Characterization of the Electromechanical Properties of EAP materials

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ABSTRACT

Electroactive polymers (EAP) are an emerging class of actuation materials. Their large electrically induced strains (longitudinal or bending), low density, mechanical flexibility, and ease of processing offer advantages over traditional electroactive materials. However, before the capability of these materials can be exploited, their electrical and mechanical behavior must be properly quantified. Two general types of EAP can be identified. The first type is ionic EAP, which requires relatively low voltages (<10V) to achieve large bending deflections. This class usually needs to be hydrated and electrochemical reactions may occur. The second type is Electronic-EAP and it involves electrostrictive and/or Maxwell stresses. This type of materials requires large electric fields (>100MV/m) to achieve longitudinal deformations at the range from 4 - 360%. Some of the difficulties in characterizing EAP include: nonlinear properties, large compliance (large mismatch with metal electrodes), non-homogeneity resulting from processing, etc. To support the need for reliable data, the authors are developing characterization techniques to quantify the electroactive responses and material properties of EAP materials. The emphasis of the current study is on addressing electromechanical issues related to the ion-exchange type EAP also known as IPMC. The analysis, experiments and test results are discussed in this paper.

Keywords: EAP, Characterization, Testing, Electromechanical Properties, Electroactive Polymers, Actuators

1. INTRODUCTION

Electroactive polymers (EAP), which are an emerging class of actuation materials, have many attractive characteristics [Bar-Cohen, 2001]. Implementing these materials as actuators requires the availability of properties database and scaling laws to allow actuator or transducer designers to determine the response at various operation conditions. A metric for the comparison of these materials' properties with other electroactive materials and devices is needed to support users in making these materials as actuators of choice [Stewart and Bar-Cohen, 2001]. In selecting characterization techniques it is instructive to look at the various Electroactive Polymers and the source of their strain-field response and two main classes can be identified:

1. Electronic EAP Materials – These are mostly materials that are dry and are driven by the electric field or Coulomb forces. This category includes piezoelectric, electrostrictive and ferroelectric materials. Generally these materials are polarizable with the strain being coupled to the electric displacement. The strain of electrostrictor and ferroelectric materials is proportional to the square of the polarization or electric displacement. In piezoelectrics materials the strain couples linearly to the applied field or electric displacement. Charge transfer in these materials is in general electronic and at DC field these materials behave as insulators. These properties have been studied for over a century in single crystals and for over 3 decades in polymers. Another group of EAP that belongs to this class is the dielectric EAP which is a material that is highly elastic and is pressed by the Coulomb forces in reaction to the presence of electric field.
2. Ionic EAP Materials – These materials usually contain an electrolyte and they involve transport of ions/molecules in response to an external electric field. Examples of such materials include conductive polymers, IPMC, and ionic gels. The field controlled migration or diffusion of the various ions/molecules results is an internal stress distribution. These internal stress distributions can induce a wide variety of strains from volume expansion or contraction to bending. In some conductive polymers the materials exhibit both ionic and electronic conductivities. These materials are relatively new as actuator materials and have received much less attention in the literature than the piezoelectric and electrostrictive materials. At present, due to a wide variety of possible materials and conducting species, no generally accepted phenomenological model exists and much effort is underway to determine the commonalties of the various materials systems. A clearer understanding of the characterization techniques would help immensely in determining underlying theories and scaling laws for these actuator materials.

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2. CHARACTERIZATION OF EAP WITH POLARIZATION DEPENDENT STRAINS

A significant body of knowledge is available for the characterization of polar polymer electromechanical materials. This includes general information garnered from other electro-mechanical materials as well as a significant body of work dealing with polymer transduction materials in the last thirty years. The characterization of the material properties of polar materials involves both the acquisition of the data (e.g., strain, stress, charge and field) and interpretation of the data using the appropriate constitutive equations.

