

# In situ indentation of dental composite materials coupling micro computed tomography and digital volume correlation

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## Introduction

Tooth decay is the most widely spread pathology in the world. An increase in the prevalence of dental caries has been recently reported [1] and dental restorations have limited longevity: a dentist spends as much time fixing defective restorations as dealing with initial tooth decay lesions. The biological and economic consequences are dramatic. When a patient gets involved in a repairing care cycle, he gets into a spiral which can lead to a weakening of the tooth and to heavier therapeutics. It has been evaluated to cost about five billion dollars per year in the US [2].

This project aims at developing experimental and numerical tools that could be of great help for composite material manufacturers to optimize their materials. One of the weakest points of current composites lays in the polymerization shrinkage which is typical of dimethacrylate resins [3]. In order to predict the final properties of the restored teeth, we believe that the solution lies partly in a coupled experimental and numerical approach, based on in-situ measurements during polymerization and a multiscale numerical model of the restoration stage (at both composite and tooth scales).

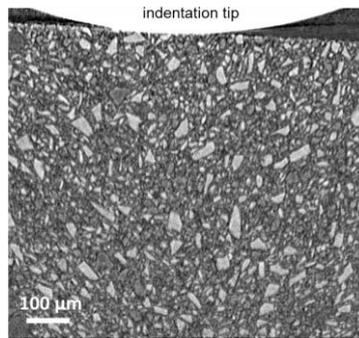
The aim of the current work was to couple micro computed tomography ( $\mu$ -CT) and digital volume correlation (DVC) techniques to determine how a dental composite deforms while submitted to an indentation test and to evaluate the influence of different irradiation sources on regional shrinkage.

## Materials and methods

**Materials.** Light-cure resin core materials commercially available were used as samples. In particular, specimens (CLEARFIL™ PHOTO CORE, Kuraray Medical, Tokyo, Japan) consist in a methacrylate matrix (Bis-GMA and TEG-DMA) with the addition of reinforcing fillers (high filler content of about 65 vol.% and 83 weight%, mainly silica and barium glasses, with diameter between 0.49 and 75  $\mu$ m).

**Influence of irradiation sources on shrinkage.** Because of the small size of the observed particles and of the short duration of the studied phenomena (polymerization time lower than 40 s), in situ tests have been carried out using a synchrotron source. Experiments were performed at the Swiss Light Source (SLS, Paul Scherer Institute, Villigen, Switzerland). A cylindrical sample ( $\Phi$  3 mm, height  $\approx$ 3 mm) of dental composite was light-cured following manufacturer's recommendations and placed between the beam and the detector. In order to assess the influence of light-curing conditions on composite shrinkage, two different tests were performed, by putting transparent or black tapes around the sample before curing. Dental composite samples were photo-polymerized during 40 seconds continuous exposure to a commercial LED blue-light-curing unit located above them, irradiation taking place from the top downwards. Samples were scanned before and after the curing stage. Image analyses coupled with DVC technique (applied on a cubic volume included in the sample) was used to determine sample shrinkage in both cases. Additional experiments were carried out for checking the effect of the distance between the lamp and the composite, in the case of a black tape.

**Indentation tests.** A homemade micro-indentor device with a spherical indentation tip (radius 1 mm) was used to apply several loadings (ranging from 25 N to 360 N) at the top of polymerized samples. Each loading step was followed by a quick relaxation time during which image stacks were recorded (Fig. 1). The DVC technique presented above allowed to obtain displacement and deformation fields of the composite.



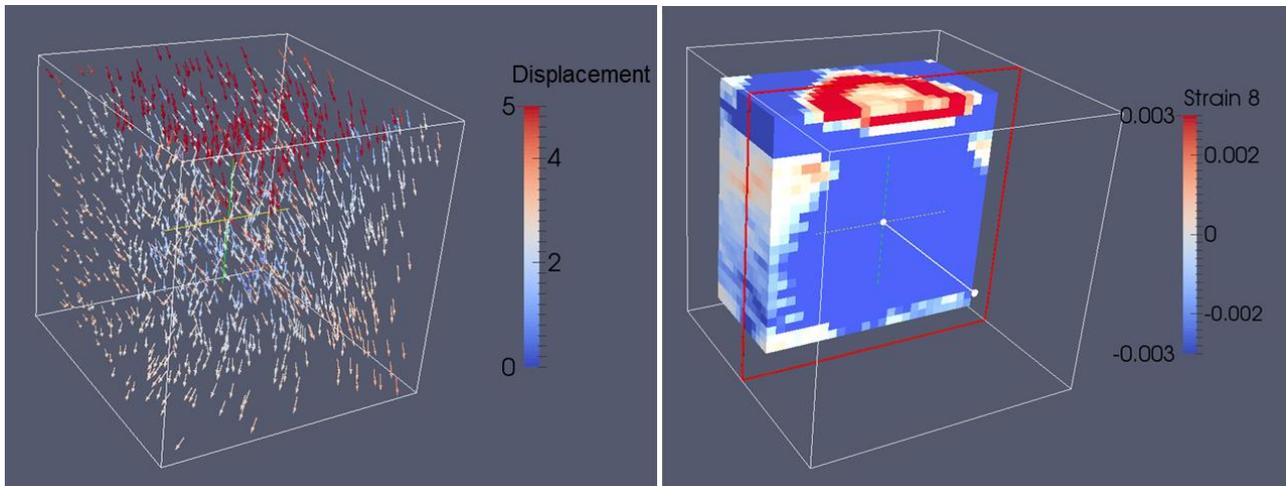
**Fig. 1 - Picture of the dental composite with fillers under the indentation tip**

## Results and discussion

Regarding the shrinkage associated to the curing stage, DVC results correspond to what we could expect. Since the light source is wider than the sample, it is much more irradiated when transparent tape is used. In both cases, we have a contraction of volume but it is slightly higher in terms of intensity in the case of transparent tape.

The results are a little more surprising, even counter-intuitive, when we look at the effect of the distance between the light-curing unit and the composite. The intensity of the displacement is higher when the lamp is far from the sample. This could be explained by the fact that the surface layer further reacts when the lamp is close and reduces the efficiency of the light rays in the deepest layers. Additional tests must be carried out using micro-tomography in order to try to capture what occurs during the first seconds of reaction and thus visualize the displacement fields (at the surface and in the depth) as a function of the distance from the lamp.

Regarding indentation tests, Fig. 2 shows the displacement (including rigid body motion) and strain fields deduced from the DVC analysis in the case of the maximum loading applied by the indentation tip. This technique clearly allows tracking the relative displacement of the fillers while the sample deforms.



**Fig. 2 - Displacement in pixels (left) and incremental strain  $\epsilon_{zz}$  (right) fields at 360 N deduced from DVC**

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## References

- [1] Bagramian, R. A., Garcia-Godoy, F., and Volpe, A. R., 2009, "The Global Increase in Dental Caries. A Pending Public Health Crisis," *Am. J. Dent.*, **22**(1), pp. 3–8.
- [2] Jokstad, A., Bayne, S., Blunck, U., Tyas, M., and Wilson, N., 2001, "Quality of Dental Restorations. FDI Commission Project 2-95," *Int. Dent. J.*, **51**(3), pp. 117–158.
- [3] Karthick, K., Kailasam, S., Geetha Priya, P., and Shankar, S., 2011, "Polymerization Shrinkage of Composites - a Review," *J. Indian Acad. Dent. Spec.*, **2**(2), pp. 32–36.