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Event: SPIE Optical Engineering + Applications, 2019, San Diego, California, United States

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Three-dimensional characterization of soft silicone elements for intraoral devices

Jeannette von Jackowski[*a], Georg Schulz[a], Bekim Osmani[a], Tino Töpper[a], and Bert Müller[*a]
[a]Biomaterials Science Center, Department of Biomedical Engineering, Medical Faculty, University of Basel, 4123 Allschwil, Switzerland

ABSTRACT

Hard X-rays tomography enables us to determine the shape and inner microstructure of a wide variety of solid states and their combinations. Biological samples, and in particular soft tissues from animals and humans, can be encapsulated in suitable containers to avoid deformations during the rotation and radiograph acquisition. The quantitative characterization of soft silicone objects, however, is challenging, because their shape depends on the orientation with respect to the gravitation force. Solid supports may help, but have some impact on the more or less complex-shaped silicone part. In medicine, silicone implants and devices play a major role, since the mechanical properties can be easily tailored. Using the nanotom® m (phoenix|x-ray, GE Sensing & Inspection Technologies GmbH, Wunstorf, Germany), we measured the X-ray absorption of silicone with pre-defined softness. The density resolution of the CT-system was insufficient to establish a density-stiffness relationship. In addition, the shape of a silicone part of a soft intraoral device was measured for three angles with respect to the gravitation force, for which the bending was obvious. These successful tomographic measurements will become the basis of finite element modeling to extract the mechanical properties including their local variations.

Keywords: Hard X-ray computed tomography, silicone, gravity-induced shape, soft oral device, nanotom m, density-stiffness relation

1. INTRODUCTION

Silicone rubbers have become increasingly often applied in medicine, although they have severe limitations concerning tear strength and resistance to fatigue [1]. The spectrum of applications is widespread, because the elastic properties can be easily tailored [2]. Because the functionality and the comfort of intraoral devices crucially depend on the mechanical properties, silicones became the main component of such aids, as illustrated by the left image in Figure 1.

(a) (b)

Fig. 1. A patient-specific device made out of silicone with integrated sensors can be used for tongue training, see (a). For the current study, a single-component, planar element with certain similarity was manufactured.

*bert.mueller@unibas.ch; phone +41 61 207 5430; fax 41 61 207 5499; www.bmc.unibas.ch

Developments in X-Ray Tomography XII, edited by Bert Müller,
Ge Wang, Proc. of SPIE Vol. 11113, 1111314 · © 2019 SPIE
CCC code: 0277-786X/19/$21 · doi: 10.1117/12.2529340

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The form of the intraoral device and the related shape changes under mechanical load are essential for functionality and patients comfort. The geometry and the density of the intraoral devices can be measured by means of high-resolution hard X-ray computed tomography [3]. The question arises whether the density resolution allows for the detection of the relevant changes in softness. Even more important, the gravity determines the shape of the soft device, and it has to be doubted whether sample rotation becomes possible to obtain a series of radiographs that allow reconstructing the three-dimensional image of the soft silicone part with a shape represented in Figure 1(b).

It should be noted that high-resolution hard X-ray tomography of soft objects with a geometry far from a cylinder is hardly known. Measuring the geometry as the function of the angle to the gravitation force, however, could enable us to determine the mechanical properties and the related elastic modulus by means of the tomography data and finite element modeling.

2. MATERIAL AND METHODS

2.1 Tailoring the mechanical properties of silicone

Polydimethylsiloxane (PDMS), a polymer also known as dimethylpolysiloxane or dimethicone, belongs to a group of compounds, we often refer to as silicones. PDMS elements with a certain similarity to intraoral devices were prepared by means of the SYLGARD™ 184 silicone elastomer kit (The Dow Chemical Company). The preparation procedure exactly follows the description of the supplier Dow Corning Europe S.A, Belgium. 10.00, 10.00, and 8.75 mL of Dow silicone was mixed with 0.50, 1.00 and 1.75 mL encapsulant to obtain ratios of 1:20, 1:10, and 1:5. The two liquid components were homogenized using a magnetic stirrer for a duration of five minutes. Subsequently, remaining gas was removed by de-airing in an exsiccator for about five minutes, *i.e.* until no gas bubbles were visible in the liquid. This liquid was filled into the Teflon mold and put into an oven for a period of six hours. Cross-linking was achieved at a temperature of 75 °C. The Teflon mold is represented in the lower part of Figure 2(c). After cross-linking the silicone part, termed silicone s, was cooled down to room temperature and removed from the Teflon mold.

