Experimental report of EC535
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1. Overview

During the imaging campaign in 2009 at ESRF, France in total ~40 shale/mudstone samples were imaged. The samples were from a number of geological settings in Europe, South Africa and the US. The main purpose was to analyze the primary fracture pattern in samples under study. However, a few imaged samples were from some presumably mature shale formations under HPHT conditions and several of the samples were exposed to gradual rising temperature under atmospheric pressure during the imaging process.

This report will start with a brief summary of this process and continues with a review of the samples imaged and the proposed way forward.

2. CT on the rock samples

A number of shale samples were imaged in two different synchrotron facilities. The samples were from:
- Karoo basin, South Africa which are denoted by "DP" and "KL" throughout this report and in the appended tables
- Kimmeridge Shale, Dorset, UK represented by "DO"
- Green River Basin Shales, Utah represented by "GRS"

Most of the samples were from immature shale deposits; however there were some coming from the Karoo basin, which were cooked already being exposed to magmatic intrusions while are called KL samples, KL being the name of the wellbore. Besides, some of the immature samples were gradually heated under atmospheric pressure to investigate the evolution of the cracks and fracture patterns.

The imaging process was as follows: The samples were rotating either 180 degrees or 360 degrees and they were imaged 1500 and 2500 times respectively. Afterwards a volume reconstruction algorithm was used to reconstruct 3D volumes and generate the horizontal cross-sectional images of various samples. To reduce the size of the images 32 bit images were converted into 8-bit images in the end.

17 samples from Green River Shales (GRS) were in total imaged. GRS formations are immature. One of these samples were cooked at 250 deg. C for 7 days and imaged and one was cooked at 350 deg. C for 12 hours and imaged. Another one was cooked for 12 hours and was scanned 68 times during heating. A cross section is depicted in the figure below showing before and after the heating process.
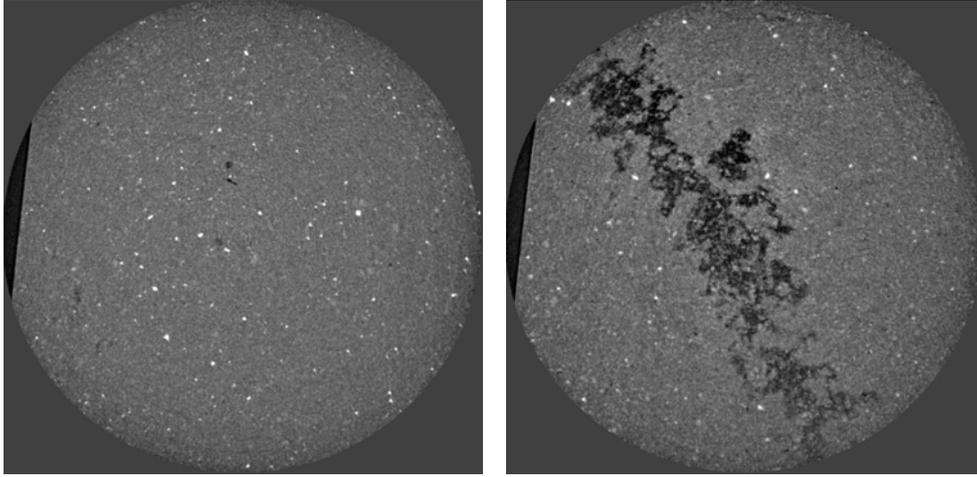
PICTURES OF A 372-th CROSS SECTION OF THE GRS_I_9T__0001 and GRS_I_9T__0068 SAMPLES

