A Combination of ESPI, microscopic ESPI and Digital Speckle-Correlation to study salt-induced Deterioration in porous Materials

Akram El Jarad, Gerd Gülker, Klaus D. Hinsch

Carl von Ossietzky University Oldenburg

mailto:gerd.guelker@uni-oldenburg.de

Salt-induced deterioration of fritted glass substrates due to crystallization or hydration pressure was studied by detecting deformations and surface micro-structure changes with ESPI. The results from these full-field measurements were not expected theoretically and give new insight into the underlying processes.

1 Introduction

Although still poorly understood, crystal growth of salts in historic monuments and objects of art is known to be a major cause of deterioration. In order to provide a better understanding the detection of deformations in porous materials due to crystallization and hydration pressure is of elementary interest. Hitherto, mainly measurements were carried out to determine merely the integral expansion of porous samples. It was found that the use of Electronic Speckle Pattern Interferometry (ESPI) combined with speckle correlation analysis can yield much more relevant information.

2 Porous substrate and salt

In our experiments fritted glass (Schott pore class 4) was used as porous substrate. It has a simple chemical composition and shows a negligible humidity expansion coefficient.

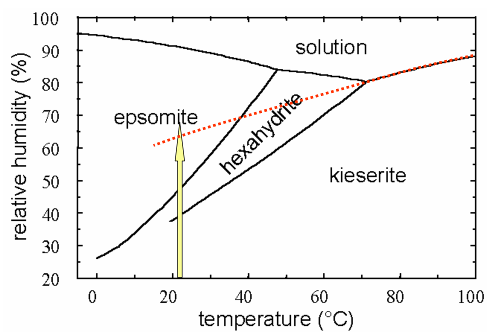

Fig. 1 *Temperature–humidity diagram of the $MgSO_4 \cdot H_2O$ system*

As damaging salt $MgSO_4$ is used which is known to be deleterious in salt crystallization tests and is subject to a number of different salt-phase changes depending on temperature and relative humidity (RH) as can be seen in Fig 1.

3 Experimental setup

To detect deformations and object surface changes a typical ESPI-setup with spatial phase shifting is used (Fig. 2). A Nd:YAG-Laser at 532 nm illuminates the specimen with a collimated beam. The area under investigation is imaged onto a CCD sensor (Sony XC 75). In cases where a high magnification is needed, e.g. for the investigation of object areas around single pores [1], [2], a microscope objective (Mitutoyo M Plan APO 20) is used. This leads to a lateral resolution down to just a few microns. The reference beam is guided by an optical fiber. To perform spatial phase shifting the end of the fiber is placed off-axis in the plane of the limiting aperture [3].

The specimen is conditioned in a climatic chamber, where the temperature and the RH of the ambient air can be controlled by the computer.

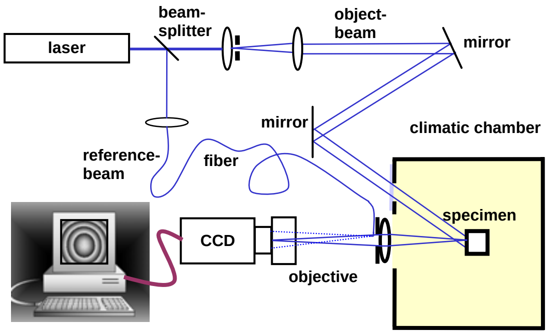

Fig. 2 *Scheme of used ESPI-setup*

4 Experimental results

In our experiments small cubes of fritted glass with dimensions of about 1 cm^3 were soaked until saturation with 20% $MgSO_4$-solution and afterwards dried for 21 days at 200° C to obtain kieserite. To avoid speckle de-correlations caused by scattered light from the bulk material the surface of the specimen is coated with a thin gold-layer.

In a first experiment a contaminated specimen was placed in the climatic chamber at room temperature and low RH of about 20 %. While increasing the humidity in one step to 65% (vertical arrow in Fig.1) ESPI images are recorded. In Fig. 3 two ESPI phase maps are shown about 30 (left) and

60 (right) minutes after reaching 65% RH. A de-correlated area with increasing dimension is clearly visible in the right part of the two images. This highly de-correlated region indicates strong surface changes, apparently liquid on the surface. This assumption could be verified by taking a probe of the specimen and investigating it by cryogenic SEM. The formation of a solution on the surface due to this humidity change was not expected theoretically, as can be deduced from Fig. 1. It shows, that the thermodynamic model, which is the basis of Fig. 1 has to be revised or adapted.

