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In Situ Experiments in the Scanning Electron Microscope Chamber

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1. Introduction

Since the first scanning electron microscope by Knoll (1935) and theoretical developments by von Ardenne (1938a, b) in the 30's, this imaging technique has been widely used by generations of searchers from all the scientific domains to characterize the inner structure of matter. Even if the obtained information is essential for matter description or comprehension of matter transformation, the main constraints associated with classical electron microscopy, i.e. the necessity to work under vacuum and the necessity to prepare the sample before imaging, have always limited the possibilities to "post mortem" characterisation of samples and avoided observation of biological samples.

Electron microscopists early identified the necessity to undergo these limits. The development of a SEM chamber that is capable of maintaining a relatively high pressure and that allows imaging uncoated insulating samples began in the 70's and has been "achieved" in the late 90's – early 00's (Stokes, 2008) with the commercialisation of the low-vacuum and environmental SEM. The availability of new generations of electron guns (and more particularly the field effect electron gun characterized by a very intense brightness), as well as the new generation of electronic columns that are now commonly associated with the environmental scanning electron microscopes opens new possibilities for material characterisation up to the nanometer scale. The development of this generation of microscopes have opened the door for performing real time experiments, using the electron microscope chamber as a microlab allowing direct observation of reactions at the micrometer scale. Many SEM providers or researchers have developed specific stages that can be used for the *in situ* experimentation in the scanning electron microscope chamber. This field is one of the most interesting uses of the ESEM that offers fantastic opportunities for matter properties characterisation. Even if numerous recent articles and reviews are dedicated to *in situ* experimentation in the VP/ESEM (Donald, 2003 ; Mendez-Vilas et al., 2008 ; Stokes, 2008 ; Stabentheiner et al., 2010 ; Gianola et al., 2011 ; Torres & Ramirez, 2011), no one describes all the possibilities of this technique. The present chapter will provide a large – and as exhaustive as possible – overview of the possibilities offered by the new SEM and ESEM generation in terms of "*in situ* experiments" focussing specifically on the more recent results (2000-2011).

This chapter will be split into five parts. We will first discuss the goals of *in situ* experimentation. Then, specific parts will be devoted to *in situ* mechanical tests, experiments

under wet conditions, and a forth part dedicated to high temperature experiments in the SEM. Last, a specific part will be devoted to the "future" of in-SEM experiments. In each part, the main limits of the technique as well as the detection modes will be reported. Each part will be focussed on examples of the use of the technique for performing *in situ* experiments.

2. Goals and implementation requirements of *in situ* experimentation

The main goal of *in situ* experimentation in the SEM (or ESEM) chamber is to determine properties of matter through the study of its behaviour under constraint. This requires the combination of data collection over a given duration (on a unique sample) and image treatment for information extraction. The studied properties are generally related to microscopic phenomena and hardly assessable by other techniques. *In situ* experiment in the SEM chamber corresponds to both imaging systems in evolution under a constraint and imaging systems stabilized under controlled conditions.

To achieve this goal, several requirements are necessary:

- The duration of the phenomenon to be observed must be suitable with the image recording time. If the system evolution is too fast, it will be impossible to record several images and observe this evolution. At the contrary, if the reaction kinetic is low, the time necessary for image recording will be too long and incompatible with experimentation. The high and low limits can be estimated ranging between 2 minutes and 48 hours.
- The system must remain stable under the environmental conditions and/or irradiation by the electron beam during the time necessary for image recording. In the case of easily degradable samples, it is necessary to adjust the imaging conditions (high voltage, beam current, aperture, working distance, detector bias…) constantly, as the sample environmental conditions are modified during the experiment. Thus, the effect of the electron beam on the sample morphology modifications must be verified. Some authors report that it can act as an accelerator (Popma, 2002) or inhibitor (Courtois et al., 2011) of the observed reactions.
- The image resolution must fit well with the size of details to be observed. Improvements in the image resolution have been achieved in the last decade thanks to the field effect emission guns. However, the presence of gas in the VP-SEM/ESEM chamber contributes to the incident electron beam scattering and subsequent degradation of the image resolution. Thus, the acquisition conditions must be adapted to the sample to be studied depending on the higher magnification to be reached.
- The gaseous environmental conditions in which the studied system evolutes (or can be stabilized) must be reproduced in the SEM/LV-SEM/ESEM chamber. The development of the ESEM offers real new opportunities in term of composition of the atmosphere surrounding the sample. The large field detector and the gaseous secondary electron detector (Stokes, 2008) have been developed specifically for imaging under "high pressure" conditions (up to 300Pa and 3000Pa respectively) whatever the gas composition (air, water, He, He+H_2 mixtures, O_2). Other detectors have been developed for very specific applications (high temperature under vacuum (Nakamura et al., 2002), EBSD at high temperature (Fielden, 2005)).

- The constraint in which the studied system evolutes (or can be stabilized) must also be reproduced in the microscope chamber. Some devices are commercialized by official sellers. Among them, we must report the Peltier stage for temperature control in the -10 to 60°C range, hot stages for temperature control up to 1500°C, stages for mechanical tests (Figure 1). Some authors have developed their own specific stages adapted to the problem to be treated (Fielden, 2005; Bogner et al., 2007). However, the development of miniaturized stages that can be positioned in the SEM chamber without creating perturbations on the incident electron beam can be really challenging. This will probably be a key in the development of *in situ* experimentation in the next years (Torres & Ramirez, 2011).

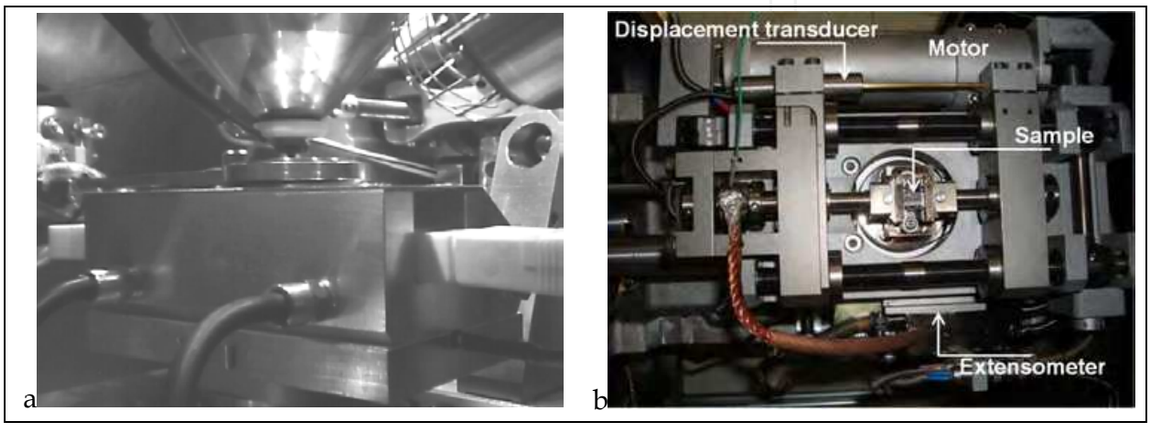

Fig. 1. a) hot stage (FEI) b) Hot tension/compression stage integrated into an SEM (Kammrath & Weiss Co.) (After Biallas & Maier, 2007 ; Gorkaya et al., 2010).

