A new tensile stage for *in situ* electron microscopy examination of the mechanical properties of "superelastic" specimens

Kalin I. Dragnevski,[a)] Trevor W. Fairhead, Rik Balsod, and Athene M. Donald
Department of Physics, Cavendish Laboratory, University of Cambridge, JJ Thomson Avenue, Cambridge CB3 0HE, United Kingdom

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We have developed a novel tensile stage that can be used for *in situ* electron microscopy examination of the mechanical properties of "superelastic" materials. In our stage, one of the specimen clamps is replaced by a cylindrical roller, which when driven by a motor can easily stretch ("roll on") any specimen irrespective of its plastic properties. We have used the so-called Roll-o-meter in the study of the tensile behavior of two different film formed latex formulations, here referred to as standard and novel. We find that the values of the tensile strength and extension to break of the studied systems, measured by using the Roll-o-meter, are similar to those measured by a Hounsfield tensile testing machine outside the microscope chamber. Further, *in situ* environmental scanning electron microscopy examination of the deformation and failure of the lattices revealed that the standard specimens exhibit a more ductile behavior, compared to the novel ones. © *2008 American Institute of Physics.* [DOI: 10.1063/1.3054766]

Tensile testing is a well-established technique and when used in combination with electron microscopy allows not only the evaluation of the mechanical properties of the studied system but also the *in situ* examination of the microstructural and fracture development of the material in question.[1–4] Most commercial and custom built stages[5,6] consist of two jaws that are driven apart by a stepper motor connected to a lead screw through a stepped down gearbox (Fig. 1). In the case of environmental scanning electron microscopy (ESEM), the stages can be adapted to include a cooling module, which allows samples to be maintained hydrated during testing.[5] The cooling module, shown in Fig. 1(b), consists of a Peltier chip sandwiched between a cold finger and a liquid cooled block. This arrangement has been successfully applied in the study of a number of material systems.[5–7]

However, there are a number of factors that can limit the performance of these tensile stages the biggest of which is the fact that they only allow elongations up to the region of 300%–500%, which therefore makes them unsuitable for the study of superelastic specimens.

In this paper, we introduce a new tensile stage which overcomes the above limitation and can be used for *in situ* examination of superelastic specimens. Here, superelastic specimens are defined as those exhibiting ≥1000% extensions prior to fracture.

The concept used for the design of the new tensile stage, schematically represented in Fig. 2(a), is based on the idea of replacing one of the specimen clamps with a cylindrical roller (in our case 10 mm in diameter), which when driven by a motor would easily stretch ("roll-on") any specimen irrespective of its plastic properties. The roller is made in two halves, joined by screws, in order to allow accurate positioning of the specimen. The other jaw is stationary and is also used to house the miniature load cell, the signal from which is displayed on a monitor in the form of a force-displacement curve. The tensile stage uses a positive clamping system that presents no sharp edges to the sample. Both fixed and rotating clamps use curved surfaces to clamp the specimen. In order to ensure that no slippage occurs, we tested (simply by stretching) a 19 mm strip of 0.05 mm thick metal shim specimen marked at the point where it exited the clamp. After the sample was unloaded, it was evident that it did not move/slip during the experiment. This, together with the fact that the data obtained using the Roll-o-meter is comparable to the data obtained when using a different tensile testing technique (described below), clearly indicates that slippage does not take place during the tensile experiment.

Another issue that needs to be considered when using the roll-on procedure is the possibility of bending resulting from an increase in the roller's diameter. However, we expect this to be quite small and that is certainly the case for the systems that we studied.

The tensile stage [Fig. 2(b)] was built in the Cavendish Laboratory, University of Cambridge. The motor used to drive the roller was purchased from Phytron-Elektronik GmbH, Germany (Model: VSS25.200.1.2.GPLFV). A 200 N miniature load cell, model XFTC310, purchased from KA Sensors Ltd., Grantham, UK was fitted to the stationary jaw for the initial experiments. The force-displacement curves were obtained by using a tensiometer software previously developed in the Cavendish Laboratory, University of Cambridge.[8] By including the sample dimensions, the software can also present the data in the form of a stress-strain curve, from which the extension to break (%) and the tensile strength (Pa) of the material can be determined.