17 presumably matured KL samples and 38 DP samples from the South African Karoo basin were imaged. One of the DP samples was exposed to rising temperatures and 12 scans were obtained during this process. The figures below show examples of KL and DP samples.
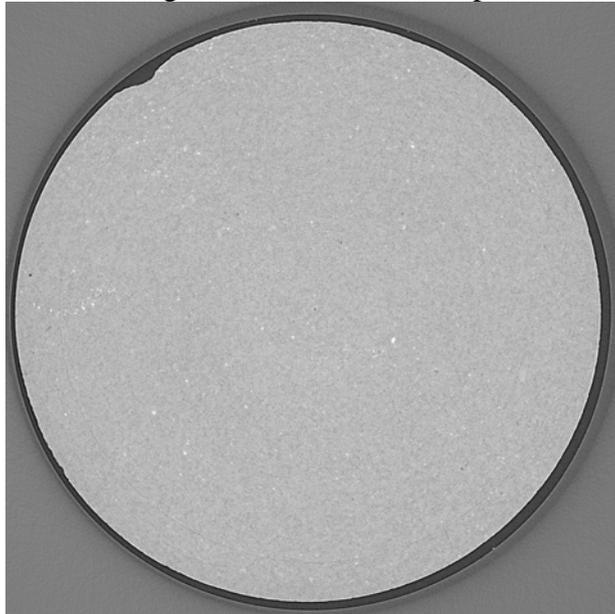
"KL1_6930_10005" SAMPLE

In total, 29 Kimmeridge shale samples were imaged. Two of these samples were imaged after heating at 350 deg. C for 12 hours. One sample was cooked at 250 deg. C for 7 days and imaged. The rest of the samples were combination of mature and immature samples. Samples of these samples are shown in the figures below.

The quality of the scanned samples was very good and we did the 3D reconstruction in the week following the experiment, using ESRF and ID19 facilities.
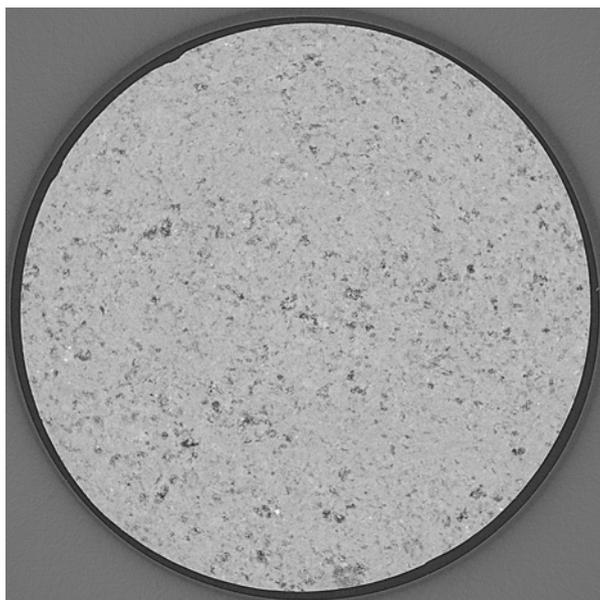
PICTURES OF CROSS SECTIONS OF ONE RANDOM "DO1_D_I0005" SAMPLE

3. The analyses and publications

The softwares AvizoFire and ImageJ were utilized to generate the 3D volumes and analyze the fracture patterns and calculate various porosities of the 3D volumes. Especially interesting are those few samples which were imaged before and after the heating process. These analyses pave the way for international publications of this study.

Two publications have been submitted on this study: one in the best journal of geophysics (Journal of Geophysical Research) is accepted pending moderate revisions, one in the Society for Petroleum Engineering is under review. In both articles, the first two authors are Ph.D. students who participated to the ESRF experiment and/or to the analysis of the 3D data. For both of them, the ESRF experiment is part of their Ph.D. project.

The results of the experiments have been presented also in two international conferences. The references of all publications are given below.

Articles in international journal
Kobchenko, M., Panahi, H., Renard, F., Dysthe, D. K., Malthe-Sorrenssen, A., Mazzini, A., Scheibert, J. Jamtveit, B., and Meakin P. (2011) Fracturing controlled primary migration of hydrocarbon fluids during heating of organic-rich shales, *Journal of Geophysical Research*, accepted pending moderate revisions.