2.1 PIEZOELECTRICITY

2.1.1 Resonance Analysis

The most widely used technique for measuring the material constants of piezoelectric materials is the resonance method, which is outlined in the IEEE Standard on Piezoelectricity [1987]. A piezoelectric sample of specific geometry is excited with an AC voltage. The phase and the magnitude of the current with respect to the excitation voltage are monitored and the AC impedance of the sample as a function of the frequency of the AC voltage is found. The impedance spectrum is complex with both a resistance R and a reactance X. The impedance spectra contain resonances, which are the result of ultrasonic standing waves in the piezoelectric material. The parallel resonance frequency f_p is defined to be the frequency at which a maximum in the resistance occurs. The sideband frequencies $f_{+1/2}$ and $f_{-1/2}$ occur at the maximum and minimum of the reactance respectively. The impedance equation governing resonance spectra similar to the spectra are derived from phenomenological theory based on the linear equations of piezoelectricity and the wave equation. The previous derivation of the thickness, thickness shear, length and length thickness impedance equations by Berlincourt, Curran and Jaffe [1964] were for materials with real material constants (loss-less materials). Holland [1967] showed that the losses of a piezoelectric material could be taken into account by representing the material constant as a complex quantity in the frequency domain. In the following sections losses are accounted for by the addition of an imaginary component to the various material constants.

2.1.2 Quasi-static Measurements

The linear model of piezoelectricity cannot explain some of the behavior of piezoelectric materials and non-linear effects have been reported including Berlincourt and Krueger [1959] looked at the general aspects of non-linearity as a function of stress and field while Krueger [1968] and others studied the stress dependence of the material properties of piezoelectric ceramics. Recent work by Vinogradov [1999] investigated the mechanical and viscoelastic properties of PVDF as a function of stress, time and temperature. The majority of the studies quoted above were done under quasi-static conditions where a stress or electric field excitation is applied over some time and the properties are monitored as a function of the stress or electric field. Generally, the piezoelectric coupled linear equations ignore an important variable associated with ferroelectric hysteresis. A mixed measurement is possible but technically the problems of measuring two variables while adjusting two other variables is quite difficult and problems arise in the interpretation of the results. There is no sound reasoning to suggest that these effects are independent and in the case of ferroelectric materials it is highly likely that they are not. If the electric field is set to zero and the strain and electric displacement are monitored as a function of the stress, different results would be expected than if both a stress and electric field are applied since both stress T and field E can affect the polarization in the material. A variety of instruments can be used to measure the strain and include interferometers, capacitance dilatometers, linear variable displacement transducers, optical levers and fiber optic sensors and direct optical methods. Tiersten [1981] and others suggested to study the piezoelectric impedance resonance under high electric bias field. These studies looked at single crystal piezoelectric materials and the effect of the bias field on the resonance frequencies. In all cases the materials were assumed to be loss-less and in some of the studies the field dependence of the piezoelectric and dielectric coefficients was assumed to be small. The use of a DC bias field with a small AC field is very sensitive to non-linearities because the measurement is differential. It should be noted that only reversible contributions to the piezoelectric, elastic and dielectric response are measured. The DC impedance of the samples is usually large and heating effects due to the DC field are much smaller than for an AC field of the same order therefore the isothermal conditions used to derive the equations describing the experiment are maintained. The technique requires additional protection circuitry to isolate and protect the impedance analyzer from the DC field [Sherrit and Mukherjee, 1998]. Since this technique is more suited for studying nonlinear behavior we discuss the technical details of the measurement in more detail in the next section.

2.2 QUADRATIC RESPONSE - MAXWELL STRESS, ELECTROSTRICTION

Typical plots of the response to excitation by an electric field of an electrostrictive material for a linear and non-linear dielectric are shown in Figure 1 below. In both cases the strain versus electric displacement are quadratic. The strain versus electric field for the non-linear dielectric displays saturation and higher order even terms in field are required to model the strain-field behavior. It should also be noted that any hysteresis (loss) in the linear dielectric term would produce a hysteresis in the strain field data independent of any loss in the electrostrictive coefficient Q. The response shown assumes no hysteresis in the electrostrictive Q coefficient. Losses in the electrostrictive Q coefficient may be represented by a complex electrostrictive constant $Q = Q_r + iQ_i = |Q| e^{i\theta}$. The addition of this phase can be shown to introduce hysteresis in the strain – electric displacement plots.

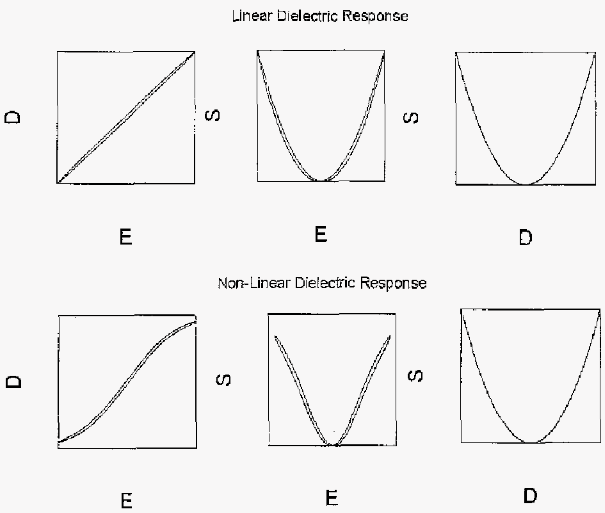


FIGURE 1: The electrostrictive response to a linear and non-linear dielectric material. The strain is quadratic in D however in the nonlinear dielectric material saturation is seen in both the electric displacement and the strain as a function of the electric field.

