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An instrument for environmental control of vapor pressure and temperature for tensile creep and other mechanical property measurements

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An instrument for measuring the creep response of a material maintained under a controlled environment of temperature and vapor pressure is described. The temperature range of the instrument is 20–250 °C while the range of vapor pressure is 0–1 atm. Data are presented for tests conducted on this instrument with Nafion®, a perfluorinated ionomer developed by DuPont and used as a membrane in polymer exchange membrane fuel cells, over a range of temperature and water vapor pressure. The data are useful for predicting long-term creep behavior of the material in the fuel cell environment as well as providing insight to molecular level interactions in the material as a function of temperature and hydration. Measurements including dynamic and equilibrium strain due to water uptake as well as elastic modulus are described. The main features of the instrument are presented along with experimental methodology and analysis of results. The adaptation of the instrument to other mechanical tests is briefly described. © *2007 American Institute of Physics.*
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I. INTRODUCTION

The mechanical properties of many materials are greatly affected by both temperature and hydration. Examples include fabrics, paper and other wood products, foods, pharmaceuticals, synthetic polymers, and biological materials. Temperature and water activity can have synergistic effects on the mechanical properties of polymeric materials where hydrogen bonding and/or polar interactions affect the secondary structure of the material. Commercially available instrumentation is readily available to assess mechanical properties of materials over a wide range of temperature. However, commercially available instruments offer only limited control of water activity[1] over a range of temperature and this is usually a secondary feature.

A specific area of research where there is a strong need for measuring mechanical properties under an environment of controlled hydration and temperature is polymer membrane materials used in polymer electrolyte membrane (PEM) fuel cells. Most membrane materials developed or under development for PEM fuel cells show viscoelastic properties which are strongly dependant on hydration and temperature. In the fuel cell environment, the membrane is subjected to varying levels of hydration which, together with clamping forces and changes in temperature, create stress in the membrane which can lead to viscoelastic flow and eventually the appearance of pinholes. Even if pinhole formation does not occur, mechanical properties still affect the overall performance of the fuel cell and impact fabrication and processing.

Other investigators have attempted to modify existing mechanical testing instrumentation to achieve control of humidity over a narrow temperature range. Some examples of this work include tensile instruments (Instron) which include either bubbler controlled feeds[2] or using a plastic zipper bag with water or saturated salt solution in the bottom to create a humidified envelope around the test sample.[3] Another example is the work of Bauer *et al.* which involved modifying a differential mechanical analyzer (DMA) with a bubbler and heated block[4] to provide control over water activity during testing. Another group of investigators has created a humidified creep apparatus by flowing humidified gas through a tube containing the sample in order to study effects of hydration cycling.[5]

This work presents an instrument to measure the viscoelastic response of a material under an environment of carefully controlled temperature and hydration with tensile creep.[6] Data are presented for tests conducted on this instrument with Nafion®, a perfluorinated ionomer developed by DuPont and used as a PEM fuel cell membrane. The data are useful for predicting long-term behavior of the material in the fuel cell as well as providing insight to molecular level interactions in the material as a function of temperature and hydration. The main features of the instrument are presented and applications in addition to creep response are highlighted.

II. EXPERIMENTAL

A. Tensile creep response

Tensile creep provides a way to measure the viscoelastic response of a material and is done by simply applying a constant stress σ to the material. The constant engineering

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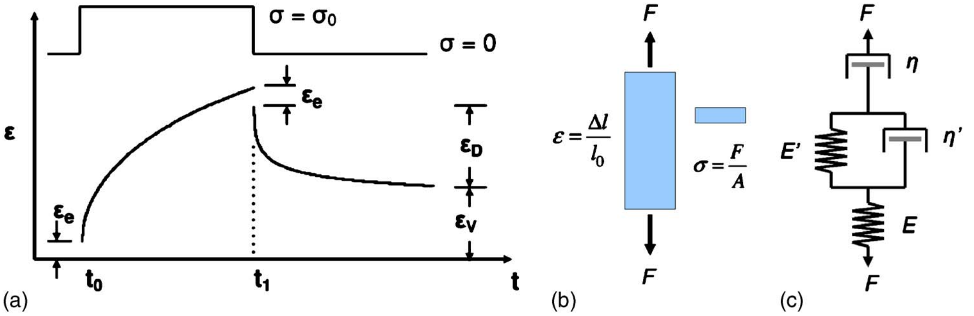

FIG. 1. (Color online) (a) Creep response of the ionomer Nafion (N1110) at 80 °C and 67% RH subjected to a stress of 2.31 MPa. Creep strain components are labeled according to Table I. The loading program is shown at the top of the plot. Sample details are shown at (b) with a spring and dashpot model at (c) representing the viscoelastic response of the material.

stress results in a strain ε which is monitored as a function of time. Stress and strain on the sample are defined as follows:

$$\sigma \equiv \frac{F}{A} \text{ and } \varepsilon \equiv \frac{\Delta \ell}{\ell_0}, \tag{1}$$

where F is the applied force, A is the cross sectional area normal to the applied force, ℓ_0 is the initial length, and $\Delta \ell$ is the change in length (Fig. 1). The resulting creep response of a representative viscoelastic material is shown in Fig. 1. For a material which behaves linearly, creep strain scales with applied stress. Creep compliance J is obtained by dividing creep strain by applied stress and allows one to compare runs with different applied stresses,

$$J = \frac{\varepsilon}{\sigma}. \tag{2}$$

The molecular contributions giving rise to the different creep response contributions are given in Table I. Because creep response is controlled by molecular interactions, creep experiments can often elucidate certain molecular interactions in a material and how these interactions change with temperature and in the presence of solvents.

