Preliminary Results for the Diffusion of Water into Fibre Optics.

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ABSTRACT

Preliminary results are presented on a measurement technique for determination of water penetration into fibre optic sensors under high pressure, high temperature conditions. Both fibre optic sensors and communication fibres were subjected to prolonged treatment in heavy water at temperatures up to 250C and pressures of 40bar. Deuterium penetration is measured by a nuclear reaction technique based on an ^{3}He micro-focussed ion beam, which permits two dimensional mapping of the deuterium penetration into cleaved fibres. Water penetration can be detected whilst still confined to the cladding, well before any optical effects become apparent, permitting prediction of likely fibre lifetime under down-hole conditions after realistic experimental times.

1. Introduction

'Down Hole' sensing of oil well conditions, in particular pressure, would be of great value in reservoir management and economic optimisation of production schedules. Oil reservoirs are

frequently complex structures of variable permeability, crossed by faults, and with underlying water layers. The much higher viscosity of the crude oil compared to the water leads to instabilities in the water/oil interface. Excessive production rates can lead to a loss of recoverable oil or the need to employ costly secondary recovery techniques such as gas or water injection prematurely. A knowledge of the local down-hole conditions, as opposed to aggregate wellhead flow and pressure recovery curves on flow cut off, would greatly improve planning of production schedules.

The requirements on such sensors are stringent in the extreme. They need to be located up to 10km from the wellhead down a well of only some tens of centimetres in diameter. Lifetimes of ten years are desirable, ideally with no electrical connections for safety reasons. The sensors must survive temperatures routinely exceeding 200C and occasionally reaching 300C, with pressures up to ~600 bar and retain stable calibration over their lifetime. Existing sensors, typically based on crystal quartz transducers, are difficult to deploy, require electrical connections, and have very limited life. Fibre optic sensors that are sufficiently thin and flexible can be deployed directly into the well by entraining them in hydraulic oil pumped down small bore tubes which are utilized for hydraulic operation of down-hole systems [1,2].

This has lead to a strong interest in fibre optic sensors for this application, which satisfy the requirements of down-hole deployability and no electrical connections [3-5]. These sensors typically use fibres with internal longitudinal holes in the cladding; the resulting asymmetry results in external pressure causing birefringence in the fibre core, which is measured optically [6]. However, to retain their essential small diameter and flexibility, characteristics which enable their deployment, only thin protective coatings can be employed. The lifetime of the sensor and the communication fibre linking it to the surface in the aggressive down-hole

environment becomes a serious issue, with a requirement to determine whether water penetration of the coating and diffusion into the fibre will cause unacceptable increases in fibre loss at the measurement wavelengths (typically 1.55 or 1.3μm) or drift in the pressure sensor.

In 1997, Jenneson et al [7] described a technique which used an ion beam to monitor the uptake of a deuterated surfactant by undamaged and damaged hair fibres. They used a Nuclear Reaction Technique (NRA) which enabled a hair fibre previously subjected to the deuterated surfactant to be monitored when the cross-section was scanned by a focussed ^{3}He microbeam. The following reaction was utilised:

$$^3\mathrm{He} + \mathrm{D} \rightarrow {}^4\mathrm{He} + \mathrm{p}, \qquad Q = 18.35\mathrm{MeV}$$

where Q is the energy released during the reaction. The protons with energy about 12.8MeV were detected. Two-dimensional maps were plotted showing the concentration of the deuterated surfactant in the fibre.

The above technique has now been applied to optical fibres pre-treated in deuterated water under conditions of high temperature and pressure. It is ideally suited to measuring diffusion into cylindrical solids at distances of tens of microns or greater. The only known alternative ion beam technique for measuring water diffusion into glass is the ^{1}H(^{15}N, αγ) reaction. This is a narrow resonance energy loss technique which excels in the study of water diffusion at distances up to a few microns through a planar surface on large area samples, but has not been applied to 2D scans needed in our application [8, 9].