The tensile stage was used in an ongoing comparative study of two film formed acrylic latex formulations, supplied by ICI Plc, here defined as standard and novel and described in detail in a previous paper.[9] These were chosen because

[a)]Author to whom correspondence should be addressed. Tel.: +44 (0) 1223 765796. FAX: +44 (0) 1223 337000. Electronic mail: kd281@cam.ac.uk.

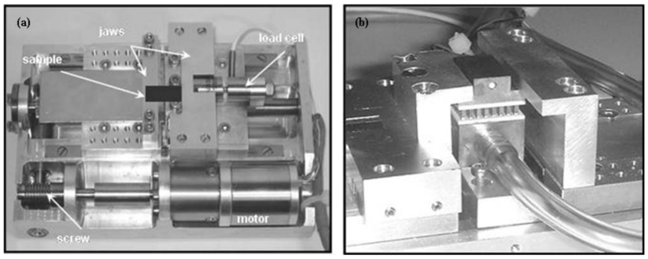

FIG. 1. Example of a conventional tensile stage (a) built in the Cavendish Laboratory and (b) photograph of the cooling module positioned between the jaws (9).

initial tensile tests carried out of the microscope chamber indicated that the lattices exhibit over 1000% extensions to break. Furthermore, ESEM examination of the surfaces of the dry novel specimens revealed the presence of previously unseen dendritic morphologies formed via crystallization of salt.[10] In contrast, the surfaces of the standard specimens contained only individual salt crystals with rectangular shapes.

For the experiments carried out in this study, the specimens from the two studied systems were prepared by casting latex onto a flat release film attached to a glass substrate and then drying for at least 72 h at room temperature. This time was required for the formation of the dendritic morphologies in the novel specimens.[10] The films were then simply peeled off from the release film and subjected to tensile testing. Sample dimensions were as follows: $L=40$ mm, $W=2-4$ mm, and $H=0.2-0.4$ mm.

The tensile stage was mounted in the chamber of an FEI Quanta 3D ESEM. The specimens were then fixed between the stationary jaw and the roller of the tensile stage. The tensile testing was carried out at a speed of 10 mm/min. Imaging was carried out at a water vapor pressure of 1 Torr and an operating voltage of 10 kV.

In order to evaluate the accuracy of the Roll-o-meter, the mechanical properties of the studied lattices were also measured outside the microscope by using a Hounsfield Tensile Testing machine equipped with a 250 N load cell at a motor speed of 10 mm/min. The experimental error for both measurements was in the range of $\pm5\%$. This was determined by testing at least three specimens in each experiment.

The results from the microstructural examination of the deformation and fracture process of the two latex compositions are presented in Fig. 3. At this point it is important to note that the bright contrast [Fig. 3(b)] seen in the images of the novel samples is caused by the presence of the salt dendrites. It is clearly seen that in both cases deformation occurs via the formation of a neck. Furthermore, in the case of the standard samples, the fracture appears to be followed by elastic relaxation, which is evident from the images in Fig. 3(a), frames 4 and 5, where it can be seen how the sample after fracture initially "folds back" and then relaxes and "opens up." This is also indicative of a ductile behavior of the specimens. However, in the case of the novel latex, relaxation was not observed, thus suggesting a more brittle behavior of the system.

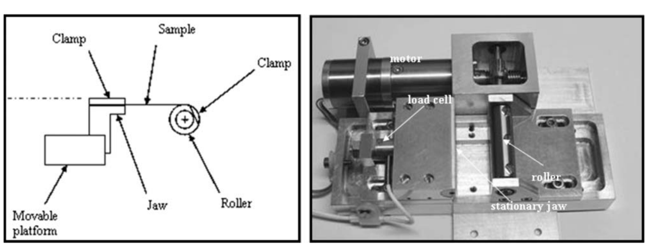

FIG. 2. Schematic (a) and photograph (b) of the Roll-o-meter. (b) ESEM image of the roller during the tensile testing of a latex specimen.

