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Nandula D. Wanasekara

University of Massachusetts - Dartmouth

V. K. Vadlamani

University of Massachusetts - Dartmouth

Animesh Agrawal

University of Massachusetts - Dartmouth

Vijaya B. Chalivendra

University of Massachusetts - Dartmouth

Paul D. Calvert

University of Massachusetts - Dartmouth, pcalvert@uow.edu.au

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Abstract

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Keywords

epoxy, indentation, gels, hydro, tissue, applications, soft, nano

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Nano-indentation of Epoxy Hydro gels for Soft Tissue Applications

N.D. Wanasekara, V.K. Vadlamani, A. Agrawal, V.B. Chalivendra, P. Calvert

University of Massachusetts Dartmouth
285 Old Westport Road
North Dartmouth, MA 02747

Abstract—Nano-indentation characterization studies on epoxy hydro gels used for soft tissue applications are conducted using atomic force microscope (AFM). Two kinds of hydro gels: (a) plain epoxy hydro gels, and (b) epoxy hydro gels reinforced with three-dimensional polyurethane fiber pattern are considered in this study. The three-dimensional fiber pattern is generated using rapid robo-casting technique. Again these two hydro gel types are tested under five different conditions: (a) dry gel, (b) saturated with certain percentage of water, (c) saturated with 100% water, (d) 100% partially immersed in water, and (e) 100% fully immersed in water. Preliminary results obtained on plain epoxy hydro gels show that the saturated conditions decrease Young's modulus values by 15%.

I. INTRODUCTION

Hydro-gels are water swollen polymeric networks. They are insoluble when placed in aqueous solution due to chemical and physical cross-links. Hydro-gels play an important role in biomedical applications [1]. In the last decade hydro-gels have played an important role in tissue engineering in which they are used as scaffolds to guide the growth of new tissues. [2]. The swelling behavior of hydrogels provides an aqueous environment comparable to soft tissue for encapsulated cells. So the first thing that comes to mind, when designing hydrogels for tissue engineering applications is the design of gels with good mechanical properties at various equilibrium swelling conditions. In this case, nano-indentation is the best technique to characterize the mechanical properties of gels. Indentations were performed at different locations to assess Young's Modulus values with their standard deviation. Especially we aim to develop an understanding of Young's Modulus values based on the water saturation level of the gels. In this way it can be proven that heavy water absorbance by the gels have a plasticizing effect on the polymeric materials reducing the Young's Modulus values [1]. A detailed analysis will be performed on the Modulus values as a function of controlled equilibrium swelling conditions using atomic force microscopy (AFM). In all these cases homogeneous swelling will be maintained.

II. EXPERIMENTAL DETIALS

Comprehensive set of experiments are planned using the Atomic Force Microscope for the gel samples. As the initial

step, gel samples were prepared with controlled cross-linking density. Gels were prepared by mixing two epoxies Butanediol di-glycidylether and Poly ethylene glycol DGE (1:1 ratio) with Jeffamine D-230. The reactive ratio of epoxy to Amine H ion is 1:2 [2]. The desired cross-linking density is controlled by the molar ratio of amine and epoxy components. The prepared mixture was kept for 24 hours to complete the reactions and totally become a gel. A three-dimensional robo-casting technique will be employed to develop epoxy hydro gels reinforced in polyurethane fiber pattern [3, 4]. The technique is computer controlled x-y-z dispensing needle unit (with a diameter of 100 microns) to generate well-defined patterned structure.

The nano-indentations were done using AFM at four different conditions, dry gel, 100% saturated wet gel without immersed in water at testing stage, gel partially immersed in water and fully immersed in water with a tiny water layer on top of the sample. Swelling percentage of the gels will be controlled to obtain a Young's Modulus value profile. The swelling percentage control methodology is yet to be designed and then the indentations will be performed on the respective gel samples to obtain the Young's Modulus values. Typical force-deflection diagram for a dry sample is shown in Fig. 1. These results will help to develop an understanding of the change of mechanical properties as a function of the swelling co-efficient of the gel samples. As the next step, fibers will be embedded to gels. The pattern of arrangement of fibers in the sample will be such that they have different enclosing spaces among them. That is the area enclosed by four fibers will be different across the plain. Nano-Indentations will be performed on these areas to determine the change of hardness values.

Fig.1. Typical Force-Deflection Diagram of a Gel

If the fiber enclosing area is small, it is expected that the restraining force applied to the indentations will be high. Therefore Young's Modulus values will be different for these cases. These samples will also be subjected to different swelling conditions and indentations will be performed to determine the Young's Modulus values. These experiments will be helpful to determine the mechanical behavior of hydrogels under different swelling percentages and different fibers embedded in them. As the preliminary steps to assess the applicability of nano-indentation techniques and to determine the softening and plasticizing effect of the gels, AFM indentations of gels under different conditions were performed. The gel samples were cut into small square shape pieces with smooth surface in order to test them in the Atomic Force Microscope. Non-contact mode scanning was performed by the AFM to ascertain the smoothness of the sample surface. AFM non-contact mode image of the dry gel sample is shown in Fig. 2. Calibration of AFM cantilever stiffness was done by taking an optical photograph of the cantilever and determining the dimensions of the cantilever. In order to determine the Young's Modulus values using AFM, a methodology provided by Reynaud.et.al [5] was used. A MATLAB program was written for easy calculation of the Young's Modulus values.

III. PRELIMINARY RESULTS

Nano-indentations were done on the completely dry sample without water. Indentations were performed in several locations to make sure the values have higher consistency. The average Young's Modulus value for the dry sample was 1.1 MPa. Then the remaining gel samples were kept fully immersed in water for 24 hours to have them 100% saturated in water. The importance of doing the indentations immediately after taken out from the water was to keep the surface 100% saturated and increase the accuracy of the values to be obtained. A consolidative summary of the results of Young's Modulus Values with 95% confidence level are shown in the Table 1.

Fig.2. AFM non-contact mode Image of the dry hydrogel sample

TABLE 1
YOUNG'S MODULUS VALUES OF HYDROGELS

Hydrogel condition	Young's Modulus (MPa)
Dry	1.10 ± 0.038
Wet sample taken out from water	1.02 ± 0.015
Partially Immersed in water	0.92 ± 0.013
Fully Immersed in water	0.91 ± 0.014

Next step was to partially immerse the saturated gel sample in water and do indentations to find out the Young's Modulus values. A 5mm × 5 mm gel sample with 100% water saturation was stick to tiny plastic container by using a double side tape. The container was filled with water until the sample was partially immersed in water. Several indentations were performed at different locations and Young's Modulus values were calculated. The Young's modulus value was 0.92 MPa. Then as the fourth step, the gel sample was fully immersed in the water and a tiny layer of the water was on top of the gel sample. The indentations were done on the gel sample and the Young's Modulus value was again 0.91 MPa. Therefore the gel was completely saturated at the time when it was partially immersed and has no further softening effect. The Young's Modulus values are indicative of the plasticizing effect of the water in gels.

Fabrication of various three-dimensional patterns using robo-casting technique is in progress. Experiments on epoxy hydro gels with three-dimensional fiber pattern are underway at various conditions. The complete set of results, comparison and analysis will be presented at the time of presentation.

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