A scanning ^{3}He micro-beam was utilised to enable two-dimensional maps of the sample cross-section to be produced. The NRA technique was employed to measure the penetration of the deuterated water into fibre optics and PIXE was used to see the structure of the fibre optic. The

inter-sample normalisation of the deuterium peak was made using the Rutherford Backscattering signal (RBS) from the copper blocks which clamp the fibre in position [10]. These techniques were all measured simultaneously making this a multi-analysis procedure [10].

2. Experimental Details

Simulation of down-hole pressure and temperature conditions was achieved by heating closed cells in a temperature-controlled oven. Initial experiments showed that this required considerable attention to cell design and experimental procedures.

Cells were initially constructed with bursting disks and a PEEK seat valve (rated to 232C by the manufacturer) to enable safe relief of any over pressure during heating or gas generated. The PEEK seat exposed to the superheated water was found to degrade, and deposit organic contamination on samples at 200C after ~30 days. However no gas generation or physical distortion of the cells occurred. Future cells were therefore constructed without relief or vent valves, with quarter-inch 'Hoke Swagelock' 316 stainless steel fittings and stop ends. During the heating cycle the cells were treated as potentially explosive, and the furnaces only opened after cooling to room temperature. They were opened with caution, but no gas release was ever noted.

The initial experiments showed that fibre corrosion by the superheated water was extremely variable under nominally identical conditions, and in a few cases the entire fibre dissolved! This appears to be due to trace contamination of the water (increased pH is well known to enhance water attack on silica under more moderate conditions.) Chemical attack upon fibres as a function of conditions will be the subject of a separate study. Stringent anti-contamination

procedures were followed during cell preparation. The cell itself is made from electropolished seamless 316 stainless steel tubing, and pre-cleaned with electronic grade solvents. It was then baked disassembled in air at 150C for a few hours, followed by baking sealed over-night with a few cubic centimetres of heavy water at 200C to both clean and pre-deuterate the cell. This heavy water fill was discarded, before loading the cell with samples. The fibre ends are flame sealed, and short lengths of fibre placed in a short 3mm bore silica 'test tube' so that most of the fibre sample protrudes, before placing the 'test tube' in the stainless steel cell, adding heavy water and sealing. The silica tube makes fibre sample loading and retrieval much easier, and prevents the samples adhering to the stainless tube wall by surface tension. The heavy water (>99.8 atom % D, Fluka) fill quantity was calculated to ensure that a vapour space remained in the cell at operating temperature, but that samples were fully immersed; the cells remained vertical after loading and in the oven. Cells were weighed accurately before and after heating to check for leakage, which occurred on a significant number of occasions under these arduous conditions, despite rigorous attention to assembly procedures and use within the fitting's ratings. Cells which leaked were not used for measurements, owing to the resulting uncertainty in exposure times, although there is limited evidence that leakage most often occurs immediately at the start of a run, or right at the end, presumably due to thermal expansion movements.

Two different types of optics were investigated. Pressure sensor fibres were 125μm in diameter whilst communication fibres were 100μm, both with pure silica cladding and germania doped cores produced by VAD (vapour phase axial deposition) with a numerical aperture of 0.125 and core diameters of approximately 7μm. Those used for sensing pressure contain two circular 23μm holes placed symmetrically on a common diameter. The fibre optics were immersed in deuterated water at 210C temperature for just under fifty two days, (1247 hours at temperature.)

The saturated vapour pressure at 210C is 19 bar. They were then hand cleaved with a new alumina knife to obtain a flat cut at right angles to the shaft of the fibre. Some qualitative difference was noted in the cleaving properties of exposed fibres as compared to fresh samples; the fibre appeared more brittle, and obtaining a clean, flat cleave was more difficult. The samples were mounted in cross-section between two copper blocks on an aluminium target plate. The plate was then placed inside an evacuated chamber and connected to a liquid nitrogen cooled cold finger; the samples were cooled to prevent water egress. Control fibres which had not been in contact with the heavy water were also cleaved and analysed.