Panahi, H., Kobchenko, M., Renard, F., Mazzini, A., Scheibert, J., Dysthe, D. K., Jamtveit, B., and Meakin P. (2011) 4D X-ray tomography imaging of hydrocarbon escape pathway formation in heated organic-rich shales: a proxy for primary migration?, *SPE Journal*, submitted.

Conference proceedings
Kobchenko, K., Panahi, H., Renard, F., Dysthe, D.K., Malthe-Sorenssen, A., Mazzini, A., Scheibert, J., Jamtveit, B. and Meakin, P. (2011). Talk. Invasion-percolation fracturing controlled primary migration of hydrocarbons during heating of organic-rich shales, European Geosciences Union, Vienna, 3-8 April 2011, Austria.

Kobchenko, M., Panahi, H., Renard, F., Malthe-Sorenssen, A., Scheibert, J. Dysthe, D., and Meakin, P. (2010). Talk.Primary migration of hydrocarbon fluids through invasion-percolation cracking in a source rock, American Geophysical Union, 13-17 December 2010, San Francisco, USA.

4. Tables summarizing the info with respect to the acquired images of the samples

sample	comment	date	acquisition	recons. 3D	volume size	256 gray level	saved source files DISK_FR4 (860 GB)	saved 3D volumes DISK_FR3 (376 GB)	saved 3D volumes DISK_FR1 (916 GB)
1_1X	Elodie			xx			-	-	-
6_35X	Elodie			xx			-	-	-
ANS_6	German	14/11/2009, 01-07	ss OK	xx		-0.74 – 1.39	x	x	x
ANS_6C	German			xx		-0.74 – 1.39	x	x	x
ANS_9	German	14/11/2009, 01-07		xx		-0.74 – 1.39	x	x	x
ANS_9C	German			xx		-0.74 – 1.39	x	x	x
Dixie2_a	6 subsamples of the Dixie2 sample. 9 mm vertical displacement between each scan. ROI 2048x1500	15/11/2009		xx		-0.48 – 1.13		x	x
Dixie2_b		15/11/2009		xx		-0.48 – 1.13		x	x
Dixie2_c		15/11/2009		xx		-0.48 – 1.13		x	x
Dixie2_d		15/11/2009		xx		-0.48 –			x

					1.13			
Dixie2_e		15/11/2009		**xx**	-0.48 – 1.13			x
Dixie2_f		15/11/2009		**xx**	-0.48 – 1.13			x
DO1_D_I		14/11/2009, 08-11	ss OK, mq OK	**xx**	-0.74 – 1.35	x	x	x
DO1_D_II		14/11/2009, 08-11	ss OK, mq OK	**xx**	-0.76 – 1.31	x	x	x
DO1_D_III		14/11/2009, 08-11	ss OK, mq OK	**xx**	-0.73 – 1.34	x	x	x
DO1_D_IX		14/11/2009, 08-11	ss OK, mq OK	**xx**	-0.75 – 1.31	x	x	x
DO1_D_VII		14/11/2009, 08-11	ss OK, mq OK	**xx**	-0.69 – 1.29	x	x	x
DO1_D_VIII		14/11/2009, 08-11	ss OK, mq OK	**xx**	-0.71 – 1.31	x	x	x
DO1_D_X		14/11/2009, 08-11	ss OK, mq OK	**xx**	-0.69 – 1.38	x	x	x
DP1_7760_1		14/11/2009, 05-08	ss OK, mq OK	**xx**	-0.72 – 1.37	x	x	x
DP1_7980_1a	same sample as DP17980_1	14/11/2009, 05-08	a&bspikes&movem, a&b,aa&bb: ss OK, mq OK	**xx**	-0.56 – 1.40	x	x	x
DP1_7980_1b	same sample as DP17980_1	14/11/2009, 05-08	a&bspikes&movem, a&b,aa&bb: ss OK, mq OK	**xx**	-0.56 – 1.40	x	x	x
DP1_7980_1ba	same sample as DP17980_1	14/11/2009, 05-08		**xx**	-0.72 – 1.54	x	x	x
DP1_7980_1bb	same sample as DP17980_1	14/11/2009, 05-08		**xx**	-0.72 – 1.54	x	x	x
DP1_8195_2a	long, 2 scans	14/11/2009,	b: ss OK, mq OK	**xx**	-0.72 –	x	x (except	