The thermal stress correction for an isotropic dielectric is dependent on the thermal boundary conditions (isothermal / adiabatic), coefficient of thermal expansion and heat capacity [Kloos, 1995]. Typically this correction is quite small however if the coefficient of thermal expansion is large or the heat capacity (constant stress) is small the order of this correction should be calculated to determine its significance. In some elastomers the quadratic response is due primarily to the presence of the Maxwell stress in the material [Zhang and Scheinbeim, 2001; Kornbluh and Pelrine, 2001; and Su, et al, 2000]. These materials can exhibit very large lateral strains of the order of 10 - 215%. It should be noted that when dealing with strain levels of this order that the engineering strain approximation ($S = \Delta l/l$) is no longer valid approximation to the Lagrangian/Eulerian strain [Saada, 1974] and one should use $\Delta l/(l+\Delta l)$ as was noted by Kornbluh and Pelrine [2001]. It should be kept in mind that due to the coupling between the longitudinal and transverse strains that an area correction is required to determine the proper dielectric response since the capacitance is dependent on the area of the film. A variety of technical issues arise when trying to characterize these materials. The primary problem is the large dispersion that is present [Zhang, et al, 1997] in the elastic, dielectric, and electrostriction constants. The properties change as a function of frequency and unless the specific relaxation mechanism is known one cannot in general extrapolate results measured at one frequency to other frequency ranges. This requires characterizing the material over the frequency range to be used by the transducer/actuator in order to correlate measured material properties to performance of the transducer or actuator. Another complication is at higher field levels higher order terms in the thermodynamic potentials will affect the overall response of the material. In quasi-static measurements these would be seen as saturation in the response of a Strain field plot or frequency components in the strain time curve that are greater than 2ω assuming the applied field is $E_0\cos(\omega t)$. One approach that has been used to characterize electrostrictive ceramics for high frequency transducer materials is the biased resonance measurement. By analyzing the impedance resonance curves and plotting the results as a function of frequency one can separate the dependencies of the elastic, dielectric and induced piezoelectric constant on the applied field [Sherrit and Mukherjee, 1998]. It should be noted that under a large bias field the material would no longer be isotropic but rather isotropic in the plane perpendicular to the bias field. As in the case of the quasi-static measurements the geometry (area and thickness) must be known as a function of the field to evaluate the fields and material constants when the quasi-static strains exceed 1%.

2.3 HIGHER ORDER EFFECTS - FERROELECTRICITY

Although the phenomena of ferroelectricity in not generally used to couple electric to mechanical energy a switching strain is associated with a ferroelectric material driven to field levels above its coercive field. In general this data is useful for the transducer/actuator designer since it put limits on the size of the AC drive field or alternatively determines the size of the bias field required to inhibit switching. The electric displacement as a function of the electric field is typically hysteretic and is characterized by the coercive field E_C, saturation D_S and remnant D_r displacement. The strain response is characterized by the switching strain ΔS and the coercive field.