For all the analyses (NRA, PIXE, and RBS) the samples were irradiated with a 1.9MeV ^{3}He beam of 1nA using the Van de Graaff accelerator based at the University of Surrey. The beam spot was 10μm in diameter and was raster scanned across the fibre sample. The x-rays were detected using a Si(Li) detector placed at 135° to the incident beam, and scattered ^{3}He ions and reaction protons (RBS and NRA) were measured using a surface barrier detector at an angle of 165°. The PIXE, NRA and RBS data were collected simultaneously for 20 minutes.

3. Results

The map collected for the silicon in the fibre with diameter 125μm, which was immersed in the deuterated water, is shown in Figure 1. Also the corresponding map for the deuterium penetration of the optic is shown (Figure 2). Figures 3 and 4 shows the corresponding maps for the control fibre. The scan size covered an area of 250μm by 250μm. The grey scale given indicates the high and low concentrations of either the silicon or the deuterium content. The scale is divided into ten sections calculated by dividing the difference of the highest and lowest concentration by ten.

Figures 1 and 3 show the silicon content in the fibre optics and indicate their respective structures. These figures show that there has been no change to the structure of the optic after being treated. Consequently this indicates that the treatment does not damage the fibre which could allow water penetration. From Figure 2 it can be seen that the water penetrates the optic by about 20✪m, and Figure 4 shows that there is no water uptake in the control fibre optic. Therefore all the deuterium in the treated fibre is due to the water penetrating the fibre.

Figures 5 and 6 show the silicon and deuterium maps for the 100μm fibre optic after treatment respectively. Figures 7 and 8 show the silicon and deuterium maps for the control fibre. This fibre contains no side holes, and is used to communicate between the wellhead measurement system and the down-hole sensor. From the deuterium map for the treated sample (Figure 6) it can be seen that the deuterium penetrates throughout the whole fibre. This shows that the design of this particular type of fibre (100✪m) is not resistant to high temperature and pressure.

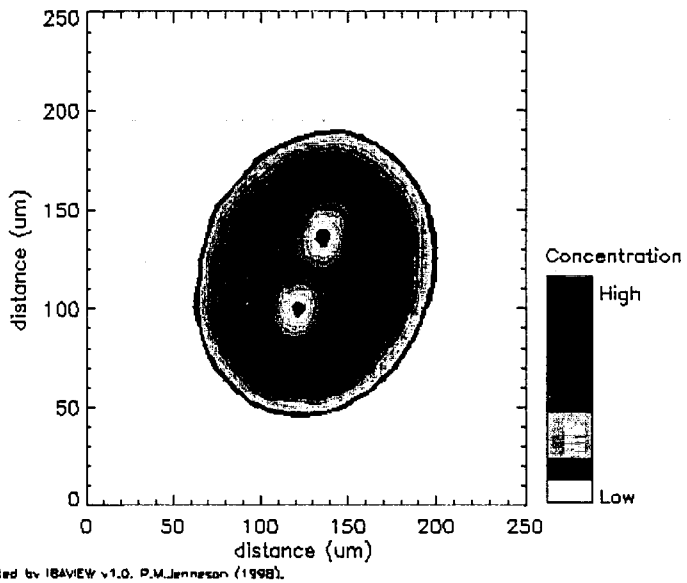

Figure 1. Silicon PIXE Map of the Treated 125✪m Diameter Fibre Optic.