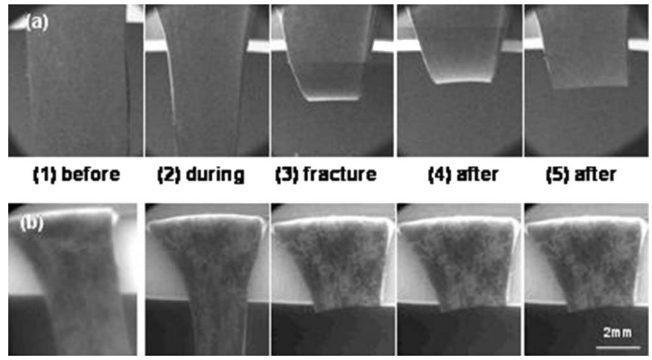

FIG. 3. ESEM images of (a) standard and (b) novel latex specimens during tensile testing using the new tensile stage.

The values of the mechanical properties [tensile strength (Pa) and extension to break (%)] measured with the Roll-o-meter and the Hounsfield machine are presented in Table I.

It can be seen that they are very similar, which indicates that the new tensile stage can be used not only for *in situ* examination of the microstructural development of superelastic specimens but also for accurate measurement of their mechanical properties. From the data it is also evident that the extension to break of the standard system is higher compared to the one of the novel, but the values for the tensile strength of the novel latex are higher than those for the standard material. Therefore, it may not be unreasonable to suggest that the crystal structures forming in the novel system have a twofold effect on the properties of the latex: on the one hand their formation re-enforces the material, which leads to an increase in the strength of the latex, but on the other results in a decrease in the plastic properties of the system.

A unique tensile stage that can be used for *in situ* electron microscopy examination of superelastic specimens has been successfully designed and commissioned. The results suggest that there is a difference in the mechanical behavior of the studied latex systems, which is consistent with the microstructural observations. The properties of the samples measured by using the Roll-o-meter are comparable to those measured by conventional tensile testing machines, thus indicating that the stage can also be used for accurate measurements of tensile strength and extension to break.

TABLE I. Comparison of the mechanical properties of the studied lattices measured with the Hounsfield machine and the Roll-o-meter.

	Standard		Novel	
	Hounsfield	Roll-o-meter	Hounsfield	Roll-o-meter
Tensile strength (Pa)	16 500	16 200	19 000	18 500
Extension (%)	1 140	1 110	1 000	960

[1] D. T. A. Matthews, V. Ocelik, P. M. Bronsveld, and J. T. M. De Hosson, Acta Mater. **56**, 1762 (2008).
[2] S. B. Hosseini, C. Temmel, B. Karlsson, and N. G. Ingesten, Metall. Mater. Trans. A **38A**, 982 (2007).
[3] S. N. Lu, Z. Y. Guo, W. Q. Ding, D. A. Dikin, J. Lee, and R. S. Ruoff, Rev. Sci. Instrum. **77**, 125101 (2006).
[4] Z. Z. Yuan, Q. Dai, X. N. Cheng, L. Pan, and A. D. Wang, Mater. Charact. **56**, 79 (2006).
[5] R. Rizzieri, F. S. Baker, and A. M. Donald, Rev. Sci. Instrum. **74**, 4423 (2003).
[6] D. J. Stokes and A. M. Donald, J. Mater. Sci. **35**, 599 (2000).
[7] B. L. Thiel and A. M. Donald, Ann. Bot. (Lond.) **82**, 727 (1998).
[8] N. Traiter, Ph.D. thesis, University of Cambridge, 2008.
[9] K. I. Dragnevski and A. M. Donald, Colloids Surf., A **317**, 551 (2008).
[10] K. I. Dragnevski and A. M. Donald, Prog. Org. Coat. **61**, 63 (2008).