F. Mechanical Reliability of Electronic Materials and Electronic Devices

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Objectives

- Identify and use mechanical test methods that accommodate the testing of hydroxylated polystyrene (PVOH) dielectric film specimens (and geometries thereof) provided by Sandia National Laboratories (SNL).
- Measure and interpret mechanical properties of PVOH films and identify which are the best candidates for future manufacturing scale-up based on mechanical robustness.

Approach

- Perform mechanical properties microprobing on a suite of PVOH-elastomer films to quantify the elastic modulus (E) and hardness (H) of each.
- Perform additional mechanical tests (e.g., scratch-testing, indentation creep) in an effort to further assess the PVOH-elastomer films' propensity to crack or tear under a tensile strain.

Accomplishments

- Measured the E and H of 15+ PVOH-elastomer films, finding that the use of 4010 elastomer in PVOH resulted in lower E and H than the use of 5015 and tetraethylene glycol divinyl ether (TEGDE) elastomers in PVOH.
- Conducted scratch testing (which exploits a film's propensity to crack or spall) that showed PVOH-4010 films tend to be more spall resistant than PVOH-5015 and PVOH-TEGDE films.

Future Direction

- Measure the tensile strength distribution of the two down-selected elastomer-PVOH dielectric films as a function of temperature and link the strengths to the population of strength-limiting flaws.
- Predict and verify allowable manufacturing and service strains of elastomer-PVOH dielectric films.

Introduction

A primary focus of the Power Electronics effort in the Automotive Propulsion Materials Program is to develop polymer capacitor technology that will replace current electrolytic, dc bus capacitors for power electronic modules in hybrid electric vehicles. The ultimate objective is to make the power modules more compact while maintaining tight voltage and temperature requirements and long service life without compromise caused by mechanical breakdown of the dielectric film. Toward that end, we collaborated with SNL in this project to mechanically evaluate a suite of SNL-manufactured PVOH dielectric polymers that have the potential to satisfy the objectives. The present effort has two objectives: (1) measure baseline mechanical properties of that suite of PVOH compositions and interpret their results so as to suggest which are most suitable for manufacturing scale-up, and (2) quantify the mechanical performance of those manufactured films so manufacturers and end-users of these dielectric films may use them without mechanical breakdown.

Results

A suite of PVOH-elastomer films were provided by SNL for mechanical characterization. The PVOH films were fabricated so that each one used one of three different elastomers designated as 4010, 5015, and TEGDE in varied additive contents of up to 30 wt %. The PVOH-elastomer matrix is shown in Table 1. The films were spin-coated onto metallized (aluminum) silicon wafers; a representative cross-section of this three-material system is shown in Figure 1. The PVOH-elastomer film was nominally 600 nm thick and was well bonded to the aluminum metallization layer under it. Because of the small film thickness and strong bonding (i.e., film removal was prohibited), the mechanical characterization was limited to use of a mechanical property microprobe (MPM) and other techniques

Table 1. Test matrix of elastic modulus and hardness tests

Dielectric film	Elastomer content	Anneal (ºC)
PVOH	0%	220 and 250
PVOH/4010	5%	220 and 250
	15%	220 and 250
	20%	220 and 250
	30%	220 and 250
PVOH/5015	5%	250
	15%	250
	20%	250
	30%	250
PVOH/TEGDE	5%	250
	15%	250
	20%	250
	30%	250

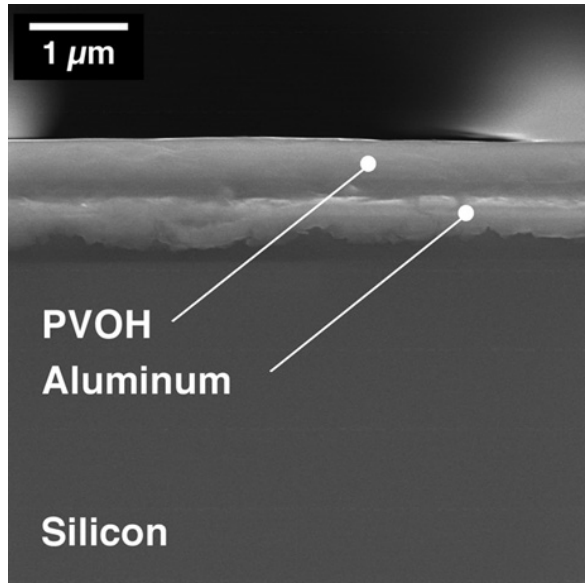

Figure 1. Cross-section of PVOH dielectric film (top), aluminum electrode layer (middle), and silicon substrate (bottom).

that could test the mechanical robustness of the PVOH-elastomer film in situ.