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ID	Description	Date	Notes		Pixel	Range			
		05-08				1.47		8th volume)	
DP1_8195_2b	long, 2 scans	14/11/2009, 05-08	b: ss OK, mq OK	**xx**		-0.72 – 1.47	x	x	x
DP1_8195_3a	long, 2 scans DP1_8185_3a ou DP1_8195_3a	14/11/2009, 08-11	b: ss OK, mq OK	en cours			x		
DP1_8195_3b	long, 2 scans	14/11/2009, 08-11	b: ss OK, mq OK	**xx**		-0.76 – 1.53	x	x	x
DP1_8195_4a	long, 2 scans	14/11/2009, 08-11	ss OK, mq OK	**xx**		-0.72 – 1.54	x	x	x
DP1_8195_4b	long, 2 scans	14/11/2009, 08-11	ss OK, mq OK	**xx**		-0.72 – 1.54	x	x	x
DP1_8195_4c		14/11/2009, 08-11	ss OK, mq OK	**xx**		-0.72 – 1.54	x	x	x
DP1_8195b_20C_small	many oven scans	14-15/11/2009	ss OK? mq no OK	**xx**	830x830 pixel	-0.59 – 1.74	x	x	x
DP1_8195b_20C_small_bis	many oven scans	14-15/11/2009	ss OK? mq no OK	**xx**	830x830 pixel	-0.59 – 1.74	x	x	x
DP1_8195b_80C_small	many oven scans	14-15/11/2009	ss OK? mq no OK	**xx**	830x830 pixel	-0.59 – 1.74	x	x	x
DP1_8195T_3__0001_	12 scans during heating (ramp1.txt file)	14-15/11/2009	ss OK?	**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0002_		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0003_		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0004_		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x

DP1_8195T_3__0005_		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0006_		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0007_		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0008_		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0009		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0010_		14-15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8195T_3__0011_		15/11/2009		**xx**	830x830 pixel	-0.70 – 1.78	x	x	x
DP1_8450_1		14/11/2009, 05-08	ss OK, mq OK	**xx**		-0.65 – 1.11	x	x	x
DP1_8470_5		14/11/2009, 05-08	ss OK, mq OK	**xx**		-0.7 – 1.30	x	x	x
DP1-7980-2a	long, 2 scans	14/11/2009, 01-07	ss OK, mq OK	**xx**		-0.68 – 1.42	x	x	x
DP1-7980-2b	long, 2 scans	14/11/2009, 01-07	ss OK, mq OK	**xx**		-0.68 – 1.42	x	x	x
DP1-8185-1	name DP_8185_1	14/11/2009, 01-07	ss OK, mq OK	**xx**		-0.62 – 1.04	x	x	x
DP1-8185-2a1	long, 2 scans	14/11/2009, 01-07	ss OK, mq OK	**xx**		-0.69 – 1.35	x	x	x
DP1-8185-2a2	long, 2 scans	14/11/2009, 01-07	ss OK, mq OK	**xx**		-0.69 – 1.35	x	x	x
DP1-8185-3a		14/11/2009, 01-07	ss OK, mq OK	**xx**		-0.67 – 1.32	x	x	x
DP1-8185-3b		14/11/2009, 01-07	ss OK, mq OK	**xx**		-0.68 – 1.04	x	x	x
DP1-8195-1a	long, 2 scans,	14/11/2009,	ss OK, mq OK	**xx**		-0.70 –	x	x	x

