and the one adopted, was to use the cell manufactured for ME-673, with a new insert that held a solid block of the foam (Fig. 1). The holder was designed to allow maximum contact between the foam block and the SBF. To stir the SBF the cell was rotated on the beamline at a few Hz. The aim of this particular experiment was to follow the structural evolution on reaction with the SBF. To mimic the conditions in the body the experiment was carried out at 37 ± 1^{o}C. Temperature control was achieved by

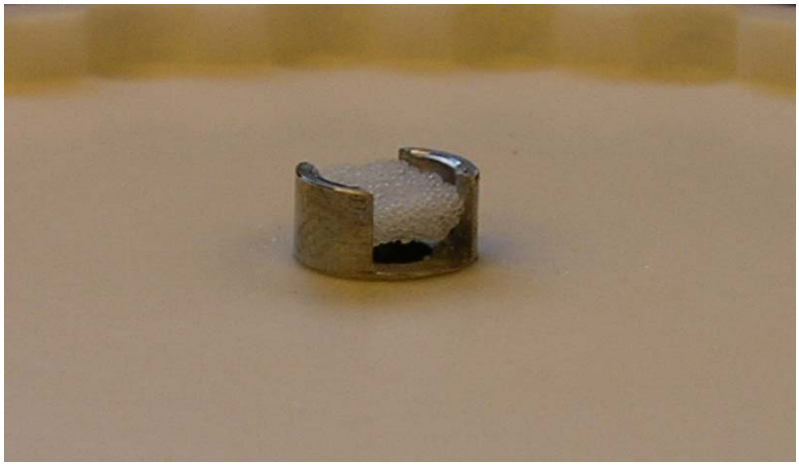

Figure 1. Expanded view of sample holder showing the X-ray entrance hole below and the calcium silicate foam block. (The approximate diameter of the holder is 1cm)

using a halogen lamp at a given distance to produce this temperature. This simple set-up proved to be remarkably effective.

For the $CaO\text{-}SiO_2$ materials in SBF scans were collected for total times at a particular step point of between 10 and 300s, made up of individual scans of 10s. Data collection on individual samples lasted anywhere up to 24hrs. For the $CaO\text{-}SiO_2$ SBF experiment a sample-detector distance of 300mm was used, corresponding to a Q-range of 0.8-23$Å^{-1}$. All samples were based around the most bioactive composition $(CaO)_{0.3}(SiO_2)_{0.7}$, and the porosity and reactivity of this composition depends on the heat treatment so that comparison was made with samples heated to 600, 800 and 1000°C as well as reaction of two different SBF solutions.

Some typical Q-space data is shown in Fig. 2 for reaction of a foam with SBF over a period of ~23hours. The data has been normalised to the beam intensity and has had the background crudely subtracted by subtracting the diffraction pattern of the SBF only with its intensity reduced so that the result would not be negative. Although no detailed analysis has yet been carried out, the direct diffraction data shows that the broad features from amorphous foam are replaced by sharper peaks from crystalline phases that form on reaction. It will require very careful correction for sample absorption, background scattering and density to allow the Fourier transform. The detailed changes in the silicate network as it reacts with the SBF and the identification of new phases forming should be revealed by complete analysis.

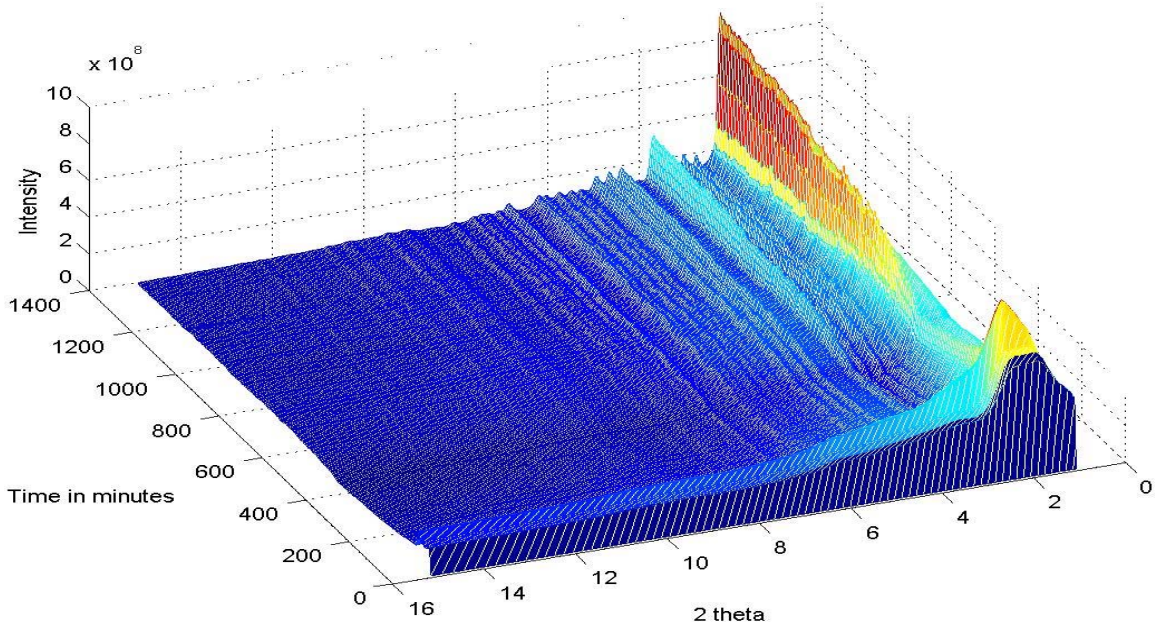

Figure 2. Diffraction data showing the reaction of a $(CaO)_{0.3}(SiO_2)_{0.7}$ with simulated body fluid at 37°C