New insights on diagenetic quartz overgrowths by cathodoluminescence

Benoît Maréchal*, Alain Chartier**, Philippe Blanc***,
Médard Thiry*, Nicole Guilhaumou** and Claudine Durand****
** ENSMP, Fontainebleau, ** ENS, Paris, *** Univ. P.M. Curie, Paris,*
*****IFP, Rueil-Malmaison, France*

Abstract: *Decrease of porosity due to quartz overgrowths damages sandstones reservoirs. To understand the causes of these overgrowths, and become able to predict them, identification and quantification are a necessary first step. Cathodoluminescence is induced in many solids by electronic irradiation, and can be observed in colors, or in a SEM in grey levels, or analysed by a spectrometer. The presence of some "activators", or a modification of the interatomic bonds may be at the origin of CL. The luminescence of quartz is weak, particularly on the overgrowths, and the quality needed for the signal recording is thus higher. We present two series of results obtained on:*

- *Fontainebleau sandstones: cathodoluminescence allows identification and quantification of the overgrowths by SEM image analysis.*
- *North Sea reservoir sandstones. we tried to interpret the observed zonations, and look at the evolution of the signal during irradiation.*

Cathodoluminescence appears as a unique means to quantify these overgrowths. Observation and quantification are still time consuming, but will be improved by a simultaneous detection of the spectra by a CCD camera. A field of research is still open to understand if these zonations come from incorporation, at some steps of the diagenesis, of any "external" element or if it is due "only", as it is in the present cases, to different growth rates, or even to recrystallization of amorphous silica.

Introduction

Decrease of porosity, and thus of permeability, due to quartz overgrowths is one of the most damaging effects of diagenesis in some sandstones reservoirs. Identification and quantification of these overgrowths are therefore a necessary first step to understand the causes of these overgrowths, and become able to predict them. Classical petrographic observation generally fails to give a quantitative estimation: there is often no crystalline demarcation between the grain and the overgrowth, only some clay rims or fluid inclusions allow distinction of the detrital and diagenetic parts.

Previous works, beginning by Sippel (1968), have shown that use of cathodoluminescence might allow a better distinction between the overgrowths and the detrital grains. The technique has been used with a particular interest in pressure solution (Sprunt and Nur, 1977), in detecting the origin of detrital grains (Zinkernagel, 1978) and has allowed further distinction of "zones" within the overgrowths (Hogg, 1989), opening the field of interpretation of the conditions, and/or steps of the growth (plate I). Quantification of the respective volumes of overgrowths versus detrital grains (Evans *et al.*, 1994, Demars, 1994) has increased the applicability of the technique.

Our work was done in the frame of diagenesis studies, and intended:

- to quantify the overgrowths, in order to get controls on the amount of precipitated silica for further modeling of diagenesis process: this was done on Fontainebleau sandstones, which have been widely used as models for clean sandstones.
- to try to understand if the "zones" are witnesses of different physical conditions during crystallisation or if impurities are acting as chemical tracers. This is also necessary to set the conditions in diagenesis modeling, and was done on samples from the Dunbar area, in the North Sea, already studied by Hogg *et al.*, (1992).

Theoretical background

When an electron beam strikes a sample, many reactions are induced, which are schematized in fig 1. The basic process of luminescence (Rémond *et al.*, 1992) involves the excitation of an electron from the ground state to an excited state, followed by a de-excitation process to the ground state accompanied by the emission of a photon, according to two possible mechanisms, fluorescence or phosphorescence. The luminescence emission may be an intrinsic property of the crystal, i.e. characteristic of the host lattice, or an extrinsic property, i.e. resulting from incorporation of transition metals, rare earths or actinides, in the crystal lattice. Figure 1 shows that the cathodoluminescence signal may come from a volume larger than the other signals, either electronic or photonic, and illustrates the resolutions that can be achieved for the different images.