The basis of *in situ* experimentation in the SEM is the study of the morphological modifications of the sample under constraint. Thus, this requires recording of numerous high quality images for image post treatment and data extraction in order to characterize the reaction or matter properties. The sample size can vary from 1µm to 50mm, and the image resolution is in the 1-10nm range, depending on recording conditions. The images are SEM images, i.e. with a large depth of field and with grey level contrasts. In-SEM experimentation can be extended to a wide range of applications, corresponding to very different materials (plants (Stabentheiner et al., 2010), food (Thiel et al., 2002 ; James, 2009), paper (Manero et al., 1998), soft matter, polymers, metals, ceramics, solids, liquids…) or problems (plant behaviour, chemical reactivity, properties characterization, sintering, grain growth, corrosion…). In the literature, the main part of the data reported has been acquired using an environmental scanning electron microscope.

3. *In situ* mechanical tests

Boehlert (2011) have recently underlined the interest of performing *in situ* mechanical tests in the SEM and summarized it as follows. "*In situ* scanning electron microscopy is now being routinely performed around the world to characterize the surface deformation behavior of a wide variety of materials. The types of loading conditions include simple tension, compression, bending, and creep as well as dynamic conditions including cyclic fatigue with dwell times. These experiments can be performed at ambient and elevated

temperatures and in different environments and pressures. Most modern SEMs allow for the adaptation of heating and mechanical testing assemblies to the SEM stage, which allows for tilting and rotation to optimal imaging conditions as well as energy dispersive spectroscopy X-ray capture. Perhaps some of the most useful techniques involve acquisition of electron backscatter diffraction (EBSD) Kikuchi patterns for the identification of crystallographic orientations. Such information allows for the identification of phase transformations and plastic deformation as they relate to the local and global textures and other microstructural features. Understanding the microscale deformation mechanisms is useful for modeling and simulations used to link the microscale to the mesoscale behavior. In turn, simulations require verification through *in situ* microscale observations. Together simulations and *in situ* experimental verification studies are setting the stage for the future of material science, which undoubtedly involves accurate prediction of local and global mechanical properties and deformation behavior given only the processed microstructural condition".

As a direct consequence of the great interest of the collected information, many different works from several scientific domains have been published for long. Thiel & Donald (1998) and Stabentheiner et al. (2010) describe the deformation of plants (carrots and leaves respectively) during room temperature tensile tests performed in the ESEM chamber. Similar tests are also reported with food (Stokes & Donald, 2000) and they are regularly performed on polymers (Poelt et al., 2010; Lin et al., 2011), composites (Schoßig et al., 2011) and metals (Boehlert et al., 2006; Gorkaya et al., 2007). Mechanical tests on metals, alloys and ceramics can also be performed at high temperature (Biallas & Maier, 2007; Chen & Boehlert, 2010). High temperature EDSB, developed by Seward et al. (2002), offers the possibility to observe phase transformations in materials as a function of temperature, as well as the direct visualization of the associated microstructural modifications (Seward et al., 2004).

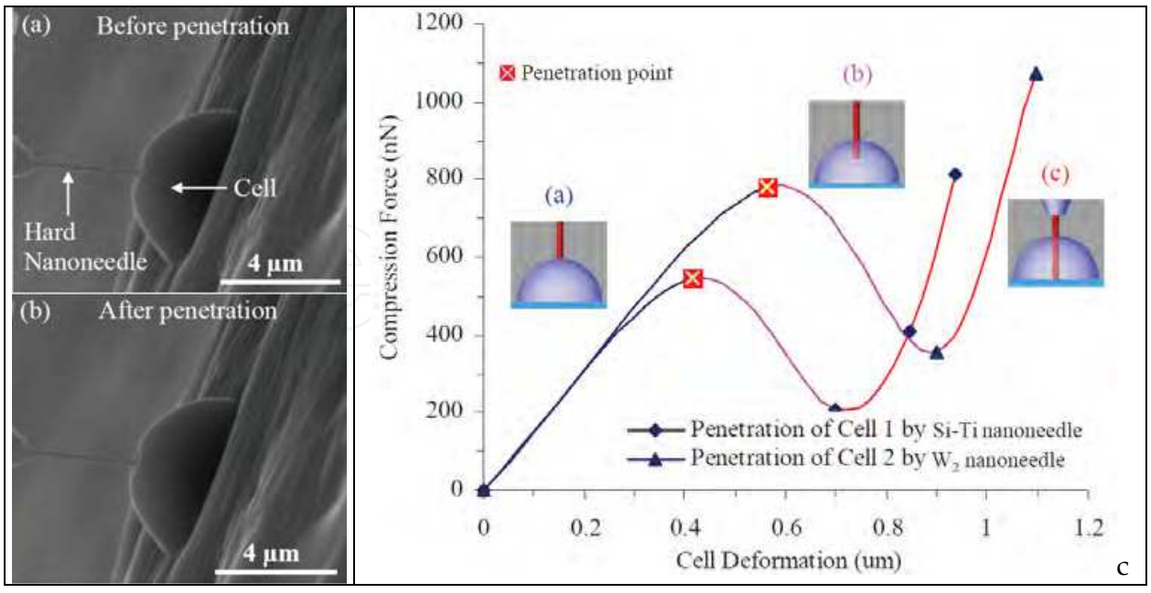

Fig. 2. (a) & (b) Single cell surgery without cell bursting using Si-Ti nanoneedle , (c) Force-cell deformation curve using Ti-Si and W2 nanoneedles at three different stages, i.e. (a) before penetration, (b) after penetration and (c) touching the substrate. (Ahmad et al., 2010).

Several recently developed techniques allow characterizing materials at the nanometer scale through both technological miniaturization and advancements in imaging and small-scale mechanical testing. Ahmad et al. (2010) have developed a coupled ESEM-atomic force microscope to characterize single cells mechanical properties (Figure 2). This ESEM-nanomanipulation system allowed determining effects of internal influences (cell size and growth phases) and external influence (environmental conditions) on the cell strength. Gianola et al. (2011) reports the development of a quantitative *in situ* nanomechanical testing approach adapted to a dualbeam focused ion beam and scanning electron microscope. *In situ* tensile tests on 75 nm diameter Cu nanowhiskers as well as compression tests on nanoporous Au micropillars fabricated using FIB annular milling are reported, the scientific question being the mechanical behaviour of nanosize materials. Both examples probably represent what will be the future of *in situ* mechanical tests using scanning electron microscopes.

4. *In situ* experimentation under wet conditions

4.1 Conditions for experimentation

Combination of the use of the ESEM and a Peltier stage with the development of specific detectors allows the possibility to control both specimen temperature and water pressure around the sample (Leary & Brydson, 2010). Water can be condensed or evaporated on the demand from the sample (Figure 3). This allows performing *in situ* experiments in a temperature-pressure domain that is reported on Figure 3a (dot zone). An easy to perform experiment, illustrated by a 6 images series, corresponding to the NaCl dissolution (during the increasing of the water pressure in the ESEM chamber and consecutive water condensation, at constant temperature) in water followed by the crystallization of NaCl (decrease of the water pressure) is reported on Figure 3b. This example corresponds to an "isothermal experiment". Another ways to work are to perform isobar experiments or to heat or cool a sample using a constant relative humidity (iso-RH experiments). These techniques allow the characterization of structural transitions of hydrated samples as a function of temperature (Bonnefond, 2011).