Using the instantaneous strain response resulting from applying and removing stress, one can obtain a reasonable plot of elastic modulus if the strain induced in the material is within the proportional limit. This can be done as part of a typical creep test or separately by cycling stress at short intervals (~30 s). Since creep experiments are usually run for an extended period of time and are destructive, doing separate elastic runs has the advantage of saving considerable time over full creep runs. This allows a much broader range of environmental conditions to be investigated in a given amount of time. Using a bubbler to supply humidity to the chamber also increases flexibility and speed, especially if water activity is varied at a given temperature since the time required for temperature to stabilize is much greater than the time required to change water activity with a bubbler.

B. Materials

Nafion was purchased from Ion Power in the form of an extruded polymer with nominal dry thickness of 0.010 in. (N1110). The polymer was pretreated according to the standard procedure: boil 1 h in 3% H_2O_2 solution, boil 1 h in de-ionized (DI) water, boil 1 h in 0.5M sulfuric acid, boil 1 h in DI water twice. The resulting clean and protonated form polymer was then dried flat on the lab bench by placing between several sheets of filter paper with a book placed upon all to ensure a wrinkle-free material. Once dried to ambient conditions, the polymer was cut into strips of uniform width using a template of the correct width (0.25 or 0.5 in.) and an Exacto® knife.

C. Creep instrument

The instrument designed to measure viscoelastic creep under a controlled environment of temperature and hydration is shown in Figs. 2–4. The sample is clamped to an upper stationary clamp and a lower movable clamp. Stress is applied uniaxially to the sample through the bottom clamp by hanging a weight from the bottom of the rod connected to the bottom clamp. The weight hung from the rod is an open container which can be filled with lead shot to give a mass range of 200–1300 g. Strain is measured with a linear variable displacement transducer (LVDT) (Macro Sensors, HSAR 750-2000) by monitoring the position of a segment of the rod (the LVDT core) connecting the hanging mass to the sample with the movable clamp. The LVDT provides a measurement of strain without contact. Polytetrafluoroethylene (PTFE) guides are in place to axially align the rod and prevent it from swinging while introducing only a small amount of friction. A universal joint at the bottom of the rod connects to the mass without transmitting torque due to misalignment. The lower clamp has a total travel of 3.25 in. between the upper clamp and base of the environmental chamber. This

TABLE I. Ideal creep response components (Ref. 7).

Component	Characteristic	Molecular contribution
Instantaneous elastic (ε_e)	Occurs immediately, completely recoverable	Bond stretching/bending, bonds between chains
Delayed elastic (ε_d)	Rate decreases with time, completely recoverable	Chain uncoiling
Viscous flow (ε_v)	Irrecoverable	Chain slipping