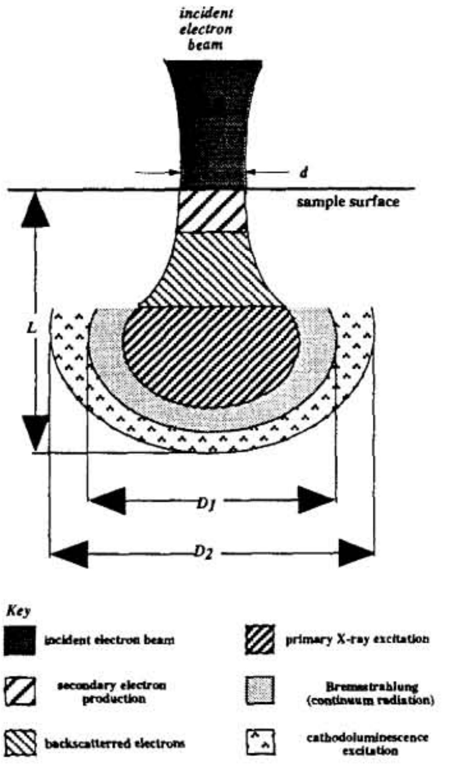
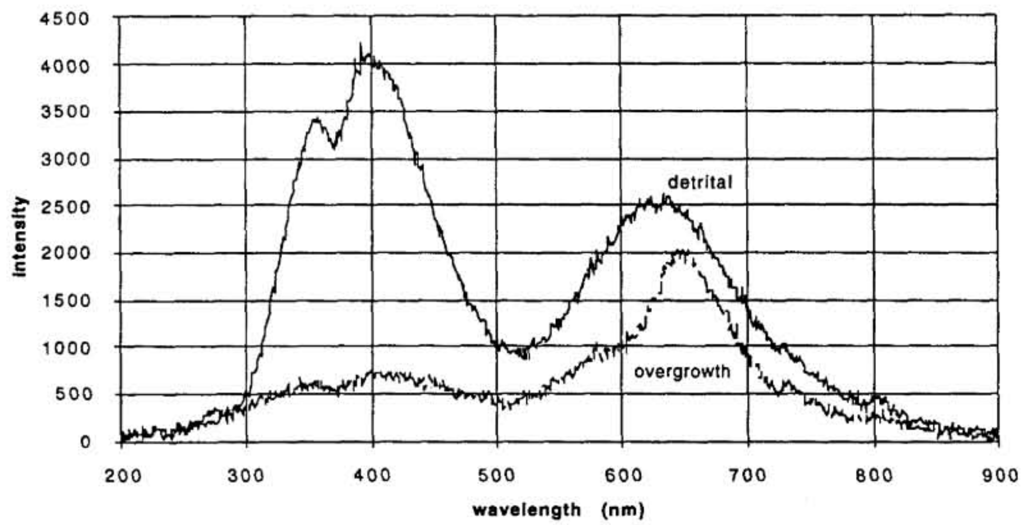


Figure 1 : A qualitative picture of the interaction of an electron beam with a solid (after Marshall, 1988). The beam is focused to a diameter *d* (10 - 100 nm) at the surface. Elastic and inelastic interactions take place within the solid and include such processes as excitation, scattering, absorption and diffusion. These lead to a reduction in the energy of the electrons with depth and an enlargement of the beam. Secondary and backscattered electrons are produced throughout the volume, but only those near the surface point of incidence will escape from the solid. X-rays are emitted from the volume featured by *D1*. Cathodoluminescence is generated over the entire region of diameter *D2* where the electrons retain more than a few eV energy. CL emission is not completely absorbed within the solid and some of that generated even at the greatest depths will exit from the surface. CL emission extends a distance *L* (estimated in the 2-8 µm range) inside the material. The enlarged region of CL production is a limitation to the resolution for CL observations on instruments linked to a SEM (compared to secondary or backscattered electrons).



U.V.	Violet Blue Green Yellow Red	I.R.

Figure 2. Luminescence spectra for detrital and authigenic quartz in Fontainebleau sandstone. The detrital grain is more luminescent than the cement. One can recognize the peaks in the "blue" (<500 nm) and "red" (>500 nm) ranges (raw data).

The cathodoluminescence of minerals has been widely studied (Walker and Burley, 1991), mostly of the ones giving more signal, as carbonates (Amieux 1982), sulfates (Blanc *et al.*, 1994), phosphates, zircons (Cesbron *et al.*, 1993)...The luminescence of quartz is weak, compared to other minerals, and is mainly observed in the blue and red regions of the visible spectrum, with an extension to the UV range (fig 2). Zinkernagel (1978) had proposed a classification for the colors of detrital grains, stating that authigenic quartz did not give any signal. Further progress in experimental devices has made possible to "see", and try to interpret, the signal produced by overgrowths (Marshall, 1988, Ramseyer *et al.*, 1988, 1989).