The MPM is an automated instrument consisting of four primary components: an indenter (Berkovich indenter) whose vertical displacement (nanometer resolution) and applied load (microgram resolution) are controllable; an optical microscope (OM) with several objective lenses; a precision X and Y stage that translates the metallographically prepared specimen between the OM and the indenter; and a computer that controls the OM's lens turret (50–1500× magnification), stage movement, and indenter load and displacement. A schematic of the MPM is shown in Figure 2.

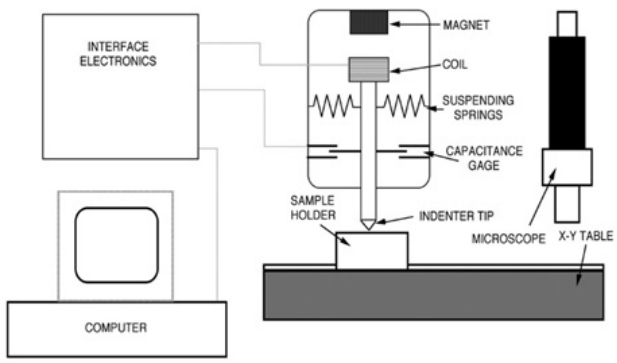

Figure 2. Schematic representation of the mechanical properties microprobe.

The computer also collects data on the indenter's displacement and load and is interfaced with a TV monitor and camera that show the microscope's field of view and allow inspection of each indentation. The MPM is housed inside an insulated cabinet that serves to minimize its susceptibility to laboratory room temperature fluctuations and vibrations (which are problematic when controlling displacement at the nanometer level). In a further effort to minimize the effects of vibration and thermal fluctuations, both the computer and the MPM-containing cabinet are housed in an insulated room within the laboratory.

The load/displacement history generated during indentation can be interpreted with the aid of an appropriate model to calculate the *E* and *H* of the material, as shown in Figure 3. *H* is calculated by dividing the peak load by the residual indent area, while *E* is determined from the slope of the unloading curve.

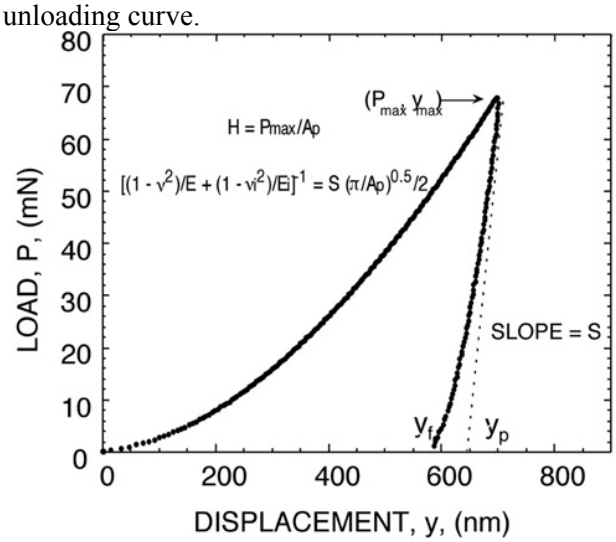

Figure 3. Typical load-displacement curve generated during and unloading with mechanical properties microprobe.

The measured *E* and *H* as a function of elastomer and elastomer concentration are shown in Figures 4 and 5, respectively. Sublayers under the top film can influence the apparent *E* and *H* of thin films if the indentation penetrates too far, so only *E* and *H* measurements from sufficiently shallow indentation depths (< ~10% of the PVOH film thickness) were considered. The *E* and *H* of the PVOH/4010 films tended to be of lower value than those of PVOH/5015 and PVOH/TEGDE films for a given additive content, particularly for additive content of 5 and 15%. The *E* and *H* of PVOH/4010, PVOH/5015, and PVOH/TEGDE were arguably equivalent for 20 and 30% additive content. Regarding the effect of annealing temperature on PVOH/4010, it was observed that annealing at 250°C rather than 220°C tended to increase the *E*, though *H* was essentially unaffected by the warmer temperature.