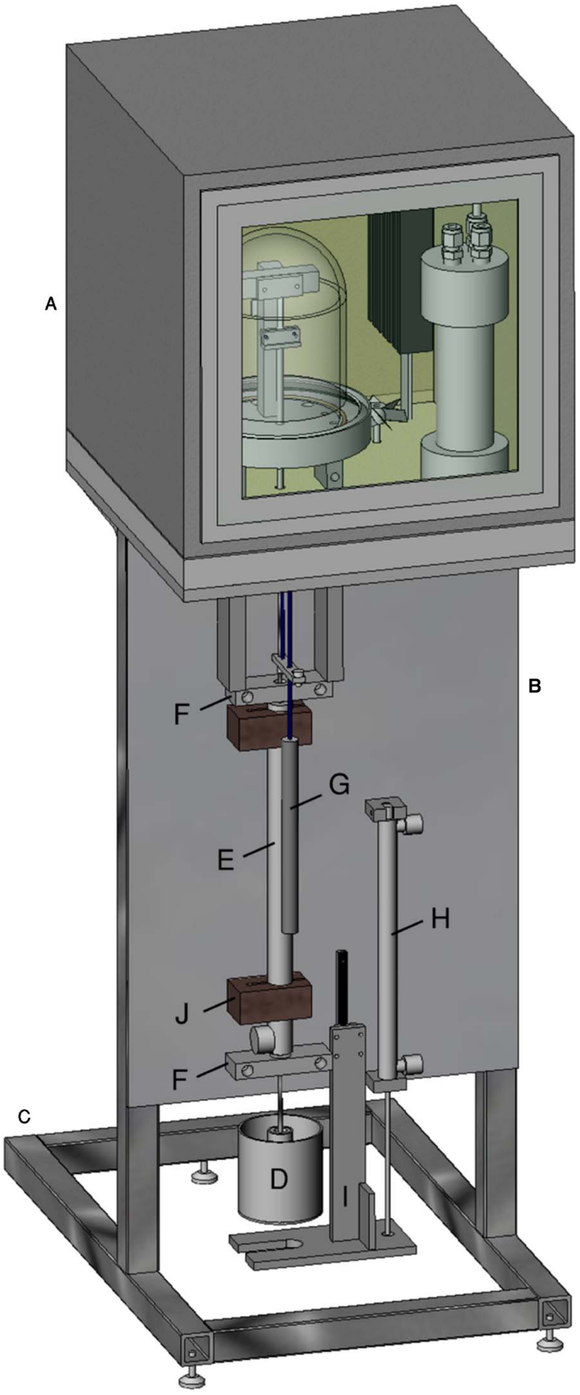

FIG. 2. (Color online) Schematic of creep instrument with environmental control: (A) environmental chamber, (B) vertical mounting plate, (C) frame, (D) variable weight, (E) LVDT, (F) rod guide blocks with PTFE bushing, (G) counterweight, (H) linear pneumatic actuator, (I) stage for applying and removing weight from sample, and (J) phenolic LVDT mounts. A photograph of the working prototype used for the experiments described in this article can be found in the online archive for this journal.

translates into a maximum strain of 2.25 for a typical 1 in. long initial sample length.

Components of the creep instrument are aligned and held vertically by mounting on an aluminum plate held vertically on a frame with leveling feet. A sliding stage, mounted on a guide rail parallel to the axial direction of creep, is used to apply and remove the mass from the sample. A pneumatic linear motion piston is in place to provide automation of this

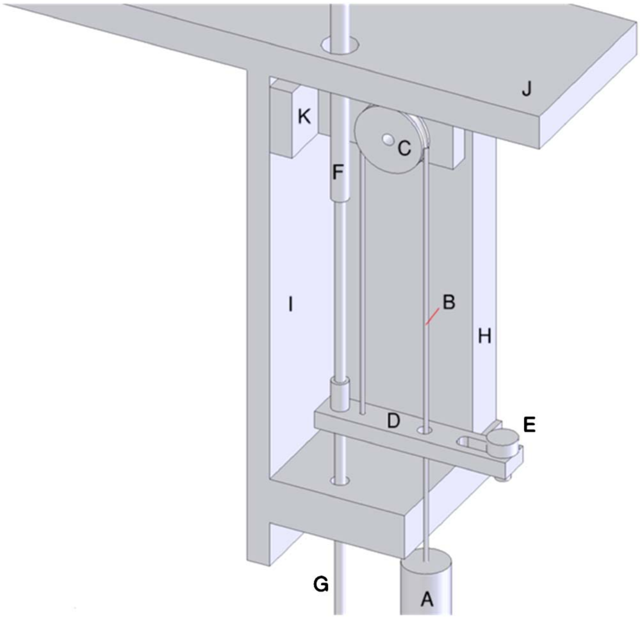

FIG. 3. (Color online) Schematic showing a section view of the counterbalance system used to reduce the minimum tensile force applied to the sample due to the weight of the clamp, LVDT core, and rod. Visible are the (A) adjustable counterbalance mass, (B) nylon rope, (C) nylon pulley, (D) torque zeroing bar, (E) adjustable mass (bolt), (F) 3/16 in. 304 stainless steel (SS) rod (to sample clamp), (G) 4–40 threaded SS rod (to LVDT core), (H) SS humidity chamber support bar, (I) fixed aluminum mounting plate, (J) environmental chamber fixed base, and (K) pulley mounting bracket.