4.2 Biology and soft matter applications

This technique is particularly well adapted for the observation or experimentation on biological samples (Muscariello et al., 2005). Images of small and highly hydrated samples such as liposomes have been obtained by several authors (Perrie et al., 2007 ; Ruozi et al;, 2011) without any particular sample preparation. Perrie et al. (2007) have also been able to dynamically follow the hydration of lipid films and changes in liposome suspensions as water condenses onto, or evaporates from, the sample in real-time. The data obtained provides an insight into the resistance of liposomes to coalescence during dehydration, thereby providing an alternative assay for liposome formulation and stability (Perrie et al., 2010). However, Kirk et al. (2009) report that ESEM imaging of biological samples must remain combined with the classical techniques for sample preparation. Several works are specifically dedicated to *in situ* experimentation. Stabentheiner et al. (2010) state that "one unrivaled possibility of ESEM is the *in situ* investigation of dynamic processes that are impossible to access with CSEM where samples have to be fixed and processed". These authors have studied the anther opening that is a highly dynamic process involving several

tissue layers and controlled tissue desiccation. This phenomenon can be observed because the sample is very stable under the ESEM conditions (Figure 4). Another recent study is relative to the closure of stomatal pores by Mc Gregor & Donald (2010). Even if the possibility for experimentation on biological samples is clearly demonstrated, the authors outline the fact that the electron beam damages are important even at low accelerating voltage (Zheng et al., 2009). Another surprising example that can be reported is the direct observation of living acarids available online: in the movie, colonies of acarids are directly observed in the ESEM chamber under several conditions (FEI movie).

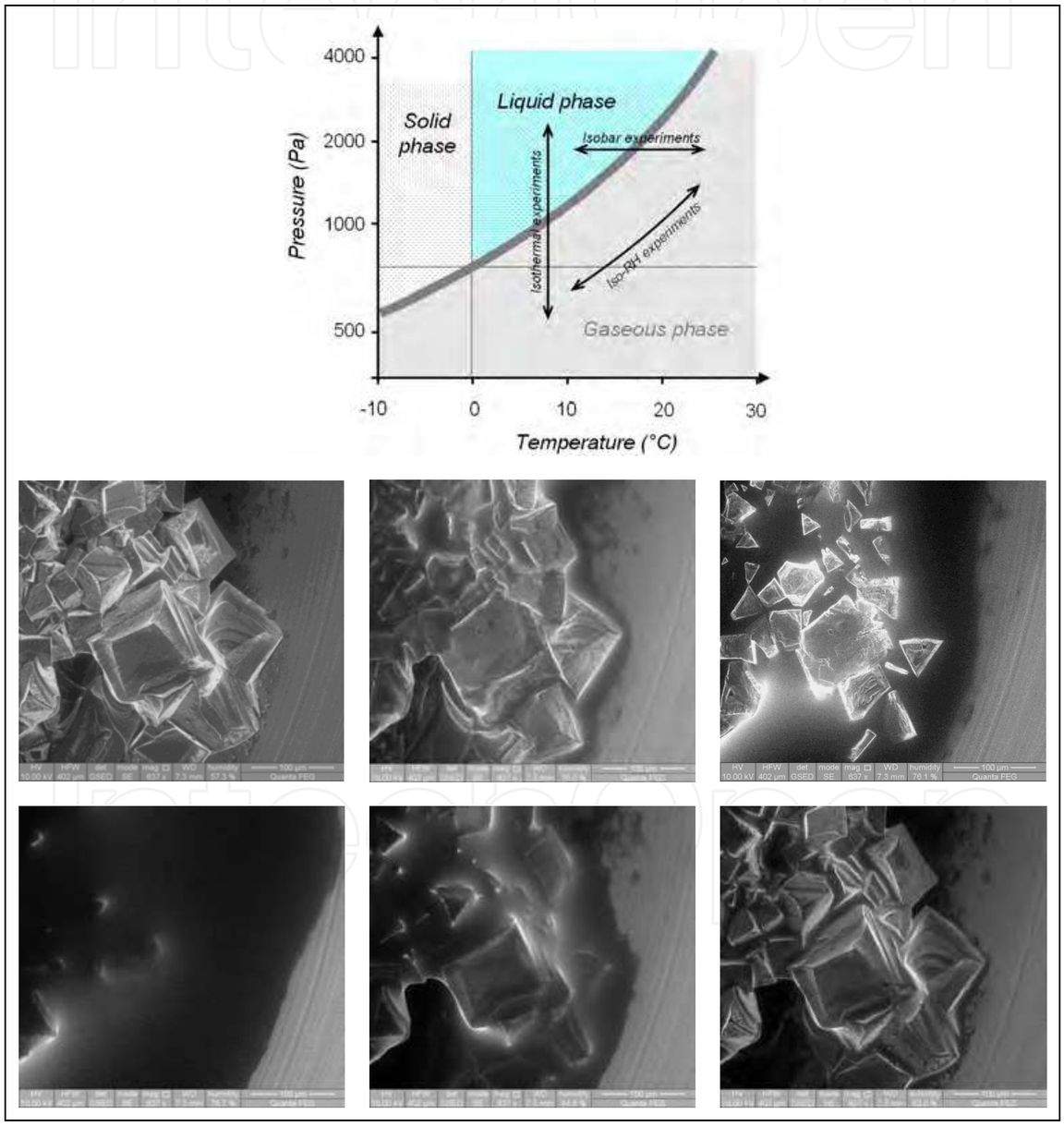

Fig. 3. (a) Simplified phase diagram for water indicating the ESEM domain (dot zone) and schemes to understand how isothermal or isobar experiments are performed.
(b) Solubilisation and crystallization of NaCl directly observed in the ESEM chamber.

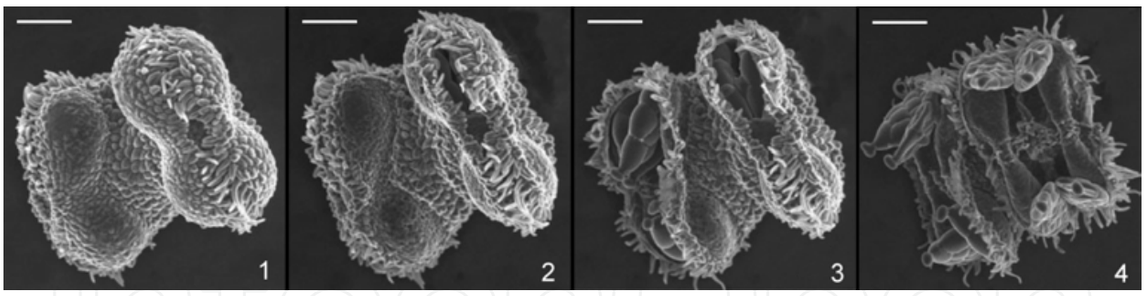

Fig. 4. *In situ* anther opening of C. angustifolia observed in LV-ESEM. 1) At the beginning, the valves of the anther are closed; 2) opening starts at the end of the stomium; 3) polyads are already seen; 4) opening proceeds till the valves are completely bent back and all eight polyads are presented (scale bar = 100μm). Time span from 1) to 3) was 25 min; 4) imaged 1 h after the start of the opening process (after Stabentheiner et al., 2010)

4.3 Applications on cements

Several works have been performed in order to study the reactivity of cement materials versus humidity. Hydration or dehydration (Sorgi & De Gennaro, 2007; Fonseca & Jennings, 2010; Camacho-Bragado et al., 2011) of phases have been followed and used to extract kinetic parameters (Montes-Hernandez, 2002 ; Montes & Swelling, 2005 ; Maison et al., 2009), as reported on Figure 5. In this work, the author uses ESEM image series to determine a three-step mechanism for bentonite aggregates evolution with relative humidity corresponding to an arrangement of particles followed by a particle swelling and a full destructuration. In SEM experiments are also used to characterize chemical reactivity (Camacho-Bragado et al., 2011). It has been recently used to characterize reaction of fly ash activated by sodium silicate by Duchene et al. (2010). These authors have determined very accurately the different steps of the reaction determining that the sodium silicate activator dissolves rapidly and begins to bond fly ash particles. Open porosity was observed and it was rapidly filled with gel as soon as the liquid phase is able to reach the ash particle. The importance of the liquid phase is underlined as a fluid transport medium permitting the activator to reach and react with the fly ash particles. The reaction products had a gel like morphology and no crystallized phase was observed.

