



	<b>Experiment title:</b> SR micro-XRD examination of ceramic fragments from a kiln excavated at the Bronze Age (ca.2300BC) site of IKTANU, Jordan.	<b>Experiment number:</b> CH-929
<b>Beamline:</b> ID13	<b>Date of experiment:</b> from: 31.01.01 to: 03.02.01	<b>Date of report:</b> 27.02.03
<b>Shifts:</b> 9	<b>Local contact(s):</b> Manfred Burghammer	<i>Received at ESRF:</i>

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**Report:**

The main objective of the experiment on Iktanu ceramics, the mapping of main diffracting phases and their correlation to the type of ceramic has been achieved. The more intricate objective of recognising the type of feldspar/pyroxenes and their relation to that of rocks from the area used as grog has been difficult to attain because of the heavy overlap of mixed phases, even at the 2-micron spatial resolution. The data is now undergoing systematic analysis in conjunction with neutron diffraction of the potsherd bulk (RAL) and powder XRD data (DL). Results have been presented at conferences [1].

This first report on CH-929 refers to a short test on textile fibres of archaeological interest.

**Identification of the textiles from Khirbet Qumran caves using microscopy and synchrotron radiation x-ray fibre diffraction [2]**

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In the caves of Qumran the famous Dead Sea Scrolls were found in 1947. An eclectic religious sect, the Essenes, are reported to have lived there. The aim of this project is to find out the type of fibres and the nature of the pigments some of the textiles were dyed with and relate this information to the archaeological questions surrounding the mysterious Essenes, devoted to chastity, purity and messianic teaching.

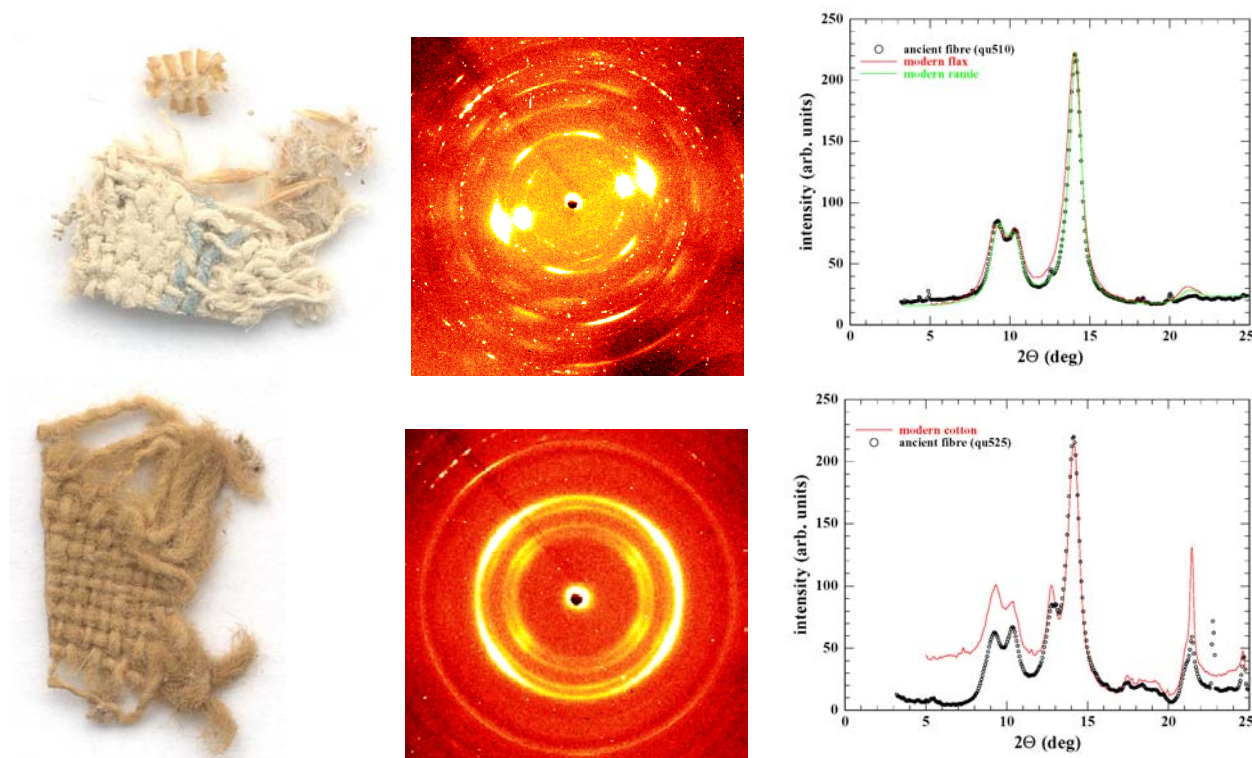
Woven and unspun fibre samples from the caves in Khirbet Qumran identified as bast fibre, cotton or wool by optical and scanning electron microscopy of small fibre bundles extracted from them. In addition, the