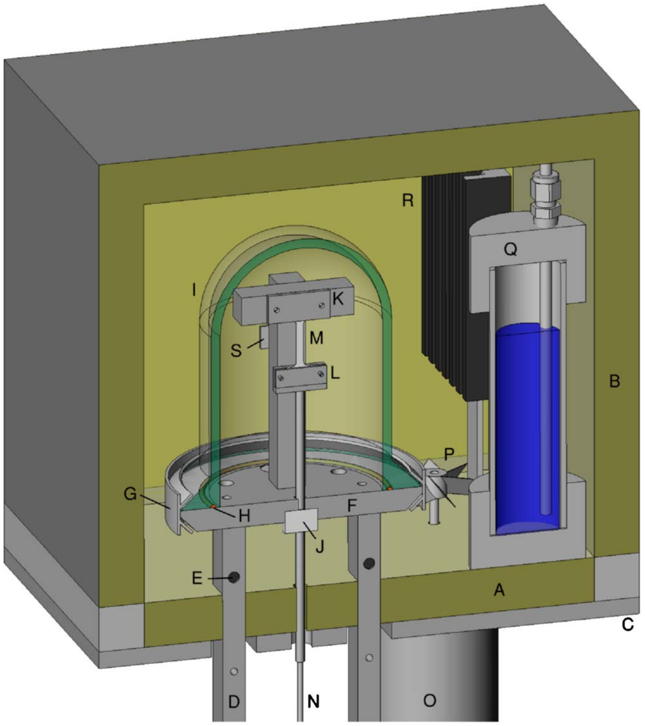

FIG. 4. (Color online) Cross section of the environmental chamber: (A) bottom insulation, (B) removable insulated top, (C) fixed aluminum mounting plate, (D) SS humidity chamber support bars, (E) cartridge heater, (F) SS humidity chamber base, (G) SS Duran clamp, (H) silicone O-ring, (I) glass reaction vessel, (J) PTFE guide bushing, (K) fixed upper clamp, (L) movable lower clamp, (M) polymer sample, (N) 4–40 threaded SS rod (to LVDT core), (O) high temperature fan motor, (P) fan blade, (Q) SS bubbler, (R) finned heater, and (S) dual temperature and relative humidity sensor.

action. In order to reduce the collective mass (~90 g) of the lower sample clamp, LVDT core, and connecting rod which is carried by the sample to the desired mass (~10 g), an adjustable counterweight is used. This is achieved by attaching a balanced (zero torque) cantilevered bar to the sample rod and applying a force opposite in direction to gravity to it by means of the pulley and rope system shown in Fig. 3.

An environmental chamber (Fig. 4) surrounding the sample consists of an insulated outer shell and a humidified inner chamber. The insulated outer shell consists of a stationary base of 3/8 in. aluminum plate and a removable upper box with a double paned glass window on one face and aluminum sheet on the remaining four faces. Both the base and upper box are insulated with 1 in. fiberglass insulation board. The base serves as a fixed mount for sensors and other components needed to maintain an isothermal and isohumid environment. A finned heater and fan together with a proportional integral derivative (PID) temperature controller create an isothermal environment around the humidity chamber. A temperature range of 25–250 °C is possible.[8]

The humidity chamber is made entirely of stainless steel, glass, and PTFE in order to resist corrosion and deterioration from water and other solvents (such as alcohols), even at elevated temperature. These materials were also selected to operate up to 250 °C. The humidity sensor, which has a temperature range of −40–124 °C, is replaced by a $\frac{1}{4}$ in. diameter stainless steel sheathed thermocouple when this range is exceeded. A stainless steel compression fitting with pipe adapter mechanically supports the thermocouple and forms a leak-free seal.

The base of the humidity chamber is made from a circular 304 stainless steel plate with a tapered edge machined to mate with a Duran clamp and a 500 ml Pyrex reaction vessel (Chemglass) which encloses the sample. The rod connected to the movable sample clamp passes through the base with a PTFE bushing in place providing a low friction guide and minimal vapor transport. Alternatively, one could use a stainless steel linear guide bearing or magnetic bearings to further reduce friction. The rod passes through a stainless steel tube spanning the gap between humidity chamber base and the fixed base of the oven to provide a quiescent environment, shielding the rod from convection. Also attached to the base is the cantilevered fixed upper clamp. Pipe to tubing adaptors for gas inlet and outlet as well as a male 3/8 in. pipe plug style feedthrough for a dual humidity and temperature sensor (Sensirion, SHT75) are also included in the base. The silicone O-ring between the reaction vessel and the base forms a good seal when clamped and can withstand temperatures up to 250 °C. The base is supported by two 304 stainless steel bars bolted to the face of the mounting plate. Composite (G-11) spacers insulate the base of the humidity chamber from the support bars. $\frac{1}{4}$ in. diameter cartridge heaters placed in holes in the bars are used to heat the bars to the temperature of the chamber to avoid condensation if very high (>90% RH) relative humidity is required.

Humidity is established in the chamber either by introducing open vials of various salt solutions to the chamber or by flowing humidified N_2 gas from a bubbler. Dry N_2 gas is purged through the chamber when a water-free environment is desired. Vapors other than water, such as alcohols and other volatile organic compounds, can also be introduced to the chamber by either bubbler or liquid-vapor equilibrium. A schematic showing humidity control with a bubbler is shown in Fig. 5. The bubbler is housed within the insulated box and heated by convection. This ensures humidified gas at the same temperature as the humidity chamber and forestalls condensation in lines leading to the chamber. For runs above the solvent's boiling point, the bubbler must be removed from the chamber (to prevent boiling) and heated separately.