4.4 Hydration and dehydration experiments

As previously reported for liposomes, new opportunities for the study of polyelectrolyte microcapsules versus their resistance to relative humidity and temperature modifications are opened and under consideration. The image series reported on Figure 6 clearly illustrate the possibility to image the native soft capsule at high relative humidity without any deformation. When decreasing the water pressure near the capsule, the object is deformed and do not shrink as observed when it is heated in water at temperature higher than 25°C (Basset et al., 2010). Thus, the walls of the object do not rearrange but collapse when submitted to a relative humidity decrease.

Similar tests have been performed on self-organized metal-organic framework compounds (Bonnefond, 2011). According to the image series reported on Figure 7, when the water pressure decreases, the size of sample remains constant up to a given water pressure (i.e. relative humidity) and for a transition pressure, the sample size decreases regularly. This

can be associated to a local reorganisation in the sample that corresponds to a water loss associated to the sample collapsing The enthalpy of water ordering in the sample can be derived from the recorded image series as reported by Sievers et al.

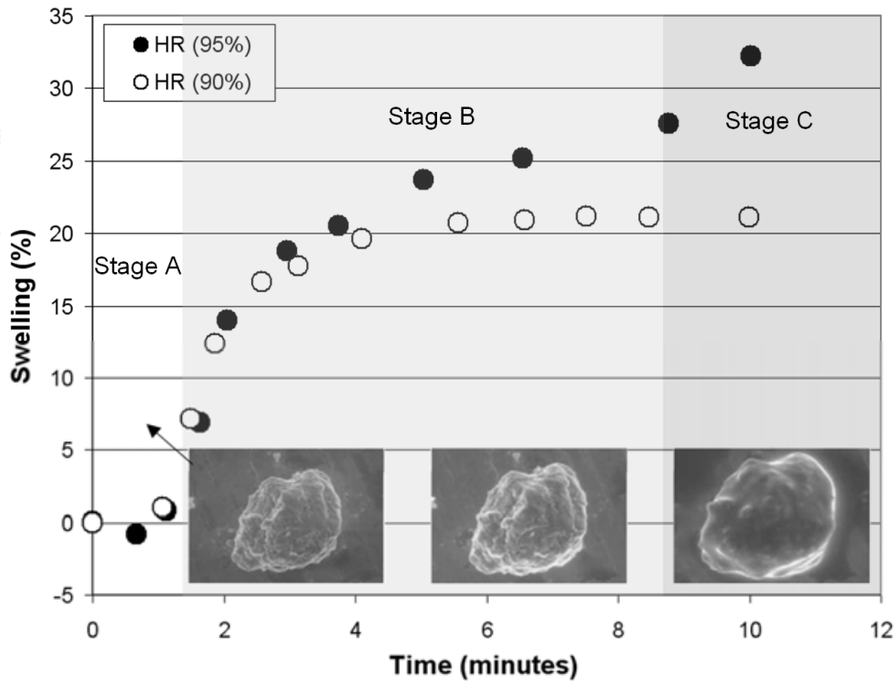

Fig. 5. Swelling kinetics of raw bentonite aggregates scale using ESEM-digital image analyses coupling (after Montes & Swelling, 2005).

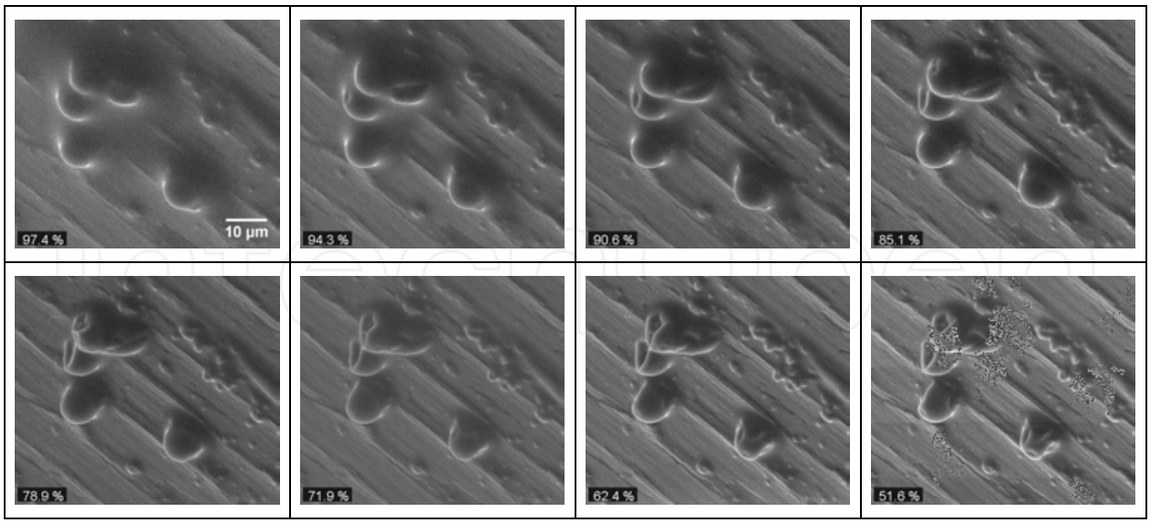

Fig. 6. ESEM micrographs of polyelectrolyte microcapsules suspended in double distilled water. Microcapsules were subjected to controlled dehydration in the ESEM sample chamber at T=5°C. At an operating pressure of 800Pa, vesicles appeared as spherical structures. (a) Gradual decrease of the operating pressure to 350 Pa showed regular deformation of the microcaspsules (b to h)