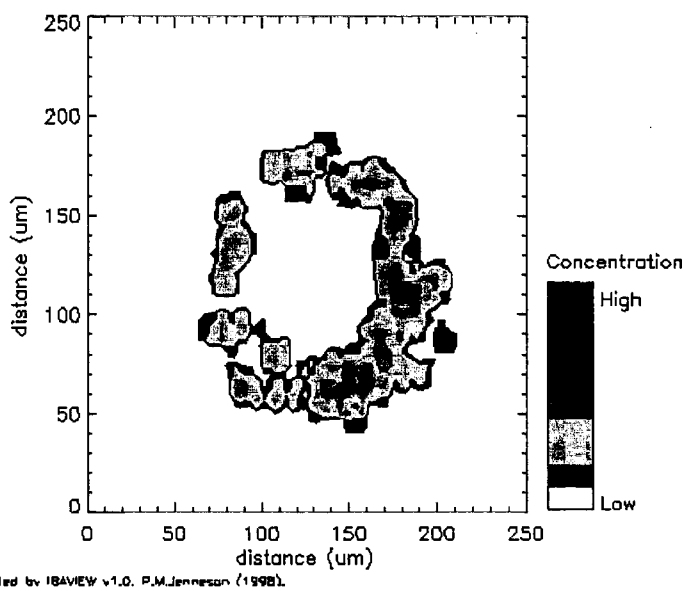

Figure 2. Deuterium NRA Map of the Treated 125✪m Diameter Fibre Optic.

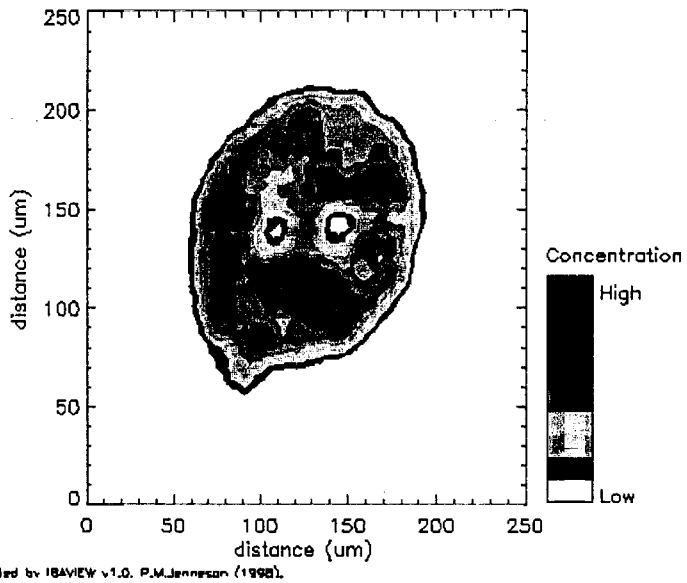

Figure 3. Silicon PIXE Map of the Control 125✪m Diameter Fibre Optic.

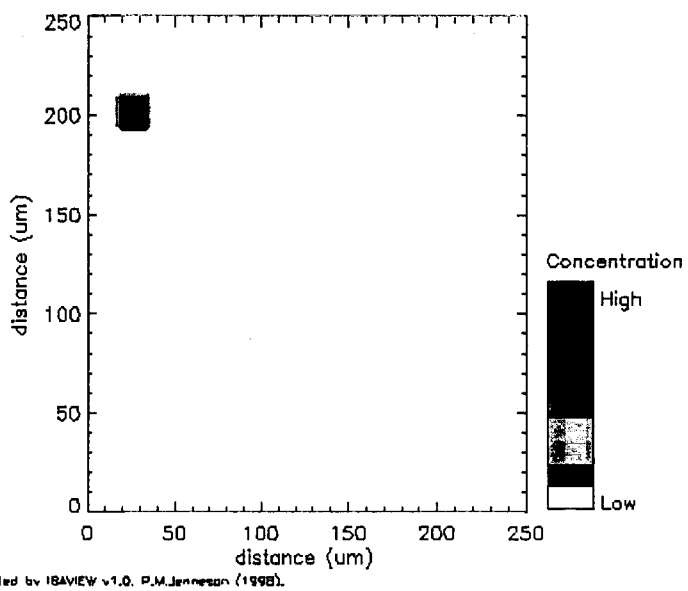

Figure 4. Deuterium NRA Map of the Control 125✪m Diameter Fibre Optic.