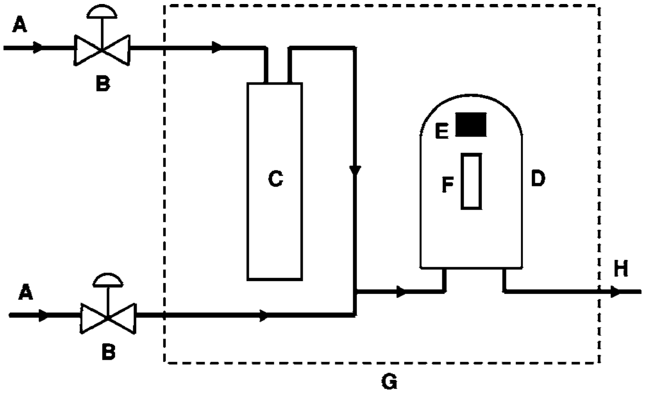

FIG. 5. Schematic of components used to control the water activity in the test chamber surrounding the test sample. The main components are (A) dry N_2 source, (B) mass flow controller, (C) humidifier (bubbler), (D) humidity chamber, (E) dual temperature and relative humidity sensor, (F) test sample, (G) isothermal oven enclosure (dashed line), and (H) gas outlet.

Using the bubbler system for humidification has several advantages over saturated salt solutions. Firstly, saturated salt solutions provide only a small number of water activity values over the range of 6–97% RH whereas the bubbler can give a continuous range of humidity from 0–100% RH. Secondly, vapor pressure above salt solutions is temperature dependent, so different relative humidities will result for a given salt as temperature changes. Finally, the time required for vapor pressure above the salt solutions to reach equilibrium in the chamber is lengthy (hours) while the bubbler system can very quickly (tens of seconds) reach steady state once at temperature.

A LABVIEW program is used to record the length of the sample along with the relative humidity and temperature of the humidity chamber in real time. Data are acquired every second for the first 20 minutes of a creep experiment, and then once every minute thereafter. During creep recovery, data capture rate is again increased to 1 Hz for the first 20 minutes. An LVDT indicator equipped with an RS-232 connection (Macro Sensors, DMI-A1-101) provides a conditioned excitation voltage for the LVDT, converts the resulting voltage into a position measurement, and interfaces with a computer for data acquisition. The LVDT together with controller give a position reading over a range of 4 in. to within ±0.0003 in. (±8 μm).

D. Dynamics of dimensional change

A salient feature of the instrument is the capability of monitoring, in real time, the changes in sample length in response to changes in temperature and water activity. The

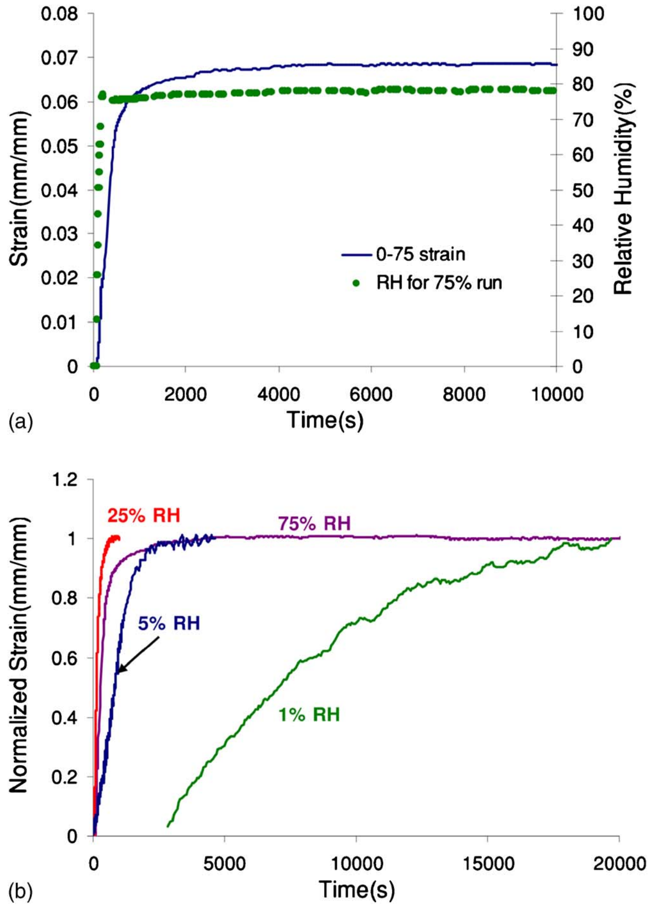

FIG. 6. (Color online) Dynamic swelling response of Nafion during sorption of water from vapor. Plot (a) shows the time required to change the relative humidity in the chamber from 0% to 77% relative to sorption dynamics. Plot (b) compares strain normalized by total strain at equilibrium for sorption of water at different water activities. Note that strain is the result of swelling due to the polymer sorbing water and that no creep is occurring since only a small stress is applied to pull sample straight.

sample is always mounted in the environmental chamber at ambient conditions with no applied stress. The environmental conditions of temperature and water activity are adjusted to the desired values. During this time, the length of the sample is followed to determine when the sample has equilibrated with its environment. These dynamic measurements provide an excellent indication of the equilibration of Nafion. We have found that it is critical to permit sufficient time for equilibration with the environment before beginning mechanical testing; water sorption can take 10^3–10^4 s to equilibrate in Nafion films.