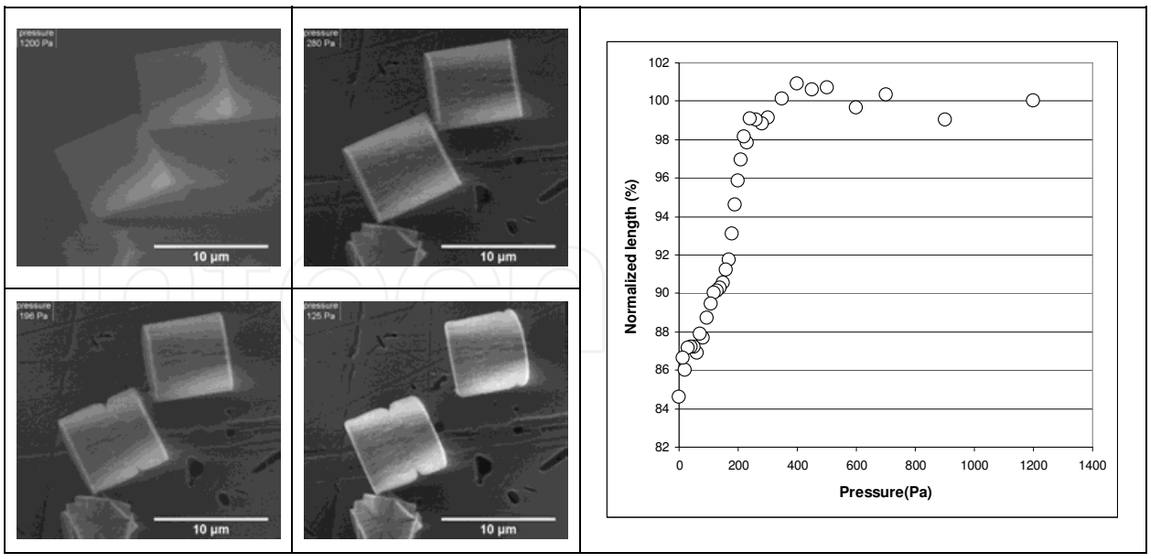

Fig. 7. Dehydration experiments performed on self-assembled organo-metallic compounds at T=22°C and corresponding size modification *versus* water vapour pressure (Bonnefond, 2011).

The effect of dehydration on lamellar bones was also studied by *in situ* ESEM experiments (Utku et al., 2008). The obtained results indicate that dehydration affects the dimensions of lamellar bone in an anisotropic manner in longitudinal sections, whereas in transverse sections the extent of contraction is almost the same in both the radial and tangential directions.

An original work on the heterogeneous ice nucleation on synthetic silver iodide, natural kaolinite and montmorillonite particles has been performed using the "increasing water pressure at constant temperature" (Zimmermann et al., 2007) in the temperature range of 250–270 K. Ice formation was related to the chemical composition of the particles. The obtained data are in very good agreement with previous ones obtained by diffusion chamber measurements (Figure 8).

4.5 Characterization of surface wetting properties

Characterization of the wetting properties of surfaces through the formation of microdroplets or nanodroplets is another important investigation field that can be explored using the ESEM. A recent review by Mendez-Vilas et al. (2009) has highlighted the main fundamental and applied results. Several strategies for the contact angle between water and the surface determination are reported (Stelmashenko et al., 2001; Stokes, 2001; Lau et al., 2003; Wei, 2004; Yu et al., 2006; Jung & Bhushan, 2008; Rykaczewski & Scott, 2011). The investigation of the hydrophobicity and/or hydrophilicity of a catalyst layer have been performed using ESEM for the first time by Yu et al. (2006). These authors have determined the micro-contact angle distribution as a function of the catalyst microstructure. Microdroplets growing and merging process was observed directly in the ESEM chamber by Lau et al. (2003).

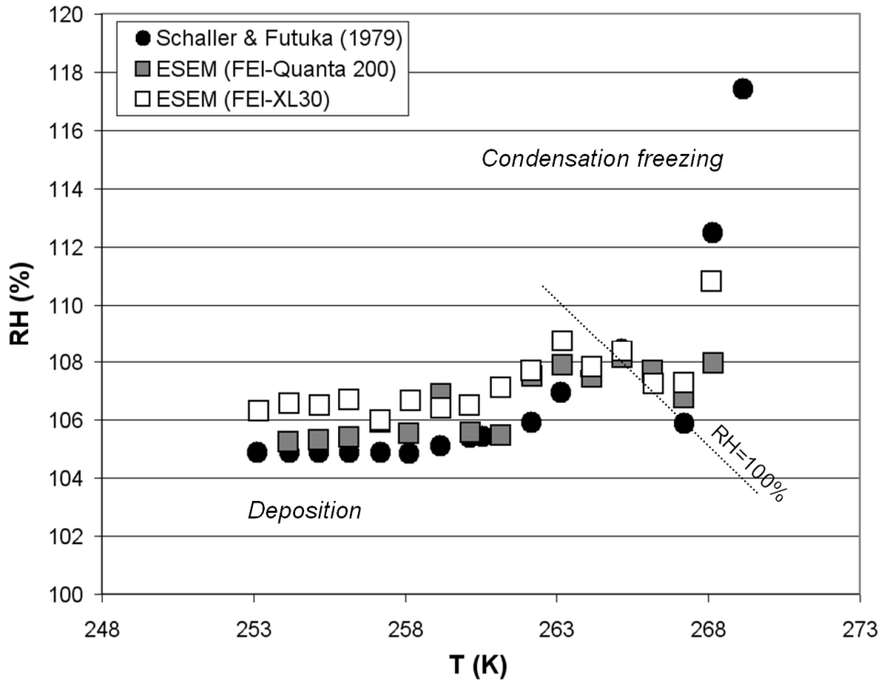

Fig. 8. Supersaturation *versus* temperature diagram for silver iodide (After Zimmermann et al., 2007).

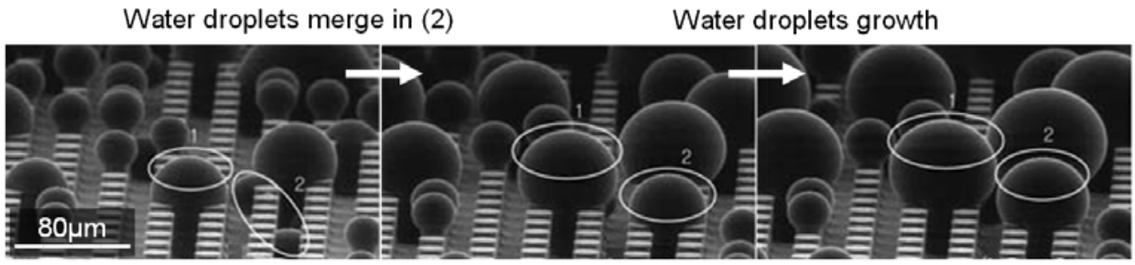

Fig. 9. Microdroplets growing and merging process under ESEM during increasing condensation by decreasing temperature. (After Jung & Bhushan, 2008)

4.6 Using the Wet-STEM mode

The development of the Wet-STEM by Bogner et al. (2005, 2007) allows observing samples in the transmission mode in the ESEM chamber, and more particularly, it offers the possibility to image directly nanoparticles dispersed in a few micrometer thin water film (Bogner et al., 2008), emulsions or vesicles (Maraloiu et al., 2010), without removing the liquid surrounding the objects of interest. One must keep in mind that images with soft matter, and more generally sample sensitive to the electron beam are very hard to obtain. Nevertheless, this technique also opens new research fields using *in situ* experimentation that only begin to be explored for wettability or deliquescence studies. By combining Wet-STEM imaging with Monte-Carlo simulation (Figure 10), Barkay (2010) have studied the initial stages of water nanodroplet condensation over a nonhomogeneous holey thin film. This study has shown a preferred water droplet condensation over the residual water film

areas in the holes and has provided corresponding droplet shape and contact angle. On a similar way, Wise et al. (2008) have studied water uptake by NaCl particles prior to deliquescence by varying the relative humidity in the Wet-STEM environment (Figure 11).