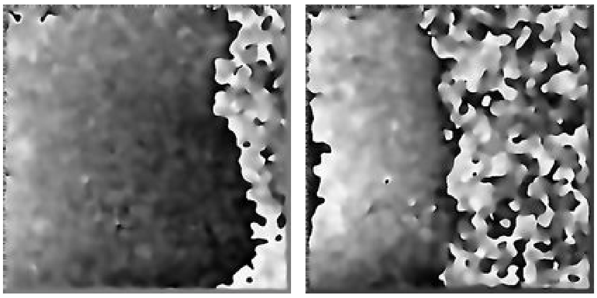

Fig. 3 *ESPI phase maps identify surface changes: (left) about 30 and (right) 60 min. after reaching 65% RH.*

By changing the order of sample preparation and using a platinum coating the surface of the specimen could be made hydrophobic. With this procedure speckle de-correlations were nearly completely avoided and deformation measurements could be performed. In this experiment the RH was increased stepwise from nearly 0% to about 80% (Fig.5). As a first result we saw that occurring deformations were not homogeneous, which is illustrated in Fig. 4 (left), where an ESPI phase map is shown representing the deformations during a time period of about 70 minutes at 70% RH. It can be seen that the edges of the specimen deform stronger than the central part. SEM investigations showed that a concentration of salt in the outer regions of the specimen was responsible for this unexpected behavior (Fig. 4 right).

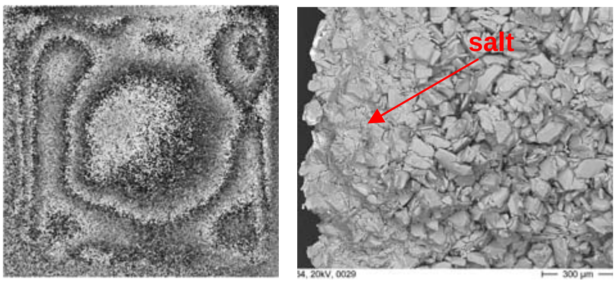

Fig. 4 *ESPI phase map (left) showing inhomogeneous deformation; SEM micro-graph (right) identifies pores filled completely with salt at the edges.*

Secondly, the local strain of the center of the specimen was evaluated by temporal phase unwrapping [4]. In Fig. 5 the measured strain is plotted vs. time and RH. It can be seen that the strain rate increases suddenly at about 60% RH., which is the deliquescence RH of kieserite at about 20°C (see Fig. 1, red dotted line). In contrast to theoretical predictions, these results again prove, that the direct transition from kieserite to hexahydrite is inhibited. Instead, kieserite firstly dissolves and recrystallizes immediately to epsomite, leading to pronounced dilatations.

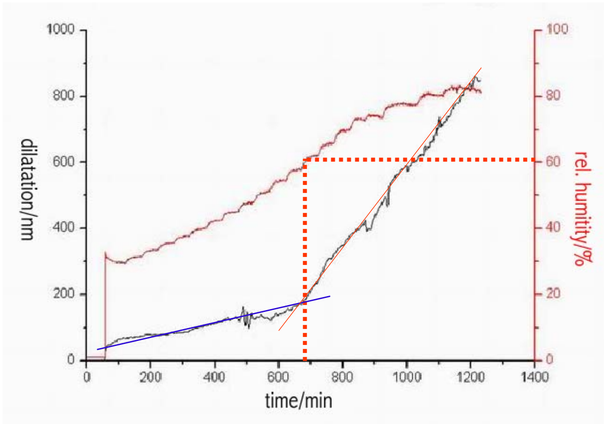

Fig. 5 *Local strain of the center of the specimen and RH vs. measuring time.*

5 Conclusions

An ESPI-setup for detecting surface changes, deformations and strain was presented. It was shown that unexpected results were obtained suggesting a necessary adaptation of the basic thermodynamic model. The investigations proved that full-field information gained by ESPI measurements could lead to new insights in salt-weathering processes, which hardly could be obtained by the usually used tactile dilatation measurement.

6 Acknowledgement

Financial support of this research by the Deutsche Forschungsgemeinschaft (DFG) is gratefully acknowledged. The work was carried out in close cooperation with Institut für Werkstofftechnik, Bremen; Deutsches Bergbau-Museum Bochum; and Institut für Anorganische und Angewandte Chemie, Universität Hamburg. Their contributions are also gratefully acknowledged.

References

[1] A. El Jarad, G. Gülker, K. D. Hinsch: „Microscopic ESPI: Better fringe quality by the Fourier transform method“ in Proc. SPIE 4933: 335-341 (2003)

[2] G. Gülker, K. D. Hinsch, A. El Jarad, „Monitoring of salt-induced deformations in porous systems by microscopic speckle pattern interferometry", Proc. Stone 2004, Vol. II: 619-626, Stockholm (2004)

[3] T. Bothe, J. Burke, H. Helmers, „Spatial phase shifting in ESPI: minimization of phase reconstruction errors", Appl. Opt., 36.22: 5310-5316 (1997)

[4] J. Huntley, H. Saldner, „Temporal phase-unwrapping algorithm for automated interferogram analysis", Appl. Opt. 32.17: 3047-3052 (1993)