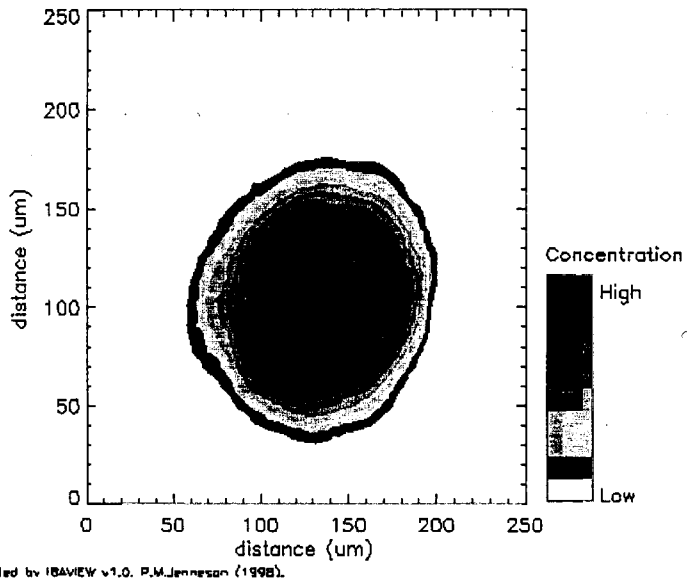

Figure 5. Silicon PIXE Map of the Treated 100✪m Diameter Fibre Optic.

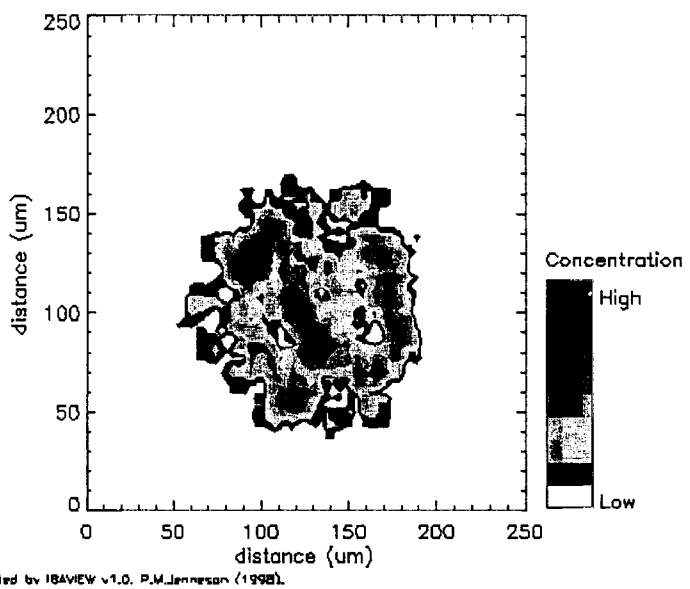

Figure 6. Deuterium NRA Map of the Treated 100✪m Diameter Fibre Optic.

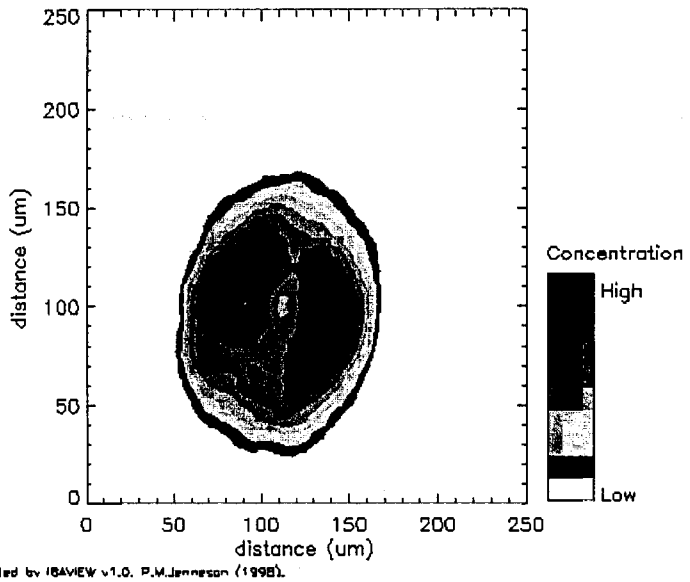

Figure 7. Silicon PIXE Map of the Control 100✪m Diameter Fibre Optic.