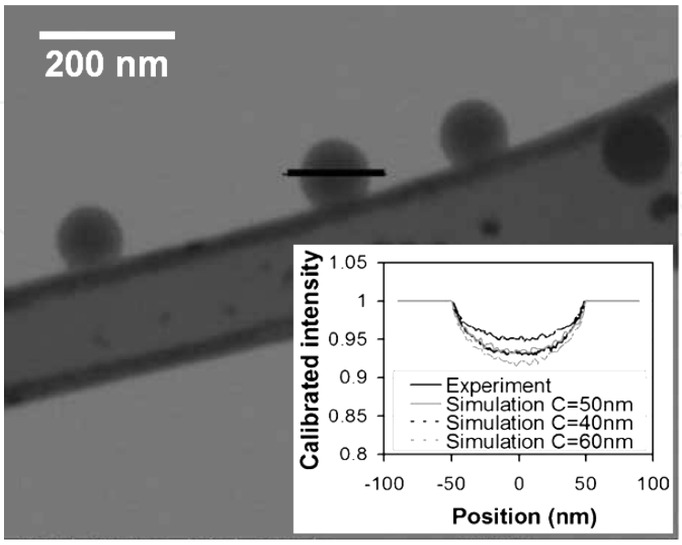

Fig. 10. Bright field image of 100 nm polystyrene latex spheres. Insert is the calibrated intensity corresponding to the dark line in the image (After Barkay (2010))

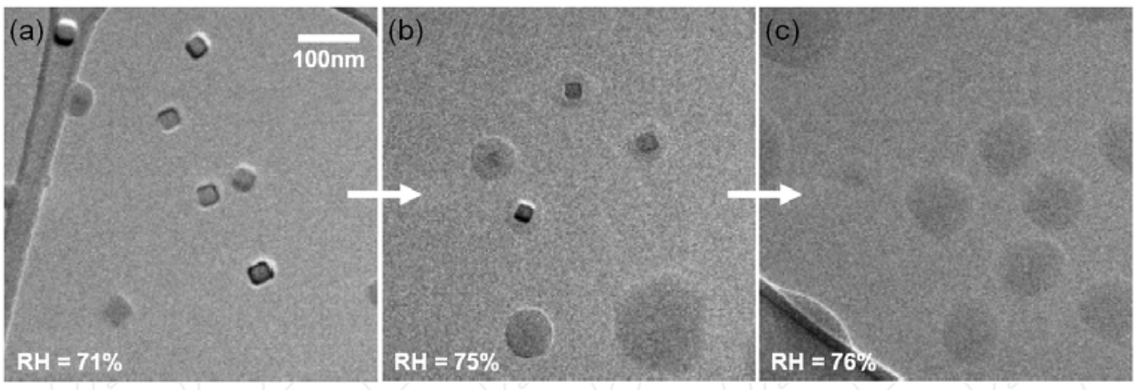

Fig. 11. ~40 nm NaCl particles as the RH was increased past the deliquescence point. Water uptake [(a) → (b)] prior to full deliquescence (c) is clearly observed. (After Wise et al., 2008)

4.7 Development of specific materials for experimentation

Several specific devices have been developed to characterize specific properties or reactions. Two of them will be shortly described below.

Chen et al. (2011) have developed an experimental platform that can be used to investigate chemical reaction pathways, to monitor phase changes in electrodes or to investigate degradation effects in batteries. They have performed *in situ* experiment runs inside a scanning electron microscope (SEM) and tracked the morphology of an electrode including active and passive materials in real time. This work has been used to observe SnO_2 during lithium uptake and release inside a working battery electrode.

Direct imaging of micro ink jets inside the ESEM chamber has been achieved using a specific device developed by Deponte et al. (2009), using a two-fluid stream consisting of a water inner core and a co-flowing outer gas sheath. ESEM images of water jets down to 700 nm diameter have been recorded. Details of the jet structure (the point of jet breakup, size and shape of the jet cone) can be measured. The authors conclude that ESEM imaging of liquid jets offers a valuable research tool for the study of aerosol production, combustion processes, ink-jet generation, and many other attributes of micro- and nanojet systems.

5. High temperature in the SEM

5.1 Application domains of HT-(E)SEM

Specific stages (and associated detectors) have been developed to heat samples up to 1500°C directly in the microscope chamber (Knowles & Evans, 1997; Gregori et al., 2001). The environmental scanning electron microscope (ESEM) equipped with this heating stage is an excellent tool for the *in situ* and continuous observation of system modifications involved by temperature. It allows recording image series of the morphological changes of a sample during a heat treatment with both high magnification and high depth of focus. The experiments can be carried out to observe the influence of all these parameters on the studied phenomenon under various conditions (heating rates, atmosphere compositions, variable pressure, final temperature and heating time). Images have been recorded up to 1400°C, with a decrease of the image resolution when the sample temperature increases (Podor et al., 2012). It is possible to work under vacuum (classical SEM) or under controlled atmosphere (H_2O, O_2, $He+H_2$, N_2, air...). Different types of studies have been reported, relative to corrosion of metals (Jonsson et al., 2011), oxidation of metals (Schmid et al., 2001a, 2001b ; Oquab & Monceau, 2001 ; Schmid et al., 2002 ; Abolhassani et al., 2003 ; Reichmann et al., 2008 ; Jonsson et al., 2009 ; Mège-Revil et al., 2009 ; Quémarda et al., 2009 ; Delehouzé et al., 2011), reactivity at high temperature (Maroni et al., 1999 ; Boucetta et al., 2010), phase changes (Fischer et al., 2004 ; Hung et al., 2007 ; Beattie & McGrady, 2009), hydrogen desorption (Beattie et al., 2009, 2011), redox reactions (Klemensø et al., 2006), microstructural modifications (Bestmann et al., 2005 ; Fielden, 2005 ; Yang, 2010), magnetic properties (Reichmann et al., 2011), sintering (Sample et al., 1996 ; Srinivasan, 2002 ; Marzagui & Cutard, 2004 ; Smith et al., 2006 ; Subramaniam, 2006 ; Courtois et al., 2011 ; Joly-Pottuz et al., 2011 ; Podor et al., 2012), thermal decomposition (Gualtieri et al., 2008 ; Claparède et al., 2011 ; Goodrich & Lattimer, 2011 ; Hingant et al., 2011), crystallisation (Gomez et al., 2009) in melts (Imaizumi et al., 2003 ; Hillers et al., 2007) and study of self-repairing – self-healing – properties of materials (Wilson & Case, 1997 ; Coillot et al., 2010a, 2010b, 2011) …

Even if numerous researchers are invested in HT-ESEM, only few of them have been successful in pursuing dynamic experiments at temperatures higher than 1100°C. Two recent studies report experiments performed at T=1350°C (Subramaniam, 2005) and 1450°C (Gregori et al., 2002). However, the resolution of the images remains poor (more than 1µm) mainly due to water cooling induced vibrations. Furthermore, the precision on the measure of the sample temperature remains poor (temperature differences up to 150°C with the expected temperature are sometimes measured). A recent device has been proposed by Podor et al. (2011) to overcome this difficulty.

A complete review specifically dedicated to *in situ* high temperature experimentation in the ESEM will be available soon. Several examples of *in situ* studies performed at high

temperature in the ESEM chamber will be reported below, on the basis of original data acquired in our laboratory.