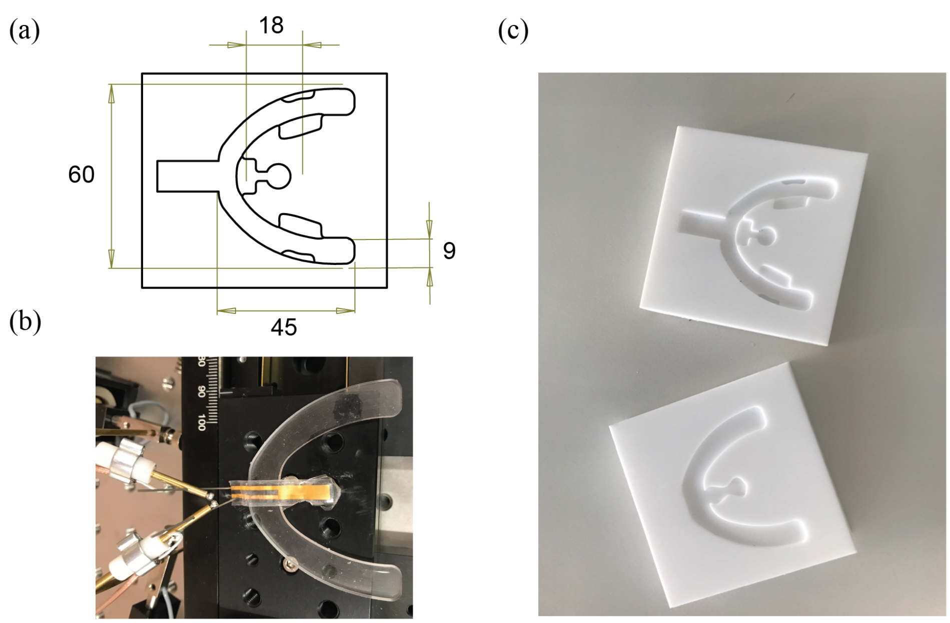

Fig. 2. The dimensions of the silicone elements for the current study are given in (a) and are comparable to the lateral size of the human oral cavity. The numbers represent the distances in millimeters. The photograph in (b) shows a two millimeter-thin silicone element combined with a pressure sensor contacted via flexible electrodes, as required for flexible electronics. The Teflon molds used are imaged in (c). For the present study the simpler mold was applied.

In addition, a similar silicone (SILASTIC® MDX4-4210 BioMedical Grade Elastomer) from the same supplier, which is used for medical applications, was prepared in the analogue fashion. It is termed silicone m. Details can be found on the related webpage: www.dowcorning.com.

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Figure 2(a) displays the scheme of the second-generation device, which has dimensions known from intraoral devices, cf. oral cavity. Using the Teflon molds, as represented in Figure 2(c), one can cast the silicone elements with a thickness of 2 mm. In the same process step or subsequently, one can combine it with sensory elements, as shown in the image of Figure 2(b). To keep the study simple, we have used silicone parts without sensors.

2.2 Nanoindentation measurements

The atomic force microscope FlexAFM, Nanosurf AG, Switzerland was employed to determine the local mechanical properties of the silicone elements prepared. On areas 40 × 40 μm^2 partitioned in 400 domains, we have applied loads of 250 nN using an AFM cantilever with a spherical tip of radius $R = (500 \pm 10)$ nm (B500 FMR, Nanosurf Liestal, Switzerland) in dry condition. The spring constant $k = (3.6 \pm 0.3)$ Nm of the cantilever was determined using the Sader method [4]. The indentation velocity was set to 4 μm/s. The elastic modulus for each subdomain was derived from the force-distance curves. It was reported that the nanoindentation measurements using relatively large spheres on soft polymers could significantly be affected by the adhesion forces F_{adh} [5], a phenomenon included into the Johnson-Kendall-Roberts (JKR) contact model. The JKR contact model is implemented in the ARTIDIS® software (Automated Reliable Tissue DIagnosticS) allowing for the fully automated analysis of the data.