In an additional effort to distinguish differences among the PVOH films, indentation creep tests were performed on selected compositions (0%, 15% and 30% of each elastomer—all 250ºC annealed) of the test matrix shown in Figure 1. A 5-mN load was applied, and the displacement of the deepening indenter penetration was

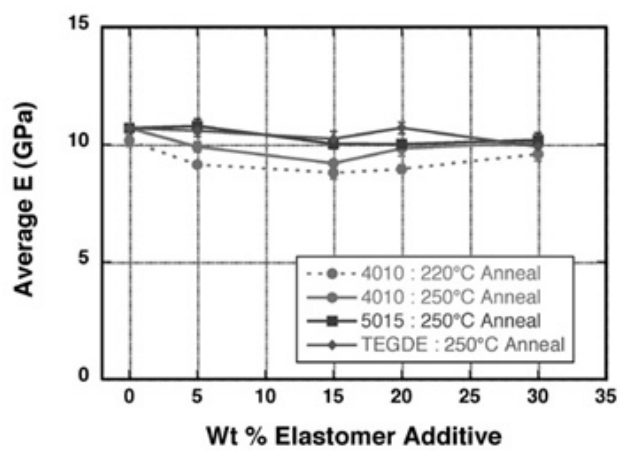

Figure 4. Comparison of elastic modulus as a function of elastomer additive content.

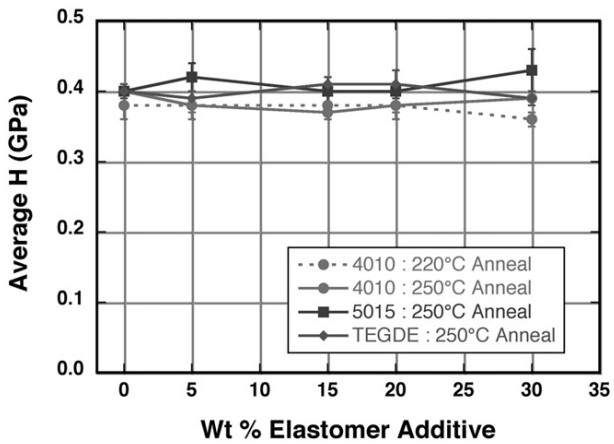

Figure 5. Comparison of hardness as a function of elastomer additive content.

measured as a function of time to 10 minutes. These results were found not to be a discriminator, as the measured creep responses of these seven films were equivalent, although the PVOH/30% TEGDE showed anomolously rapid creep deformation.

A limitation of the described indentation testing is that the results are all a consequence of compressive loading, whereas past observed cracking or tearing in PVOH (and other) dielectric films fabricated at SNL was a consequence of tensile straining. Even though that limitation was recognized early, prior to the indentation, the indentation proceeded because of the nature of the supplied PVOH/aluminum/ silicon test coupons and because a tensile test could not be performed on that PVOH film. If differences in *E* or *H* or creep resistance among the PVOH films in the test matrix existed, then these compression-based mechanical tests would have found them, and those differences potentially could have been used to infer tensile performance. As described, though, the differences in *E* or *H* or creep resistances were nonexistent or subtle.

A series of scratch tests was performed on seven PVOH films (0% additive content and 15% and 30% content of 4010, 5015, and TEGDE) using a diamond stylus, and differences were analyzed. The use of such a test was pursued because it subjected a component of tensile strain to the PVOH film, and the performance of the film under a positive sign of strain could be examined. OM images in Figures 6–8 of the 0%, 15%, and 30% content 5015, respectively, show the former two PVOH films exhibited little or no spallation (i.e., brittleness), whereas there was significant spalling in the 30% 5015/PVOH film. Assuming that the material state of the films has not changed since the time of their deposition, the scratch test results suggest that the 4010/PVOH systems