5.2 Investigation of the crystallization behaviour in silicate melts

The crystal growth and morphology during isothermal heating of glass melts can be directly observed using the hot stage associated with the ESEM. The image series reported on Figure 12 have been recorded during 10 minutes while heating the borosilicate melt sample isothermally at T=740°C. The development of large crystals in the melt rapidly yields to the complete crystallization of the melt. The crystal morphology presents cells filled with a second phase and the crystal formation yields to the deformation of the sample surface. Hillers et al. (2007) have used such data to quantify the variation of crystal length with time. They have established that the growth is only linear during the first minutes; afterward the growth rate decreases progressively with time.

This technique can also be used to determine the temperature of formation of the first crystals at the melt surface and to observe their formation. In the case of glass-ceramics, the density of nuclei as well as their size and shape development can be directly observed and used for crystallization kinetic determination (Vigouroux et al., 2011, in prep).

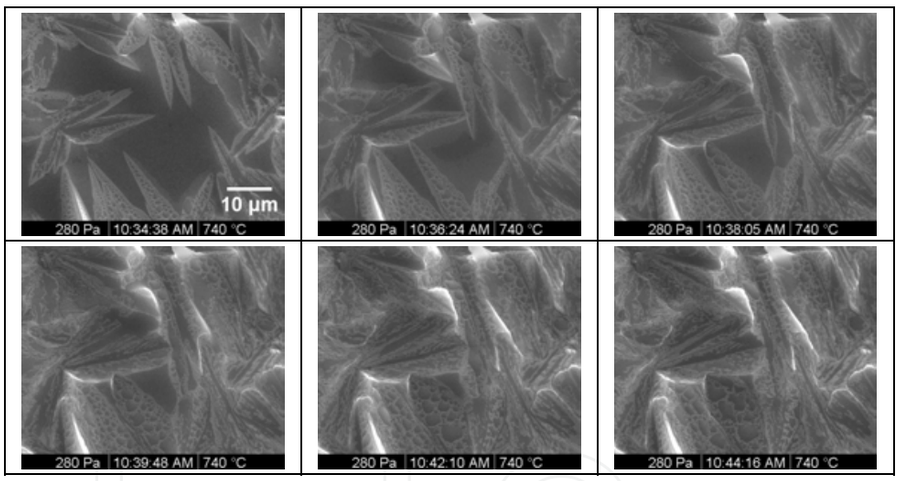

Fig. 12. Growth of crystals in a borosilicate melt during 10 minutes isothermal heat treatment at 740°C observed using the hot stage associated with the ESEM.

5.3 Decomposition of compounds

In situ thermal decomposition of composites, oxalates, oxides have been reported by several authors. Images of the heat treatment of a mixed uranium-cerium oxalate grain from 25°C to 1235°C are gathered on Figure 13. Morphological changes with temperature are directly linked with the oxalate decomposition as stated by Hingant et al. (2011) in the temperature range 25-500°C. The sample shrinkage observed when T>500°C is probably related with the first stage of the sintering process – i.e. beginning of bond formation between the nanograins and with the oxide grain growth (that can not be directly observed at this stage by HT-ESEM, but that is confirmed by X-Ray diffraction). Such a process has also been recently reported by Claparede et al. (2011) and Joly-Pottuz et al. (2011).

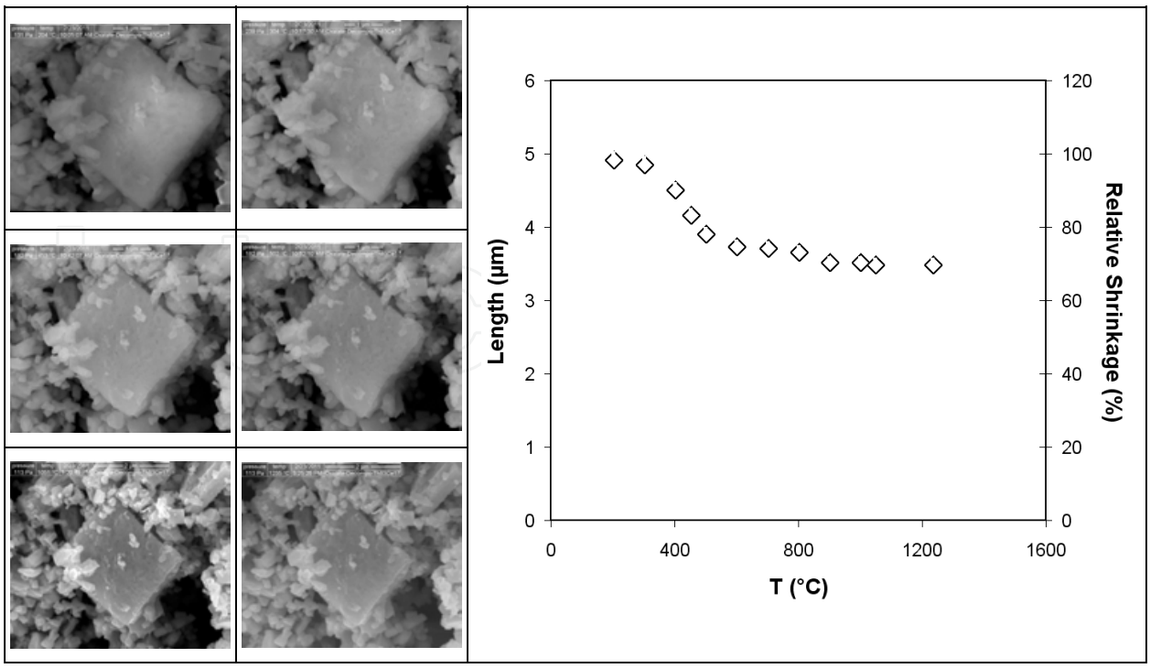

Fig. 13. Decomposition of a uranium-cerium mixed oxalate observed during *in situ* heating in the ESEM chamber and relative size and shrinkage modifications.

5.4 Study of sintering and grain growth

Several studies are relative to the sintering and grain growth processes in metals and ceramics. Depending on the system, the experiments have been performed in the temperature range 300-1450°C. The main interest of these studies is the possibility of direct observation of the individual grain behaviour during heat treatment. The example that is reported on Figure 14a corresponds to the heat treatment of the grain decomposed *in situ* (Figure 13). The image resolution is high enough to observe the nanograins growth inside the square plate agglomerate. Consequently, relative shrinkage and average grain diameter are extracted by image processing (Figure 14b). Assuming that the final density of the agglomerate is 99%, the sintering map is directly derived from these experimental data (Figure 14c). Thus, *in situ* sintering experiments can allow the establishment of the trajectories of theoretical sintering. Such data have never been already reported in previous studies, mainly due to the poor resolution of the recorded images.

The effect of the electron beam on sintering is controversy. Indeed, Popma (2002) noted that a local sintering stop was achieved by focusing the electron beam at a certain position during the *in situ* sintering experiments in the ESEM (performed on ZrO_2 nanolayers). On the contrary, Courtois et al (2011) performed experiments on the sintering of a lead phosphovanadate and concluded that the electric current induced by the electron beam was found to reduce the effective temperature of sintering by 50 to 150°C as well as to accelerate the kinetics of shrinkage of a cluster composed of sub-micrometric grains of material. Such effects were not evidenced in our study: the local sintering on sample surface zones that were not observed (i.e. exposed to the electron beam) was identical to the local sintering determined on the observed zone.