The dynamics of change in sample length to a step change in water activity provides a measure of the transport and reaction associated with water sorption. Figure 6 shows the dynamics of the change in length of a Nafion film to a step change in water activity. The active control of water activity with the bubbler-flow system provides quick and precise control over the water activity needed for these experiments. A series of measurements taken as a function of water activity can be used to obtain an equilibrium linear swelling curve (Fig. 7). Length change dynamics can be used in a similar fashion to obtain coefficients of thermal expansion.

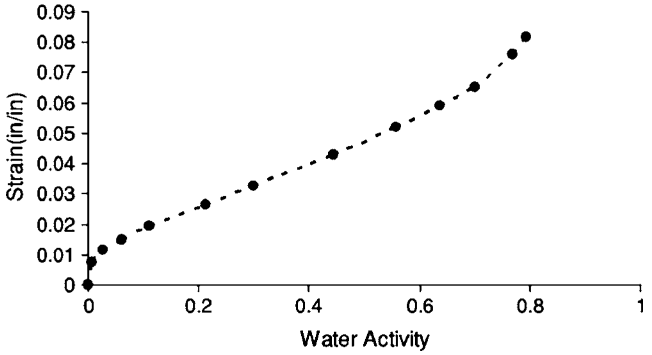

FIG. 7. Equilibrium dimensional change (strain) of Nafion N1110 at 80 °C as a function of relative humidity due to swelling. No stress is applied.

III. RESULTS AND DISCUSSION

A. Tensile creep response procedure

Tensile creep was measured for Nafion from 25 to 108 °C at 0%, 8%, and 65% RH according to the procedure outlined below. Air dried samples, prepared according to the procedure given above, were clamped in the jaws of the creep instrument and dried *in situ* at 85 °C for all runs. After drying, the temperature of the environmental chamber was changed to the desired test temperature. Once at or below the test temperature, water vapor was introduced. Saturated LiBr and NaCl solutions were used to obtain the 8% and 65% RH states, respectively. Sample length was monitored as a way of determining when the samples reached equilibrium with the surrounding environment. Figure 8 is a plot showing sample length as the temperature and relative humidity of the chamber are changed during pretreatment.

The procedural points of drying at 85 °C and then introducing water vapor only after cooled to the test temperature were developed to ensure that history effects were consistent between runs. This is important since drying temperature and the temperature at which water vapor is introduced can affect microstructure and/or hydration levels which give rise to differences in mechanical properties for Nafion. (This is similar to what occurs when water and heat act to reversibly plasticize fibers in fabrics;[9] elevated temperature and the presence

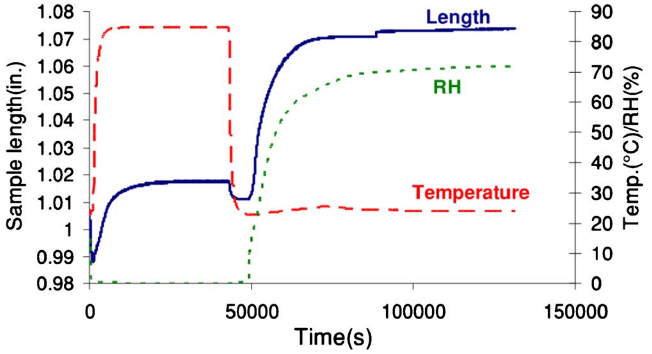

FIG. 8. (Color online) Pretreatment of Nafion *in situ* in preparation for creep run. The sample is first dried at 85 °C in the presence of a flow of dry N_2, then cooled to the test temperature of 25 °C while still dry before finally introducing an open vial of saturated NaCl solution to the chamber to bring the relative humidity to 70%. Sample length along with the temperature and relative humidity of the chamber are shown.

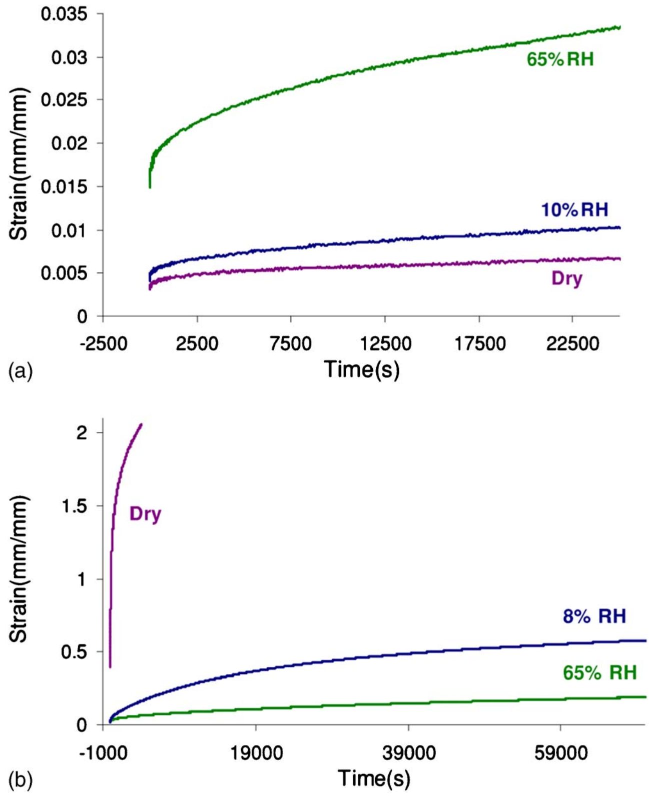

FIG. 9. (Color online) Creep strain of Nafion N1110 at 25 °C (a) and 90 °C (b) for three relative humidities and an applied stress of 1.55 MPa.

of water decrease secondary bonding forces, allowing chain motion which does not occur at lower temperatures and without water.)