Many works have already tried to explain the causes of the luminescence signals, either with a geological or a physical approach. Among the impurity (extrinsic) centres, are substitution of Si by Al or Ti. Among the intrinsic centers, the most frequently cited is the E_1', associated with oxygen vacancies. Most of the interpretations attribute the intrinsic defects to the blue-UV range, while the red range is more related to impurities. Effect of damages induced either by shock (Hanusiak and White, 1975) or by irradiation (Griscom, 1979) have also been studied.

Experimental design

The cathodoluminescence signal may be either observed in a microscope, keeping the color information, or recorded as a grey level image in a SEM, or analysed through a monochromator, to find out the spectrum. In the present study, two devices have been used, one for image observation and analysis, the other for spectral analysis. The samples have been prepared as carbon coated polished thin sections.

For image observation and analysis, the analytical equipment consists of a JEOL JSM 840 Scanning Electron Microscope fitted with an Oxford Instruments CL detector (ellipsoidal mirror) and a photomultiplier. The SEM is connected to an image analysis system, KONTRON IBAS 2.0, which collects and stores images acquired with the microscope. The conditions were a 15 kV accelerating voltage and current between 30 nA and 100 nA. CL observations are possible at magnifications higher than x180. The pictures are acquired at 512 x 512 pixels resolution with 256 grey levels per pixel. The acquisition is achieved in between 3 and 5 minutes. The geometrical setting of the microscope does not allow the simultaneous recording of cathodoluminescence and back-scattered signals, so the images were taken with secondary electrons, which, in the case of Fontainebleau sandstones, give a satisfactory contrast. For other sandstones, back-scattered electrons images would be necessary.

For spectral analysis, the SEM is also a JEOL JSM 840, fitted (Blanc *et al.*, 1994) with a parabolic mirror, a Jobin-Yvon H10 UV grating spectrometer and an Hamamatsu R636 GaAs photomultiplier. A spectrum is recorded between 200 and 900 nm, (Wood aberrations appear at 510 and 800 nm, not disturbing the spectrum) within about 4 minutes. This acquisition time may be too long to record very short-lived emission variations, and may include irradiation damages, but all spectra have been recorded in the same conditions, in order to be comparative.

The Fontainebleau Sandstones

** The type of the silicification*

The Fontainebleau Sand (Oligocene, Paris Basin) contains quartzite lenses resulting from a silicification by groundwater in near surface environments (Thiry *et al.*, 1988).The quartzite lenses are composed of tightly cemented quartz sandstone with low residual porosity. The detrital grains are very clean and in most examples display no impurities which could underline the overgrowths, preventing to estimate the cement percentage under ordinary petrographic microscope. Cathodoluminescence

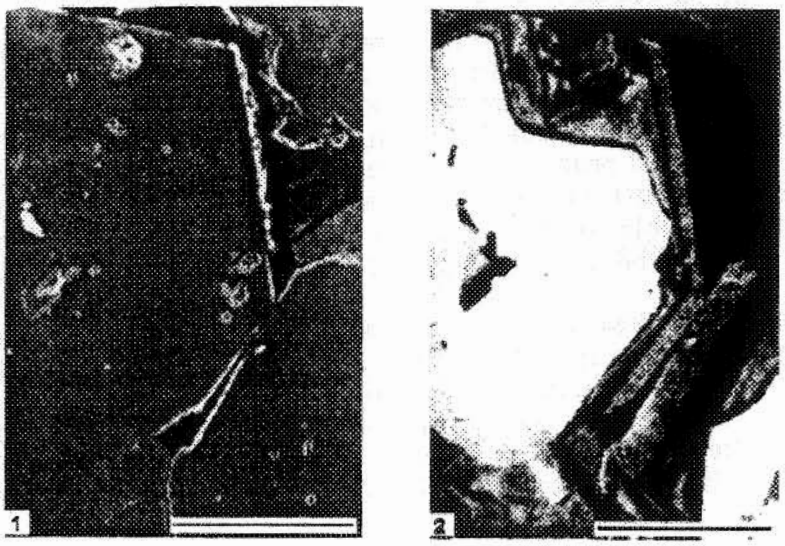


Plate I : Zonations in quartz overgrowths (Sandstone from the Brent Group, Dunbar, North Sea).
1 - Secondary electron image. The cemented grains show very regular faces of quartz crystals.
2 - SEM/CL image. Detrital grains are bright luminescent. The cement is composed of 4 zonations alternatively dark and luminescent.
Scale bar is 100 micrometers.