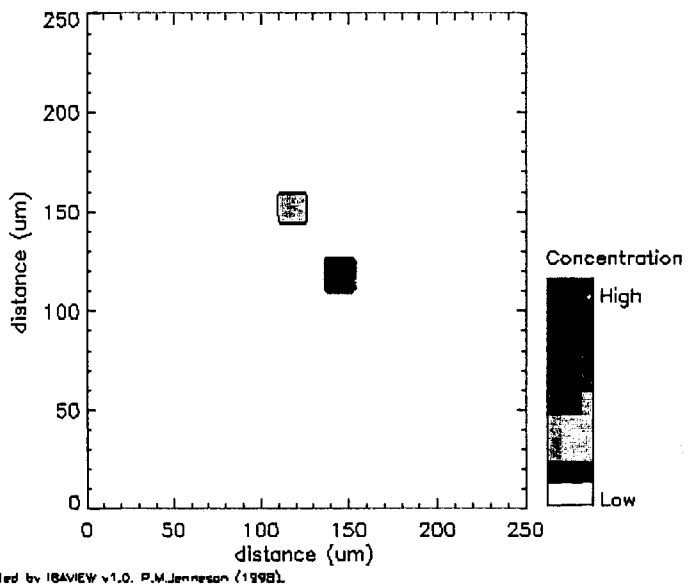

Figure 8. Deuterium NRA Map of the Control 100✪m Diameter Fibre Optic.

Using the data from the two-dimensional map the diffusion coefficient of the deuterium penetrating the 125μm fibre optic was calculated. Data was summed at different radii across the fibre and divided by the corresponding circumference. The resulting one-dimensional plot was then fitted with a Fickian diffusion profile folded with a Gaussian resolution function; this takes into account beam intensity variation across the circular cross-section of the incident beam[11]. Figure 9 shows the plot and fit obtained. The resulting diffusion coefficient was 0.2 ± 0.02$\mu m^2 h^{-1}$.