After the sample reached an equilibrium length while being held at the desired temperature and water activity, force was applied to the sample. To get the desired stress of 1.55 MPa for samples with dimensions of 0.010×0.500 in.^{2}, a weight of 510.25 g was used. The runs were initiated by quickly and smoothly lowering the mass onto the rod connected to the bottom clamp.[10] Sample length was monitored and recorded as a function of time along with the temperature and humidity of the chamber. Creep response for Nafion N1110 at 23 °C and three relative humidities is shown in Fig. 9. In this plot one can see that the creep response increases with hydration. At 25 °C, water plasticizes Nafion, increasing creep strain. Comparing these curves to the same runs at 90 °C (Fig. 9), it is apparent that the opposite trend is present; creep strain decreases with hydration at 90 °C. A full explanation of this phenomenon is beyond the scope of this publication and will be reported elsewhere in the literature by the authors.

Elastic modulus is shown for Nafion at 23, 40, and 80 °C as a function of water activity over the range of 0–0.95 (Fig. 10). Modulus was measured from the instantaneous response resulting from the application and removal of stress in 30 s cycles. The same sample was used for all points since the instantaneous response is not affected by viscous loss, which was small. The plot of elastic modulus echoes what we see in the creep response of Nafion (Fig. 9); water plasticizes Nafion at low temperature (23 and 40 °C) while it increases resistance to strain, relative to the dry state, at higher temperatures. In other words, adding water to dry Nafion at 23 and 40 °C decreases the elastic modulus while it increases elastic modulus at 80 °C. This is truly an interesting result. We also notice that the first waters added to dry Nafion have the largest effect on the elastic modulus at all temperatures shown.

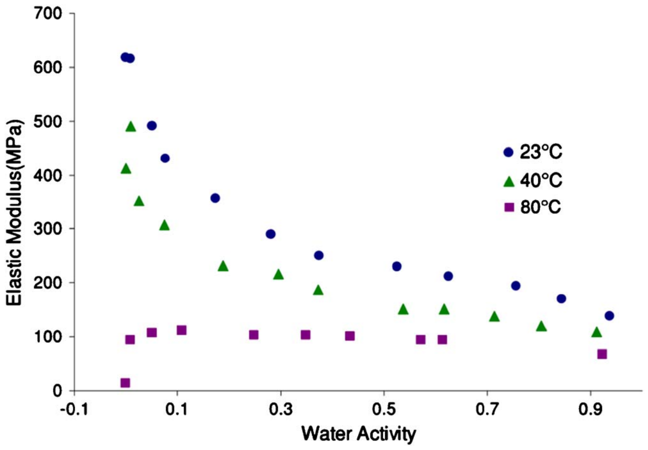

FIG. 10. (Color online) Elastic response of Nafion N1110 measured at 23, 40, and 80 °C from 0% to 95%RH. The elastic modulus was determined from the instantaneous response induced when a stress was applied and removed. All strains are below the proportional limit of the material.

B. Solvent effects

As mentioned previously, solvents other than water will affect mechanical properties of certain materials. The simplicity of the environmental chamber along with the selection of construction materials permits the substitution of a wide range of solvents for water.

For the case of Nafion, the effects of four solvents on creep response are compared to hydrated Nafion (Fig. 11). From these experiments, we can obtain insight to the microstructure and intermolecular forces of the material. Solvent activity approached a value of 1 for all runs. This was done by introducing open vials of solvent to the inner chamber and allowing the vapor pressure of solvent in the chamber to

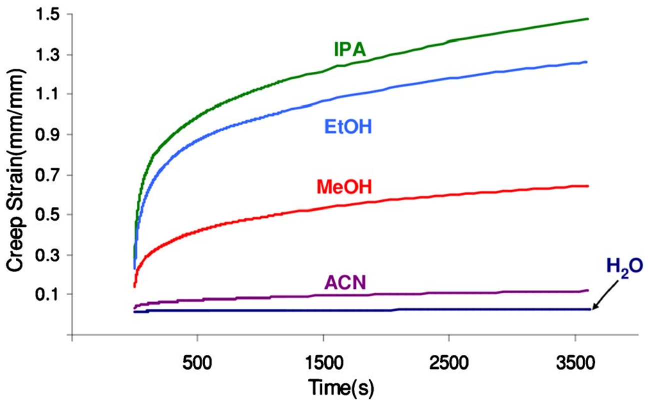

FIG. 11. (Color online) Creep response of Nafion when swollen with different solvents; IPA=isopropyl alcohol, EtOH=ethyl alcohol, MeOH=methanol, and ACN=acetonitrile. All solvents were introduced to the sample after drying and were introduced as vapor with activity approaching a value of 1. Applied stress for all runs was 0.95 MPa.

reach equilibrium overnight. Sample length was monitored during this time as an indication of progress towards equilibration.