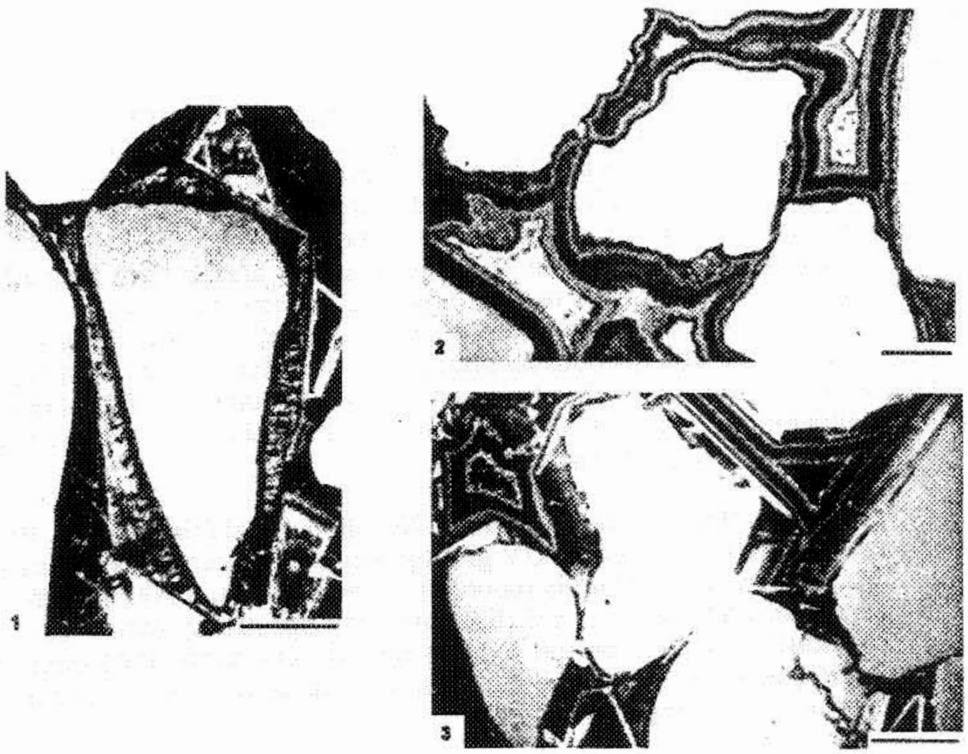


Plate II : Cement structures revealed by SEM/CL (Fontainebleau Sandstone, Paris Basin).
1 - The detrital grain at the center is luminescent and offers a well-developed overgrowth with crystalline faces. The overgrowth is composed of a non-luminescent layer around the core and flame-like figures surrounding it.
2 - Cement is composed of isopachous layers filling the pores. The final cement occluding porosity is very luminescent.
3 - Syntaxial overgrowths are surrounding detrital quartz grains. The pores are filled by isopachous layers of silica recrystallized in quartz.
Scale bar is 50 micrometers.

shows quite well the detrital grains and allows to distinguish three main habits in the cement (Maréchal, 1995):

- homogeneous and subeuhedral quartz overgrowths sutured with polygonal contacts (see plate II). Contacts between the cemented grains form straight lines that converge towards triple joining points. These are syntaxial overgrowths around the detrital grains.
- some cement shows overgrowths with several zonations of dark and clear stripes. In this case, the first strip laying against the detrital grain is always dark (without luminescence). Some of these stripes display flame- or feather-like structures make up of successive bright and dull sectors radiating from the core (plate II).
- isopachous layers surrounding the detrital grains (plate II). These coatings are alternatively bright and non-luminescent. The pores are generally filled by a late stage of quartz deposit, very luminescent and without internal structure.