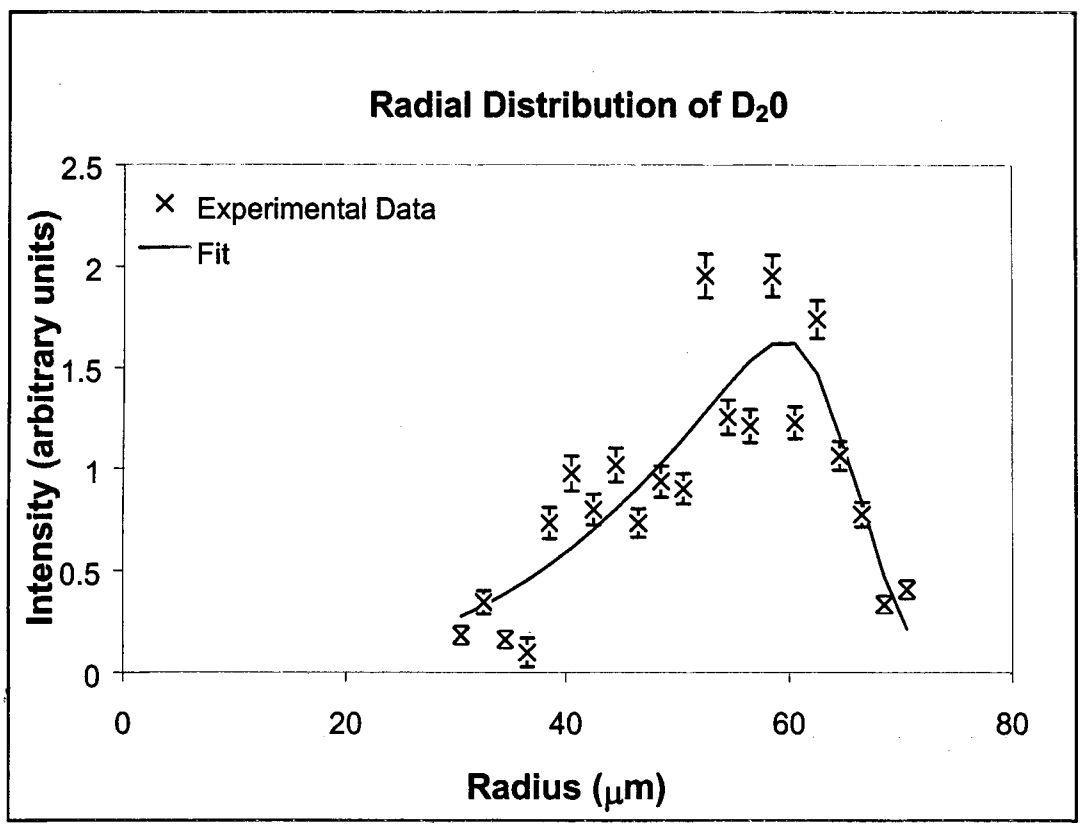

Figure 9. Graph Showing the Annular Deuterium Penetration into the 125μm Fibre Optic.

4. Discussion

Diffusion induced drift in fibre optic pressure sensors is known to be a function of temperature and exposure to liquid phase water only, and not a function of pressure provided the pressure exceeds the saturated vapour pressure of water at the exposure temperature (which maintains the liquid phase [1].) Thus our results under saturated vapour pressure superheated water are representative of down-hole conditions.

An extensive literature exists on diffusion mechanisms for water in silica, but with limited relevance to the conditions and materials employed here. Diffusion constants and mechanisms are known to depend on the 'type' of fused silica (conventionally graded I to IV depending on the manufacturing method [11]; fibre optic material most closely approximates type III, but with lower hydroxyl content.) They are also well known to depend on the detailed thermal history of the glass and its hydroxyl content [12]. For these reasons the use of existing data on conventionally prepared bulk fused silica materials is inappropriate, and many of the experimental methods used (e.g. infrared absorption measurements on flat plates which are successively thinned by polishing) are inapplicable to the fibre geometry. For a recent discussion of water diffusion in bulk silicas see [13] ad references therein.

The reasons for the differences observed in water penetration into the two fibre structures are currently unclear, but presumably relate to differing composition and heat treatment histories. These will be the subject of future studies, our objective here being to establish a credible diagnostic for the large number of samples this will require.

The focussed ion beam technique readily detects water penetration in its early phases; since the

characteristic diffusion distance scales as the square root of time, this permits evaluation of water penetration in experimental times an order of magnitude or more less than those for significant water penetration to the core. With improved micro beam focus dimensions envisaged with upgrades to the Surrey accelerator detection of diffusion on a few micron scale will enable confident prediction of fibre performance over many years with month long exposure. Additionally the technique only requires very short lengths of fibre, as opposed to direct measurements by fibre optic methods, which typically require many metres of sample and render design of the high pressure, high temperature cells problematic. The evaluation of a wide range of fibre construction and coating methods by such methods would be impractical.

5. Conclusions & Further Work:

Three complementary methods – NRA, PIXE and RBS – have been used to obtain accurate analytical data on water penetration into fibre optics and preliminary results on two different fibre types have been presented. Using the techniques the resistance of different fibres to water ingress may be compared, after relatively short diffusion times and on small samples. The technique described will enable us to determine which fibres and coatings will sufficiently inhibit the ingress of water at high temperature and pressure to allow them to be useful in oil well monitoring.

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Figure Captions:

Figure 1. Silicon PIXE Map of the Treated 125✪m Diameter Fibre Optic.

Figure 2. Deuterium NRA Map of the Treated 125✪m Diameter Fibre Optic.

Figure 3. Silicon PIXE Map of the Control 125✪m Diameter Fibre Optic.

Figure 4. Deuterium NRA Map of the Control 125✪m Diameter Fibre Optic.

Figure 5. Silicon PIXE Map of the Treated 100✪m Diameter Fibre Optic.

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