3. CHARACTERIZATION OF IONIC EAP WITH DIFFUSION DEPENDENT STRAIN

Characterization of the properties of the ionic EAP materials, which involve diffusion dependent strain, is posing unique challenges to the development of test methods [Bar-Cohen, 2000]. This emphasis of this discussion is on ionomeric-polymer metal composite (IPMC) consisting of Nafion® [Tant, et al, 1997] or Flemion® [Oguro, et al, 1999] as membranes made of fluorocarbon backbones and mobile cations (counter-ions). The exact mechanism that is responsible for the electro-activation is still a subject of a series of studies. However, recently significant progress has been made towards understanding the related phenomena [Nemat-Nasser and thomas, 2001]. When a voltage (<5V) is applied to a hydrated IPMC sample, the large ionic conductivity may promote electro-osmosis and/or hydrolysis. The former response manifests itself as a bending of the film towards the positive electrode (anode) and can be exploited in actuation applications [Sewa, et al 1998]. The induction of electrolysis is an undesired electrochemical reaction that consumes power and may damage the electrode by producing gas. Kanno, et al [1994] have shown that the bending response of Pt -electroded Nafion (Na+ counter-ion) is complicated by relaxation processes. If a DC voltage is applied for sufficient time duration, the primary deflection will gradually return to its initial position. This phenomenon is thought to be due to the excess concentration of water near the cathode and its subsequent back-flux [Okada, et al, 1998]. It is interesting to note that this behavior is not evident in Au –electroded Flemion (tetra-n-butylammonium counter-ion) [Oguro, et al, 1999]. The large size of the cation and its sluggish mobility may provide an explanation. The large bending deflections, the required hydration, and the relaxation processes that are involved with IPMC electroactivation make the task of electromechanically characterizing such materials difficult. The focus of the author effort was on testing the response of gold-electroded Flemion (tetra-n-butylammonium counter-ion). Similar tests can be applied to other ionic EAP materials, such as polypyrrole [Otero and Sasinena, 1997] and also electronic EAP materials.

3.1 MICROGRAPHY

Micrography is a well-established field and a large number of test methods is available for the examination microscopic details of test objects. Various methods are used including optical, such as visual microscopy, as well as enhancement imaging allows seeing details beyond the capability of visual technique. Scanning Electronic Microscope and other derivatives are examples of such techniques. The use of such techniques has a great significance when examining ionic EAP material since there is a lot of insight that can be gained into the microstructure supporting the efforts to understand the mechanism of operation; determine the quality of the material; and assure the conformance to a standard once it is established. A human operator can perform the evaluation of images particularly at the stage of research when there is a need to understand the characteristic structure when there is no sufficient database.

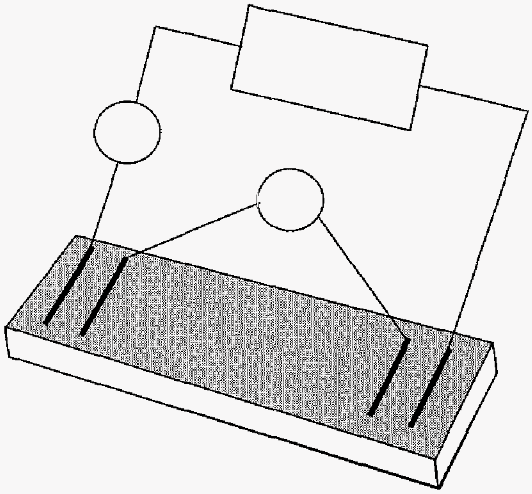
3.2 Voltammetry

Voltammetry refers to techniques in which the relationship between voltage and current is observed during electrochemical processes. The voltage is applied and the current is measured. A plot of the current versus the voltage is called a voltammogram. Peaks in these curves represent electrochemical reactions. Voltammogram are used to access reaction/deposition rates, reversibility, and reaction potentials. The voltage sweep rate can be adjusted to determine relaxation processes of deposition. In addition to the electrochemical reaction, the electronic conductivity and capacitance can also contribute to the shape of the voltammogram and these parameters need to be taken into account.

3.3 Sheet Resistance

Sheet resistance is an indicator of the quality of the electrodes and it is commonly measured using a four-probe system and a schematic view is shown in Figure 2. This test system is ideal for measuring the sheet resistance or conductivity of metal films. A current supply forces current through the sheet electrodes. This arrangement allows for an accurate determination of the impedance of a conductive material by eliminating contact resistance from the measurement. The outer current electrodes are used to force a current through the sample. The inner electrodes measure the voltage drop between two fixed points on the sample. Since the input impedance of the voltage probes is very large compared to the voltage drop due to contact resistance an accurate measurement of the sample impedance can be made while excluding the contact resistance.

FIGURE 2: Schematic diagram of a 4 probe sheet resistance measurement.



Large electrode resistance can be caused by poor conductivity, insufficient electrode thickness, micro-cracks in the electrode and inhomogeneous deposition. Large cyclic tensile and compressive stresses on the electrode during bending may cause fatigue and further increase the sheet resistance.