These cements can be associated and transitions between them have been recognised (Maréchal, 1995). They may be arranged in wide sequences within a thin section, evolving from isopacheous deposits to flame-like growths and finally euhedral overgrowths. Cement sequence may be repeated and superimposed. This sequence may point to physical and chemical changes in the environment at successive times of the cementation. The isopachous layers may be related to amorphous or colloidal silica development in supersaturated solutions, whereas the syntaxial overgrowths may be related to more dilute solutions. Repetition of the cement sequence indicates fluctuations of the environmental conditions during time of silicification.

** Porosity assessment*

Processing of the cathodoluminescence pictures by image analysis allows to quantify porosity and quartz cement. The image analysis method we used is closed to that provided by Evans *et al.* (1994). It needs the record of secondary electron and cathodoluminescence images and consists of the following steps (as described in plate III):

- getting a binary image of quartz and porosity, by segmentation of the secondary electron image from a grey level histogram.
- getting a binary image with detrital grains and other phases, by segmentation of the CL image from a grey level histogram.
- addition of both binary images to obtain a three phase image distinguishing detrital grains, quartz cement and porosity.

The image analysis shows that the cement of the quartzite lenses is more important (about 35 %) when isopachous silica layer deposited and recrystallized and the porosity is nearly occluded (residual porosity of 1 or 2 %). In contrast, when the cement is made of subeuhedral quartz overgrowths, cementation is reduced (30 %) and porosity reaches 5 %.

These decreases in porosity values can be related to decreases in permeability (Bourbié and Zinszner, 1985). Moreover, the image analysis allows to assess that the primary porosity of the sand before cementation was of about 35 % and very constant. Other parameters (e.g. apparent diameter) can also be quantified from image analysis.

North Sea reservoir samples

The cathodoluminescence of quartz cements in Brent group sandstones of Alwyn south, UK North Sea, has already been studied by Hogg (1989), and Hogg *et al.*, (1992). They distinguished up to four sequential zones, alternatively "dark" and "bright," starting from the "bright" detrital grain, within a same overgrowth, becoming increasingly euhedral from core to rim (Plate I), and interpreted them as witnesses of successive silicification pulses proceeding from variations in crystal growth rates and