2.3 Tomography measurements

The advanced microtomography system nanotom® m (phoenix|x-ray, GE Sensing & Inspection Technologies GmbH, Wunstorf, Germany) possesses a 180 kV/20 W X-ray tube. This source is optimized for long-term stability by internal cooling. The CT system includes the temperature-stabilized digital GE DXR 500L detector with 3072 × 2400 pixels.

For the present study the accelerating voltage was set to 90 kV using a beam current of 200 μA. The pixel size for the density measurements of silicones with different ratio corresponded to 40 μm. For each tomogram 2,000 equiangular radiographs were acquired with an exposure time of 1.5 s each with an skip time of 0.5 s after each angular position. For the measurements of the silicone elements a pixel size of 30 μm was selected. The supported silicone element was measured at 1,600 equiangular positions with an exposure time of 2.25 s and a skip time of 0.75 s. For the soft silicone sample 1,800 radiographs were acquired with an exposure time of 2 s and a skip time of 0.5 s.

3. RESULTS

3.1 Local elastic moduli of silicone elements

Figure 3(a) shows the experimental results of the nanoindentation measurements on ten selected locations for silicone s with the base-to-crosslinker ratio of 1:20. Although the experiments should provide identical curves for a homogeneous silicone sample, the scattering is obvious. Therefore, averaging of many measured data is recommended, as demonstrated in the diagram of Figure 3(b). Nevertheless, the Gaussian fit is significantly broadened and gives rise to an error bar of about 25%, see Figure 3(b).

The comparison of the experimental data from silicone s with ratios of 1:10 and 1:20, however, yielded a clear difference. Silicone s with a ratio of 1:20 possessed a Young's modulus of (590 ± 150) kPa. The silicone s with a ratio of 1:10 is much harder and has a about 50% larger elastic modulus, namely (920 ± 130) kPa.

The preliminary measurements carried out within the present study showed that the experiments were reproducible and reliable, but have to be repeated under well-defined conditions. As many bubbles were present after curing, we have to substantially improve the preparation procedure of the silicone parts.

3.2 Density measurements using nanotom m

The modification of the stiffness is often related to a change in density. Computed tomography enables us to determine the local X-ray density of the sample investigated. Therefore, silicone s and silicone m was not only casted and cross-linked in the Teflon mold represented in Figure 2 but also in Eppendorf tubes. Considering a cuboid volume of the cylindrically shaped silicone blocks, we derived relative density data from the measured local X-ray absorption.

For silicone s with ratios of 1:5, 1:10, and 1:20, we found 564 ± 12, 554 ± 12, and 555 ± 9 a.u., respectively. For silicone m with the ratios of 1:5, 1:10, and 1:20, we identified 581 ± 11, 582 ± 10, and 589 ± 15 a.u., respectively. Hence, we could conclude that the change of the mixing ratios, which gave rise to significant differences in the mechanical

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properties, are below the detection limit of the tomography setup. Although silicone s and silicone m exhibit close similarity according to the information of the supplier, their X-ray density differs by about 5%.