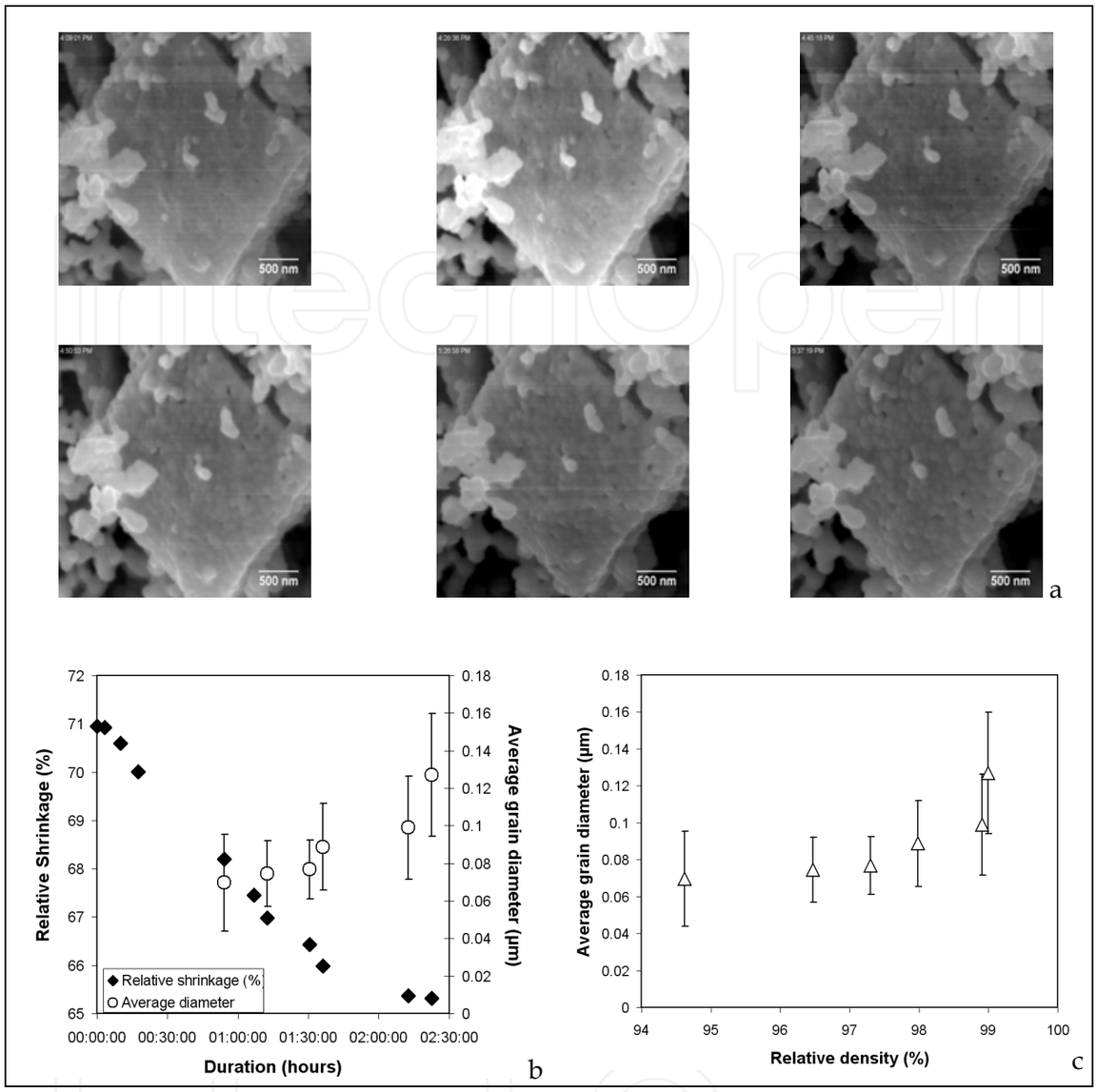

Fig. 14. (a) Sintering and grain growth of a uranium-cerium mixed oxide observed *in situ* in the ESEM chamber at T=1235°C, after 55', 70', 90', 95', 130', 140' (a). Corresponding Relative (b) Shrinkage and Average grain diameter versus duration and (c) derived sintering map - Grain growth versus densification rate –

6. Conclusions and perspectives

In situ scanning electron microscopy experimentation, that is generally associated with the use of the ESEM, allows the study of very different problems, the main limit being the availability of specific devices. Torres & Ramirez (2011) have written the best conclusion indicating that "the new generation of SEMs shows innovative hardware and software solutions that result in improved performance. This progress has turned the SEM into an extraordinary tool to develop more complex and realistic *in situ* experiments, achieving even at the subnanometer scale". In the near future, new SEM imaging modes, nanomanipulation

and nanofabrication technologies (Miller & Russell, 2007 ; Romano-Rodriguez & Hernandez-Ramirez, 2007 ; Wich et al., 2011) will make possible to replicate more closely the conditions as the ones associated to the problems to be treated. *In situ* ESEM will probably be used to overcome technical and fundamental challenges in many scientific domains. The recent developments of a high temperature stage in the FIB (Fielden, 2008), a new tomography mode in the ESEM (Jornsanoh et al., 2011) and of the atmospheric scanning electron microscope (Nishiyama et al, 2010 ; Suga et al, 2011) can be cited as examples for this future.

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8. References for videos

Reactivity of a salt with silicate melt at high temperature	http://www.dailymotion.com/icsmweb#videoId=xjknrt
Sintering of CeO_2 at T=1200°C	http://www.youtube.com/watch?v=4ijIUdQe3M4
Self-healing of a metal-glass composite at high temperature	http://www.dailymotion.com/icsmweb#videoId=xjknpp
Deformation of vesicles during dehydration	http://www.dailymotion.com/icsmweb#videoId=xjk75u
NaCl solubility and precipitation in water	http://www.dailymotion.com/icsmweb#videoId=xk22i9

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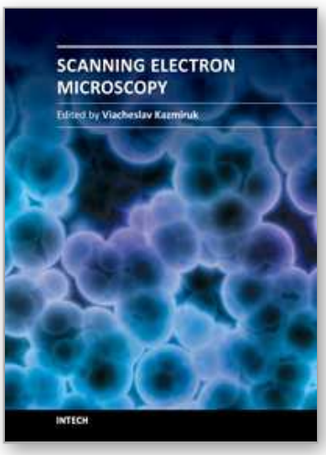

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Today, an individual would be hard-pressed to find any science field that does not employ methods and instruments based on the use of fine focused electron and ion beams. Well instrumented and supplemented with advanced methods and techniques, SEMs provide possibilities not only of surface imaging but quantitative measurement of object topologies, local electrophysical characteristics of semiconductor structures and performing elemental analysis. Moreover, a fine focused e-beam is widely used for the creation of micro and nanostructures. The book's approach covers both theoretical and practical issues related to scanning electron microscopy. The book has 41 chapters, divided into six sections: Instrumentation, Methodology, Biology, Medicine, Material Science, Nanostructured Materials for Electronic Industry, Thin Films, Membranes, Ceramic, Geoscience, and Mineralogy. Each chapter, written by different authors, is a complete work which presupposes that readers have some background knowledge on the subject.

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