Isopropyl, ethyl, and methyl alcohols all served to highly plasticize Nafion, as evidenced by the large creep responses relative to the other curves. Acetonitrile increased creep over the dry state (not shown), but not much beyond water. Ethanol, methanol, and isopropanol are all polar protic solvents with polar and nonpolar ends. The fact that the alcohols are polar allows them to be sorbed by the acid clusters of the Nafion while the nonpolar ends disrupt the secondary bonding between chains, allowing chains to slip past one another. Acetonitrile, on the other hand, is polar and aprotic. Therefore, we would expect less of an interaction with acetonitrile since it is less easily incorporated into the acid clusters and thus less intimate with the Teflonic regions.

C. Uptake dynamics

The dynamics of water uptake was measured for Nafion 1110 at 50 °C by monitoring the change in length of the sample (strain) in the absence of an applied stress (Fig. 6). Only a small stress was applied; enough to pull the sample taught, but not enough to induce creep. For the curves shown, Nafion was first dried in the instrument. At time zero, the relative humidity in the chamber was changed rapidly to the desired level. Figure 6(a) shows that the time required to change the relative humidity in the chamber from 0% to 77% is fast (by an order of magnitude) compared to the time required for sorption to occur. Water sorption at several water activities is shown. It can be seen that the time for swelling to occur is much faster for high water activities than for very low water activities. Swelling at 5% and 1% RH is very slow, taking over 24 h for the case of 1% RH. Slow swelling for drier membranes can be explained by either slow diffusion or slow molecular rearrangement. To give a feel for the sensitivity of the instrument, we point out that the total strain for the 1% RH curve is 0.2% which corresponded to a total length change of 0.002 in. (50 μm) for the sample which was 1 in. long initially. This measurement was repeatable.

The equilibrium strain of Nafion due to water uptake is shown at 80 °C as a function of water activity (Fig. 7). These data are very similar to equilibrium mass uptake as a function of water activity for Nafion (a mass uptake plot for Nafion can be found in the work of Yang *et al.*[11]). The similarity between equilibrium mass uptake and strain for Nafion suggests that swelling controls mass uptake and gives a direct indication of mass uptake.

IV. CONCLUSIONS

The instrument presented in this publication was used for tensile creep testing of a polymer under a well-controlled environment of temperature and relative humidity. With this instrument, the temperature and relative humidity surrounding the sample was maintained within $\sim\pm 0.2$ °C and $\sim\pm 0.7\%$, respectively. The design of the instrument allowed for *in situ* monitoring of the sample length during equilibration and as a result of changes in temperature and hydration. Although originally designed for testing polymer membranes for fuel cells, the instrument has utility in many other areas. Furthermore, the general design concept used to obtain stable environmental conditions during tensile creep tests can be easily applied to other mechanical testing methods including DMA and tensile testing.

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[1] Activity is a measure of the concentration of a component in the liquid or vapor phase relative to the saturated concentration of the pure substance at that temperature. In the vapor phase, activity is defined as the ratio of vapor pressure of the component in the mixture to the vapor pressure of the pure substance at that temperature. Relative humidity, which refers specifically to water vapor, is defined as water activity × 100.

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[7] Y. M. Haddad, *Viscoelasticity of Engineering Materials* (Chapman and Hall, London, 1995), pp. 33–35.

[8] Temperatures lower than ambient can be achieved by replacing the heater element with a liquid to air heat exchanger. Flowing a cooled liquid from a chiller or using liquid nitrogen will allow access to stable low temperatures. However, controlling relative humidity at low temperatures becomes difficult.

[9] S. L. Rosen, *Fundamental Principles of Polymeric Materials*, 2nd ed., (Wiley, New York, 1993), p. 98.

[10] Application of the weight at the start of a creep run must be done slowly enough to avoid a spike in stress (beyond the desired static stress) caused by impact of the weight with the rod. It must also be done with sufficient speed to be completed in less than 2 s so that (1) instantaneous elastic response can be separated from the delayed elastic and viscous components and (2) creep strain is occurring under the full static stress equal to the force applied by the entire mass divided by the cross sectional area of the sample. The initial strain rate of the sample under the applied stress dictates the slowest acceptable speed for lowering of the weight. A reasonably wide range of lowering speeds gave repeatable results. Only when initial creep rate was very large ($>20\%$ per second) did repeatability become an issue, a point beyond the region of linear response of most materials. We found that lowering the mass by hand gave the most flexibility.

[11] C. Yang, S. Srinivasan, A. B. Bocarsly, S. Tulyani, and J. B. Benziger, J. Membr. Sci. **237**, 145 (2004).