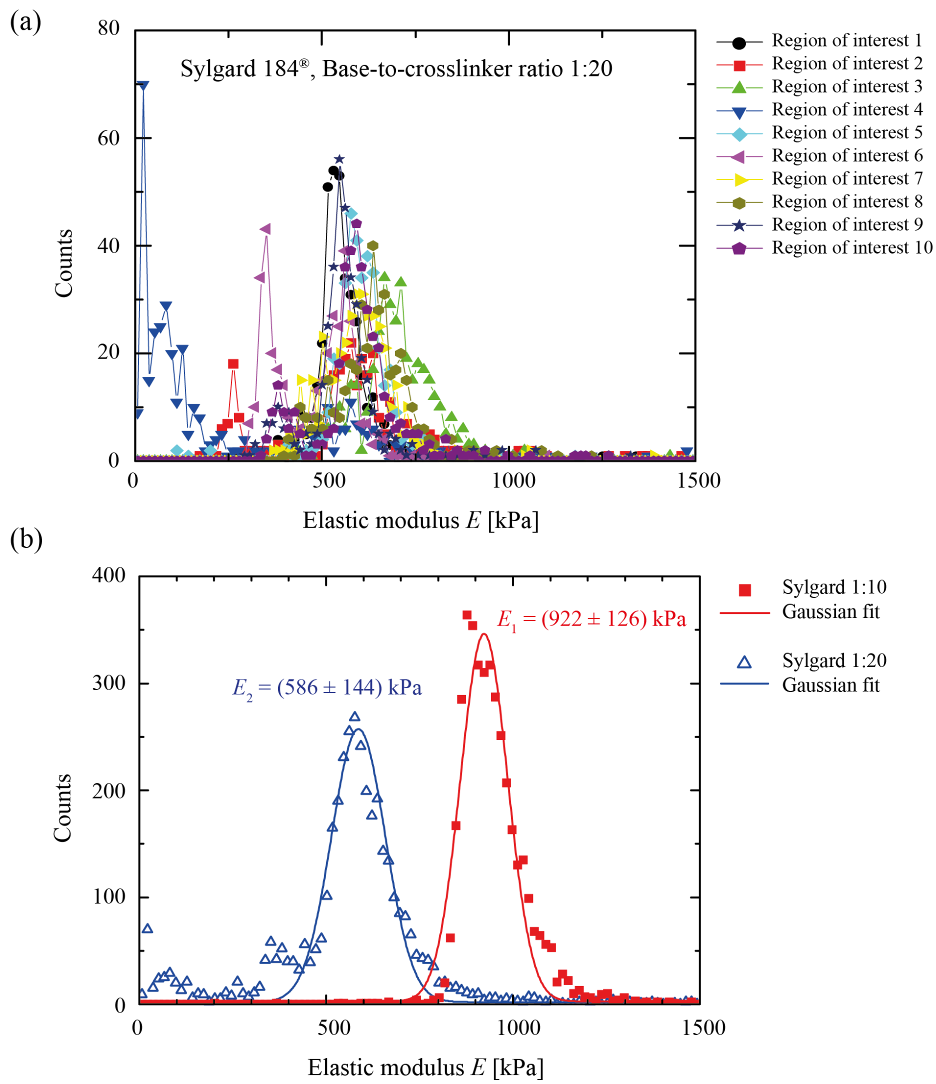

Fig. 3. Nanoindentation experiments permit the detection of the elastic modulus locally. In most cases, however, a single dataset could be misleading, as indicated for ten selected regions of interest on the overall homogeneous elastomer termed silicone s in (a). The averaging generally yields data, which can be reasonably fitted using a Gaussian, see diagram (b). The peak position provides the elastic modulus of the material, whereas the full-width-at-half maximum is often regarded as related error bar.

3.3 Tomography measurements of the supported silicone element

The silicone element is so soft that the gravity led to substantial bending. In a first tomography experiment, the silicone element was supported by a polystyrene plate taken from a Petri dish. This is an appropriate choice, because it is less dense that the silicone. The polystyrene plate is fixed on a cylinder, given in light blue color in Figure 4, by means of a thermoplastic adhesive known as hot glue. All these parts are less X-ray dense than the silicone, a choice, which prevents streak artefacts by strongly X-ray absorbing components.

The histogram of the tomography data exhibit more peaks than originally expected, see Figure 4. Several of the peaks do not show the expected Gaussian shape, cf. the peak near zero related to the air. The shoulders between the peaks may

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result from partial volume phenomena. Most striking, however, is the double peak related to the silicone element. The double peak probably appears due to internal beam hardening correction of the reconstruction software.