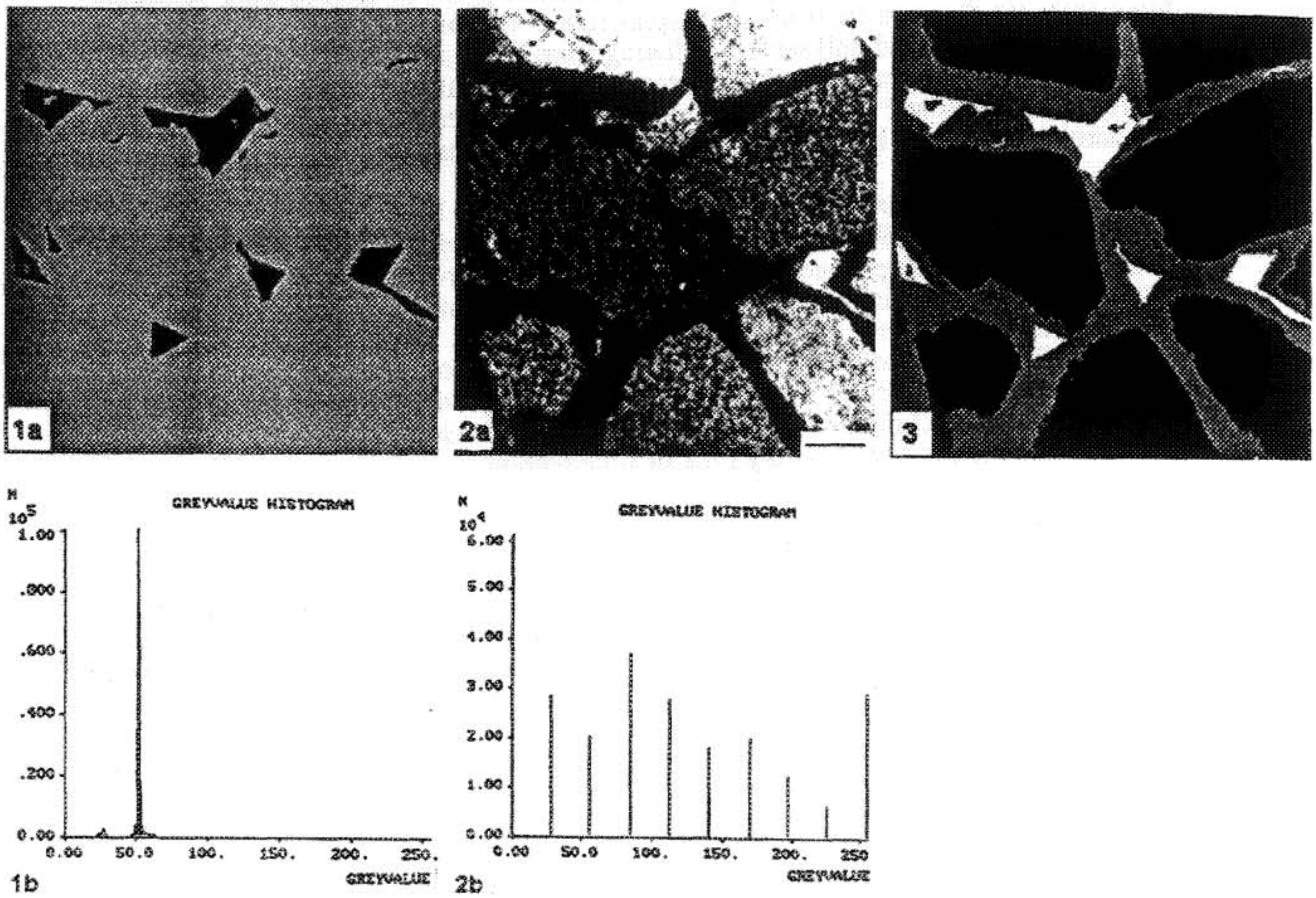
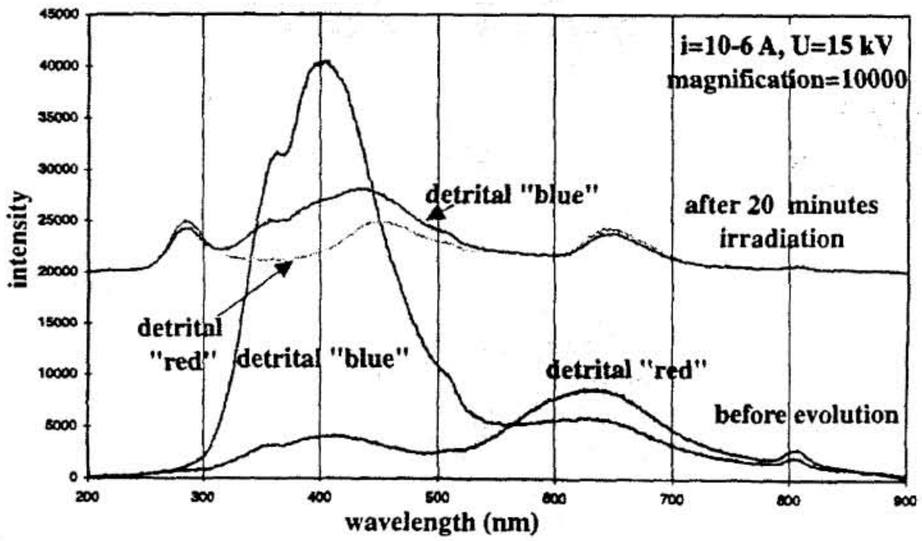


Plate III : Sequence of steps in image analysis process. Sample of quartz cemented sandstone (Fontainebleau).
1 - a) Secondary electron image, after corrections and filtration. Pores are darker than minerals. b) Grey level histogram from image 1a. The little peak around 30 represents pores. Segmentation at 45 separates quartz from porosity.
2 - a) SEM/CL image acquired and filtered in order to reduce noise and increase contrasts. Detrital grains are more luminescent than cement. b) Grey level histogram from image 2a. Segmentation is not easy, but detrital grains are over 80. Manual corrections are needed to optimize the segmentation.
3 - Addition of both segmented images. The final image is in three phases : black for detrital quartz, common grey for cement (here, 32 %) and white for porosity (4,8 %).
Scale bar is 50 micrometers.