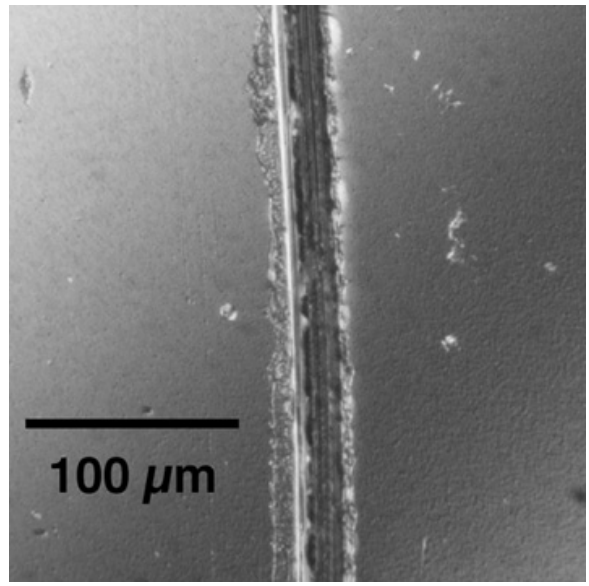

Figure 6. Scratch of PVOH (no elastomer).

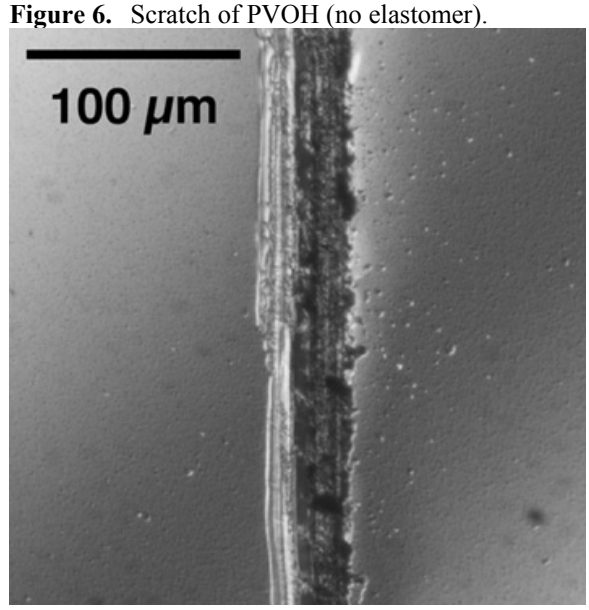

Figure 7. Scratch of PVOH 15% 5015/PVOH.

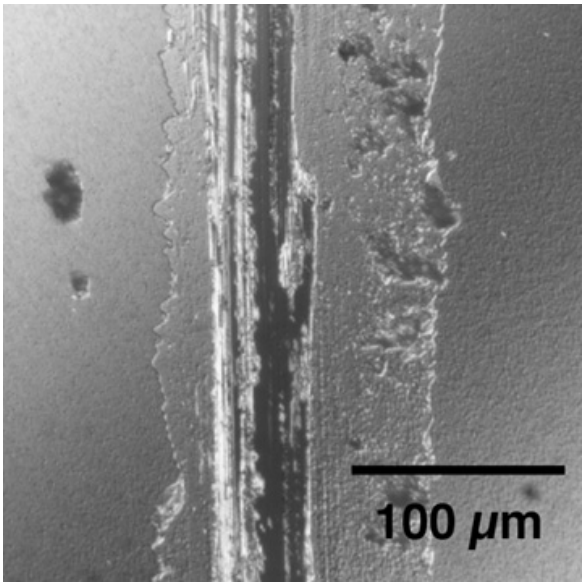

Figure 8. Scratch of 30% 5015/PVOH.

are the least brittle in that there was no significant evidence of spallation in the vicinity of the applied scratch for either the 15 or 30% concentrations of 4010. The 30% 5015 and 30% TEGDE films showed significant amounts of spallation around the scratch, indicating a greater degree of brittleness in those films. There was a small extent of lobing around the scratch in the 15% TEGDE film, indicating a moderate level of brittleness was being exposed.

Conclusions

Spallation is believed to be an indicator of brittleness, and this brittleness is likely linked to a PVOH dielectric film's propensity to tear or crack when strained in tension during manufacture or, ultimately, during service. The 0% PVOH, 15% 4010/PVOH, 15% 5015/PVOH, and 30% 4010/PVOH films exhibited little or no brittleness and are therefore the best candidates for manufacturing scale-up. This lack of brittleness in the PVOH containing the 4010 elastomer is perhaps consistent with its generally having lower E and H than the PVOH films that contain 5015 or TEGDE elastomers. If any of the examined films are apt to tear or crack when subjected to tensile straining, then those would be the 30% 5015/PVOH and 30% TEGDE/PVOH films.