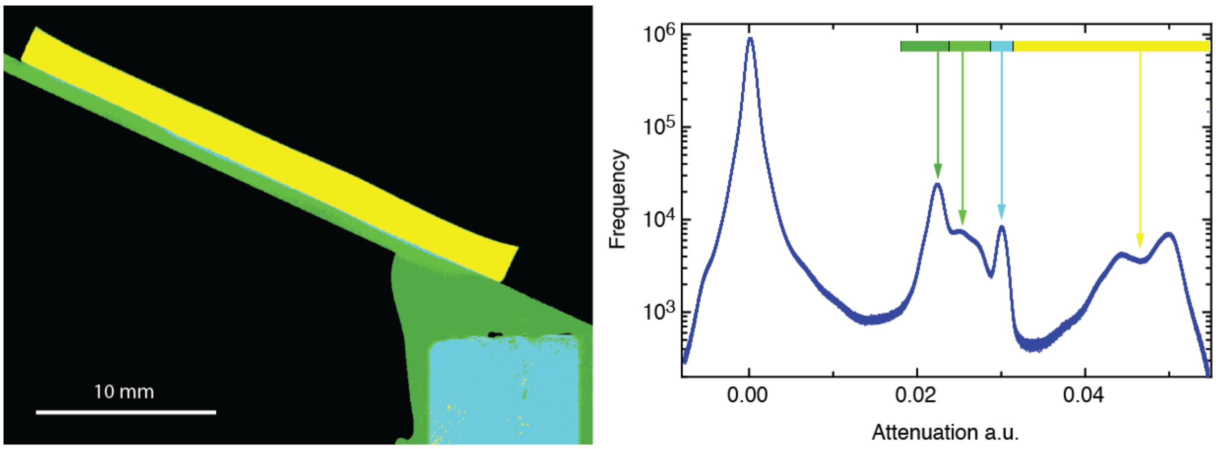

Fig. 4. The silicone element represented in yellow color within the virtual cut of the tomography data is supported by a polystyrene plate (lighter green), which is fixed on a polymer cylinder (bright blue) using a hot melt adhesive. The diagram on the right of the virtual cut is the histogram of the three-dimensional data with the color bar used for the visualization of the virtual cut. The silicone element seems to consist of two or even three differently dense components.

3.4 Tomography of a soft silicone sample

Figure 5 illustrates the bending of the silicone element caused by the gravitation force. Each color represents one tomogram. White is selected to show the situation of the smallest impact of the gravity. The rotation of the silicone element by 90 degrees resulted in the tomogram in red color, which showed the strongest bending. The situation in between, *i.e.* 45° to the gravitation force is represented in blue. The assembly of the three three-dimensional representations is displayed on the right of Figure 5 and a related animation is given in Video 1.