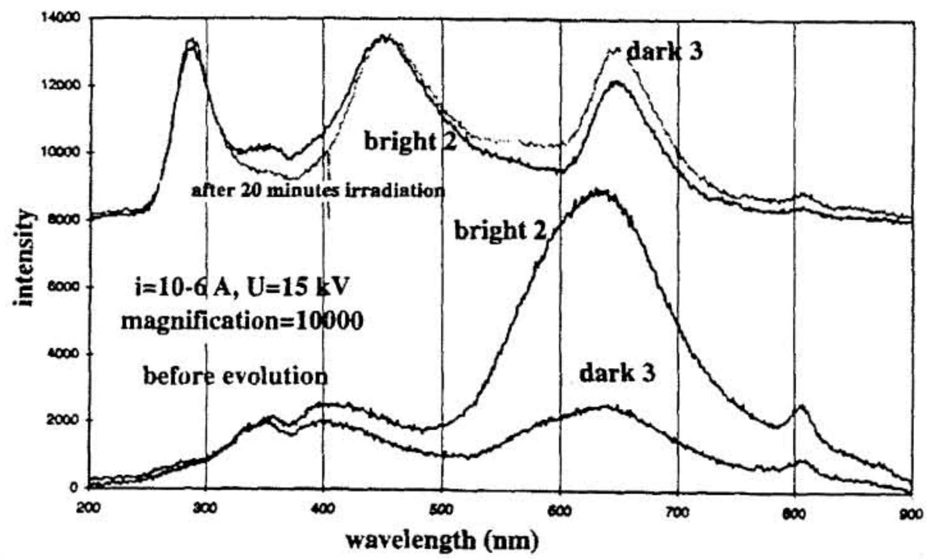
silica supply. The observations were confirmed by Cordon (1994), who used cathodoluminescence as a tool to locate fluid inclusions (Cordon and Guilhaumou, 1995).

The purpose of our spectrometric study was to find fingerprints in the "zones" of the overgrowths, in order to decide between possible impurities brought by different water sources, or different crystallisation conditions. Spectra recorded on the detrital grains and on the successive dark and bright zones are shown on figure 3a and 3b, lower spectra.

The spectra of respectively "detrital blue" (< 500 nm) and "detrital red" (> 500 nm) grains show that the wavelengths are in the ranges usually found for quartz. The overgrowths signals are weaker, in intensity, but at the same position in energy as the detrital grains. No signal at "new" position is detected. From this we infer



a.



b.

Figure 3: a) spectra of detrital grains before (lower) and after 20 minutes irradiation (upper):"new" bands have appeared during irradiation, superimposed to the initial "blue" and "red" ones.
b) spectra of overgrowth zones, respectively "dark" and "bright", before (lower) and after irradiation (upper), showing that both the signals before and after irradiation are within the same energy domains whatever the localisation (detrital/overgrowth) in the sample: only the relative intensities differ (raw data).

that there is probably no new cause of CL signal, e.g. impurities acting as activators, as REE in zircons (Cesbron *et al.* (1993), in the overgrowths, relative to the detrital grains.

Obviously, the signal width is such that it may include several elementary signals. So, in order to try enhance the contrast between these "signatures", the current intensity has been increased from 10^{-7} to 10^{-6} A. The overall signal was increased by a factor about 50. But this led to a continuous modification of the signal as a function of time: some peaks increased, other decreased, and new ones appeared. After 20 minutes irradiation, a kind of plateau was reached, the signal of the detrital grains was lesser than it was at the beginning, with an evident modification of the spectra: the blue part is reduced, while the red one is included in a wider band. The spectra of the overgrowths is of the same order of magnitude as at the beginning, but with also obvious modifications. Comparison between the spectra after irradiation suggests that new signals have arisen, which are superimposed to the initial ones, at the same position whatever the initial spectrum: 286 nm, 450 nm and 640 nm (i.e. 4.3 eV, 2.75 eV and 1.9 eV). This appears then as a modification induced by irradiation, but does not allow, as could have been hoped, a "revealation" of pre-existing differences, because the "new" peaks are at the same position in all the grains, "blue" or "red" detrital as well as "bright" and "dark" overgrowths.