intact fibre samples and crystalline particles attached to them have been characterised by synchrotron x-ray diffraction. Single fibrils (10-20  $\mu\text{m}$  thin) extracted from flax and cotton threads were examined by microdiffraction on ID13 where the beam footprint was 2  $\mu\text{m}$ . Nine of the ancient fibre samples were scanned through the beam in 20 steps of 3  $\mu\text{m}$  each. At every position, a two-dimensional pattern was acquired in a 30 s exposure time. Diffraction patterns were compared with those of modern flax, ramie and cotton. The crystalline properties of the ancient fibres was remarkably well preserved in most cases. The details of the diffraction patterns and the crystalline parameters of the ancient plant fibres thus determined (see table) are characteristic of the type of fibre independent of visually recognised morphological features (see figure). The doubtless identification of cotton is most unexpected from the archaeological point of view. Diffraction from micro-crystalline particles attached to some of the textile fragments has been used to investigate their nature; analysis is still in progress.

**Table:** Crystallographic parameters (crystal sizes) of the samples investigated. The values (in  $\text{\AA}$ ) are calculated from the width of the three Bragg reflections 110, 1-10, 200.

Sample	110	1-10	200
Modern flax	43	36	47
Modern ramie	51	39	57
Modern cotton	-	-	61
QUM510	54	47	59
QUM512	51	41	56
QUM518	45	48	56
QUM524	48	35	44
QUM525	-	-	57

**Figure:** Photographs of two textile samples (left), corresponding microdiffraction patterns from single fibres extracted (centre) and diffraction profiles (right). The textile in the first row has been identified as made from plant bast fibre (flax or ramie), The 2D diffraction pattern exhibits the high orientation characteristic for bast fibres, the cellulose crystal size is in good agreement with that of modern bast fibres (see table). The second row presents date from a cotton textile fragment. The typical helical structure of cotton fibrils leads to two crossed fibre patterns.



[1] Murphy B.M, Roberts M.A., Pantos E., Burghammer M, Müller M., Flot D., Riekel C, Zhilin M.G., Prag K., and Prag A.J.N.W. Phase composition of ancient ceramics by x-ray scanning micro-diffractometry and high resolution powder diffraction. 32nd Symposium of Archaeometry, Mexico City, May 2000. <http://srs.dl.ac.uk/arch/posters/mexico/mexico-dl-esrf/>

[2] M. Müller, M. Z. Papiz, D. T. Clarke, M. A. Roberts, B. M. Murphy, M. Burghammer, C. Riekel, E. Pantos, and J. Gunneweg. Identification of the textiles from Khirbet Qumran using microscopy and synchrotron radiation x-ray fibre diffraction. In: Jean-Baptiste Humbert and Jan Gunneweg, editors, *Archaeological Excavations at Khirbet Qumran and Ain Feshka - Studies in Archaeometry and Anthropology*, volume II, chapter XII, pages 177-186. Presses Universitaires de Fribourg (Suisse), Fribourg, in press. <http://srs.dl.ac.uk/arch/publications/qumran-textiles-micro-xrd.pdf>.