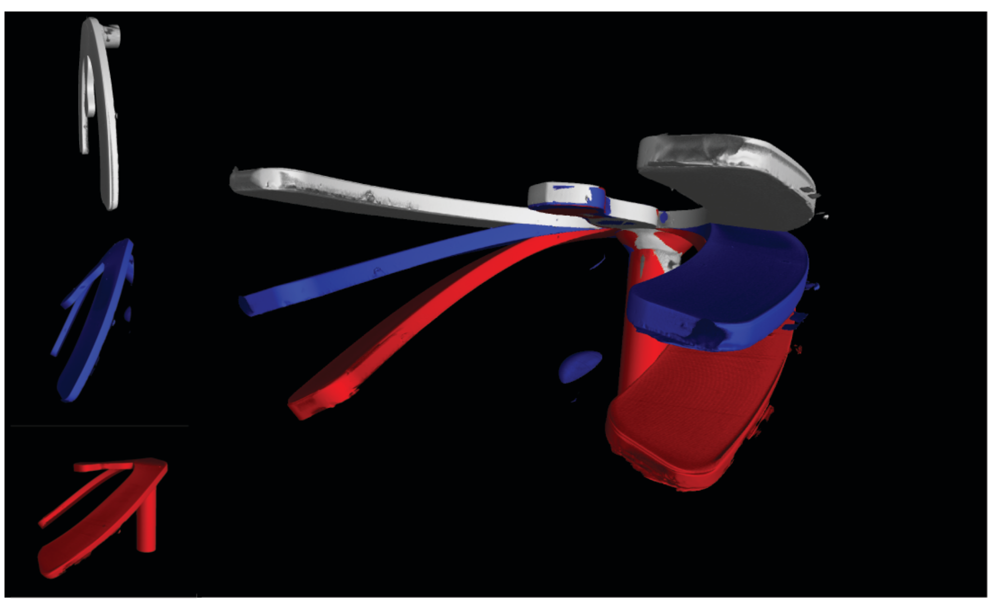

Fig. 5. The soft silicone element (silicone s, ratio 1:20) was imaged using the advanced CT-system nanotom® m in three orientations to the gravitation force; white – 0°, blue – 45°, and red – 90°. The assembly of the three tomograms clearly shows the impact of the gravitation force on the shape of the silicone element.

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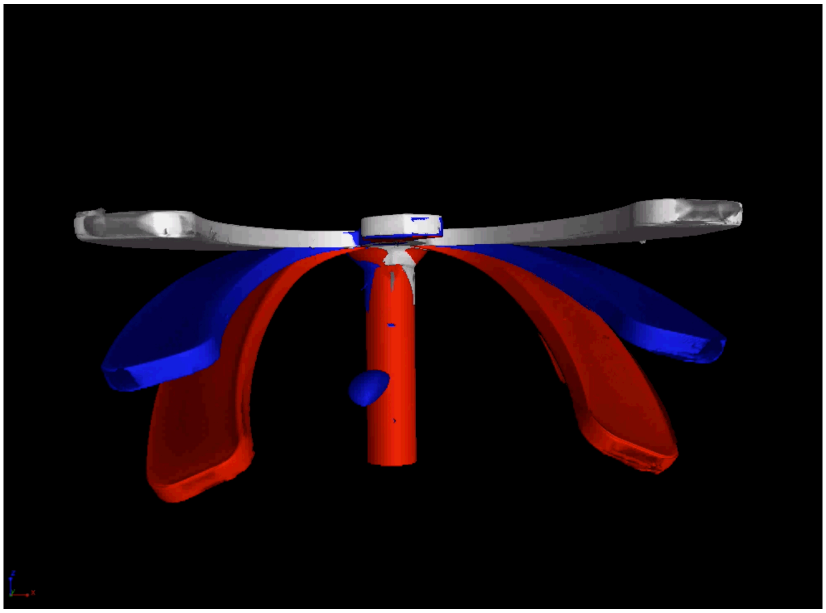

Video 6. The rotation of the assembly from the three tomograms perfectly demonstrates the gravity-induced bending of the silicone element. Its softness is characterized by a Young's modulus below 1 MPa. https://doi.org/10.5281/zenodo.3460309

4. DISCUSSION

High-resolution tomography experiments are generally performed using solid states, see e.g. [6]. Soft tissues or even liquids can be visualized within containers such as Eppendorf tubes. Tomographic imaging of silicone elements with specific shape, as used within the present study, however, is challenging, since the rotation might cause shape changes. The present study proves that, although the gravitation force causes significant bending, the imaging renders possible.

The elastic modulus derived from the nanoindentation measurements of silicone s with the ratio 1:10 and 1:20 fit the elastic moduli given in the relevant literature: 1.19 MPa and 0.57 MPa [7, 8]. Nonetheless, extended series of nanoindentation measurements on samples of improved quality are necessary to identify the relation between the ratio of the two components and the elastic properties of the obtained specimen.

The elastic modulus of the silicone elements, however, can also be derived from the tomography data. For this purpose, the silicone element has to be imaged at selected angles to the gravitation force. Based on these three-dimensional data and finite element modeling, one should be able to derive the Young's modulus of the silicone parts with necessary precision.

ACKNOWLEDGEMENT

The authors thank for the financial support of the Swiss National Science Foundation in the frame of the R'equip initiative, proposal #316030_133802.

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