ID	Name / Description	Date	Status		Pixel	Range			
	name DP_8195_1a long, 2 scans, name	01-07				1.31			
DP1-8195-1b	DP_8195_1b Schistes carton	14/11/2009, 01-07	ss OK, mq OK	xx		-0.70 – 1.31	x	x	x
Ei1_2430	Ei12430 + cuttings 920 GLTI (3) 2480 (1)		ss OK, mq OK	xx		-0.77 – 1.54	x		x
EST_1	German	14/11/2009, 01-07		xx		-0.74 – 1.41	x		
EST_1C	German			xx		-0.74 – 1.41	x		x
FS-1	syenite, Frederic			xx		-0.74 – 4.83			x
GRS_I_9T__0001_ to GRS_I_9T__0068_	68 scans of the GRS sample during heating (ramp2.txt file)	15-16/11/2009		xx	830x830 pixel	-0.70 – 2.05		x	1 to 7
JS19	altered peridodite, Jorn			xx		-0.75 – 3.44			x
KL1_8920_1	file name: DP1_8920_1	14/11/2009, 05-08	ss OK, mq OK	xx		-0.79 – 1.54	x	x	x
KL1_8920_3	file name: DP1_8920_3	14/11/2009, 05-08	ss OK, mq OK	xx		-0.76 – 1.65	x	x	x
KL1_8920_4	file name: DP1_8920_4	14/11/2009, 05-08	ss OK, mq OK	xx		-0.75 – 1.67	x	x	x

KL1-6930-1	fasttomo360	13/11/2009, 20-24:00	ss OK, mq OK	**xx**	-0.71 – 1.08	x	x
KL1-6930-2	fasttomo360	13/11/2009, 20-24:00	top 1mm missing, ss OK, mq OK	**xx**	-0.73 – 1.12		x
KL1-6930-3	fasttomo360	13/11/2009, 20-24:00	ss OK, mq OK	**xx**	-0.70 – 1.09		x
KL1-6930-4	fasttomo360	13/11/2009, 20-24:00	ss OK, mq OK	**xx**	-0.76 – 1.11		x
KL1-8715-1	fasttomo, scan over 180 degrees	13/11/2009	spikes&movem, ss OK, mq OK	**xx**	-0.46 – 1.16		x
KL1-8715-2	fasttomo, scan over 180 degrees	13/11/2009	spikes&movem, ss OK, mq OK	**xx**	-0.47 – 1.17		x
KL1-8715-3	fasttomo, scan over 180 degrees	13/11/2009	spikes&movem, ss OK, mq OK	**xx**	-0.43 – 1.15		x
KL1-8715b-1	fasttomo360, same sample as KL1_8715-1	13/11/2009, 20-24:00	ss OK, mq OK	**xx**	-0.73 – 1.48		x
KL1-8715b-2	fasttomo360, same sample as KL1_8715-2	13/11/2009, 20-24:00	ss OK, mq OK	**xx**	-0.71 – 1.46		
KL1-8715b-3	fasttomo360, same sample as KL1_8715-3	14/11/2009, 01-07	ss OK, mq OK	**xx**	-0.74 – 1.54		
KL1-8825-1	fasttomo360	13/11/2009, 20-24:00	ss OK, mq OK	**xx**	-0.76 – 2.44		x
KL1-8825-3	fasttomo360	13/11/2009, 20-24:00	ss OK, mq OK	**xx**	-0.74 – 2.30		

KL1-8825-4	fasttomo360	13/11/2009, 20-24:00	ss OK, mq OK	xx	-0.74 – 2.29	x
ROR-ST-01	priority 1, Oliver		ss OK	xx	-0.75 – 3.79	
ROR-ST-02	priority 2, Oliver		ss OK	xx	-0.75 – 3.79	