Comparison with interpretation of damages induced by other types of irradiation (Griscom, 1979), suggests that the band at 4.1 eV may be due to the stabilisation of an irradiation induced oxygen hole by trapping an electron. The line at 2.8 eV (440 nm) might be associated to the formation of transient oxygen vacancy-peroxy linkage intimate pairs. No evident interpretation have been found yet for the band at lower energy. However, the similarity between all spectra after irradiation seems consistent with the absence of specific elements in the overgrowths relative to the detrital grains.

Discussion

One of the most important difficulty of the image analysis method is always the statistical validity of the quantitative results (Durand, 1994). In our study, cathodoluminescence images with SEM could only be done at relatively high magnifications (x 180 and x 250 with the ellipsoidal mirror while parabolic mirrors allow magnifications down to x 80). Under these conditions, only 3 or 4 grains and pores were caught on one image and acquisition of numerous pictures was needed (in our study we used 40 views per thin section) to have a statistical mean. The other point is that automatisation of the grey level segmentation is still difficult on reservoir samples, due to variations in the grey levels of both the CL and the BSE images. Acquisition and analysis are still time consuming.

However, with better appropriate equipment, e.g. simultaneous detection of the spectra by a CCD camera, the study of quartz overgrowths using SEM, cathodoluminescence, spectroscopy and image analysis could be more powerful than other methods, as has been studied by Evans *et al.*, 1994 (e.g. point counting gives large errors, mainly underestimations) and provide also secondary information (grain size and shape,...) that could be used for detailed analyses.

Demars *et al.* (1996) had found Al and Li in quartz overgrowths of sandstones from the Paris Basin, and explained by this reason the signal they found at 330 nm, which is different from our values. Bruhn *et al.* (1996), studying Fontainebleau quartz, relate the colors to the presence of trace elements, Fe to orange, and Ti to blue, as Sprunt had already done (1981) in her comprehensive study, but they draw attention on the fact that PIXE may encompass heterogeneities at the scale of 50 µm, and thus they do not state that the trace elements replace effectively silicon in the lattice.

In the spectra of the samples we have studied here, the "bands" correspond to the same wavelengths, or energy, for the major part of the signal, before irradiation. Further irradiation fails to "reveal" substantial differences between detrital grains and overgrowths in the reservoir sandstones samples. This leads to the interpretation that, even if the water composition may have changed during the time of growth of the overgrowths, this did not result in incorporation of specific elements in the quartz lattice, nor in the creation of specific defects. More probably, physical conditions like temperature and growth kinetics, as suggested in detrital grains by Zinkernagel (1978), should be involved in the differentiation of zones. But local traces analyses would be useful to confirm this hypothesis, and further work is needed to understand the effect of kinetics of quartz crystallization on cathodoluminescence signals.

Conclusions

Cathodoluminescence appears as a unique means to observe and quantify the quartz overgrowths, that may reach, in Fontainebleau sandstones, up to 35 % , i.e. a nearly complete occluding of the initial porosity. Even if they are often less developed in reservoir sandstones, their consequences on permeability decrease are heavy.

Quantification of quartz overgrowths is still time consuming, but will be improved by progress in equipment. A better quantification of the overall quantities of overgrowths in reservoir sandstones will then be available, and provide sound data on the extent of quartz precipitation, which is up to now poorly evaluated, and is necessary for geochemical mass balance of chemical reactions.

The causes of zonations are still not clearly understood. In the cases studied, it seems that they are due "only" to different growth rates, or even to recrystallization, without incorporation of external elements. Extra irradiations did not provide a better way of characterization. Fundamental research is needed to help understand the phenomena, and relate them to the paleo-conditions, providing thus a better frame for diagenesis modeling.

Acknowledgments: Part of the work presented here was financed by TOTAL-CST, and image analysis was performed in their laboratory of St-Rémy-les-Chevreuse, with the appreciated technical assistance of H. Cadillac. F. Sommer and C. Ahamdach-Demars are thanked for samples supply and fruitful discussions. J.P. Duraud, from CEA, brought us interesting suggestions on the interpretations of irradiated samples spectra.

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