3.4 MECHANICAL TESTING

Mechanical testing of polymers involves measuring the stress-strain behavior as a function of frequency *f*, temperature ϑ, stress T, time t and relative humidity for ionic EAP. A variety of standard tests are available for the mechanical testing of polymers for various properties. These include:

- Stress Analysis
- Ultimate Strength
- Energy Dissipation and Damping
- Impact Testing
- Fatigue Behavior
- Elasticity
- Glass Transition and Thermal Behavior
- Creep

The American Society has published variety of standards for Testing of Materials [www.ASTM.org] including ASTM Standard E1640-99, ASTM Standard D4065-95, and ASTM Standard - D6049-96. Also, number of books on mechanical characterization of polymers has been published by [Swallowe, 1999, Ward, et al 1993 and Lakes 1998]. The mechanical properties of EAP are tested in a similar manner to other polymer materials, however in the case of IPMC and other ionic EAP materials the relative humidity has to be controlled or accessed during the experiment [Yeo and Eisenberg, 1977; & Nakano and MacKnight, 1984].

3.5 DISPLACEMENT MEASUREMENT TECHNIQUES

Although IPMC show longitudinal and transverse strains under the application of the applied voltage, the effect is found to be much smaller than the developed bending strain. Measurement of the small longitudinal and transverse strains can be accomplished using the same apparatus that is used for piezoelectric materials however the measurement procedure for IPMC is complicated by the need for a "wet" system. The material demonstrates a hysteresis when subjected to applied voltage as shown in Figure 3 for 1.0V, 2.0V, and 3.0V at 0.05Hz, probably due to the large size and low mobility of the tetra-n-butylammonium counter-ion. The relaxation process is quite different than that observed for Pt-Nafion (Na^+ counter-ion) [Kanno, et al, 1994]. Tip force was also measured after the sample was removed from water and 3mm of its free end was in contact with load cell. A 5 V, 0.05 Hz cosine wave was applied and using sampling frequency of 1.0 Hz the measured force is shown in Figure 4 indicating a small force (~0.6mN peak). The horizontal line in the plots represents the initial displacement of the sample before voltage was applied.

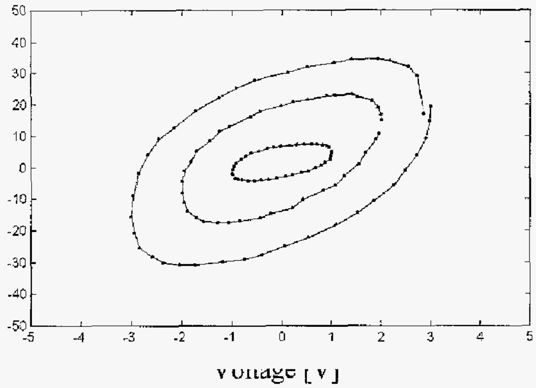


FIGURE 3: Hysteresis of tip displacement for 1.0V, 2.0V, and 3.0V at 0.05Hz.

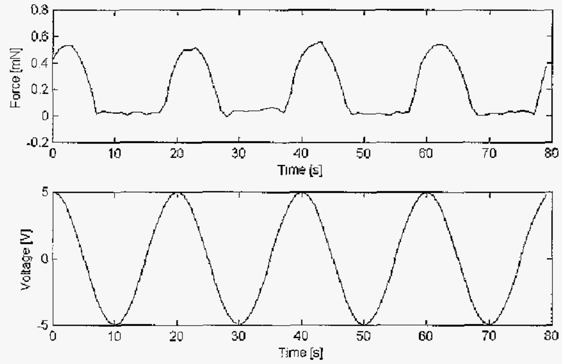


FIGURE 4: Tip force and applied voltage.

In order to measure the tip displacement and the effect of tip-mass loading an experimental setup was constructed as shown in Figure 5 and 6. A function generator and an amplifier were used to subject samples to low frequency square voltage signals (0.1-Hz) and the observed deformation is digitized using a video camera and an image-processing algorithm.

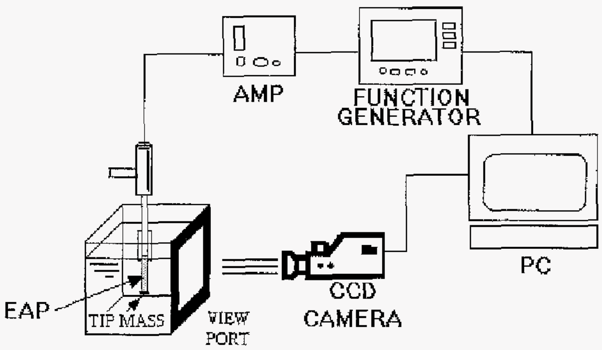


FIGURE 5: Schematic view of the test setup.

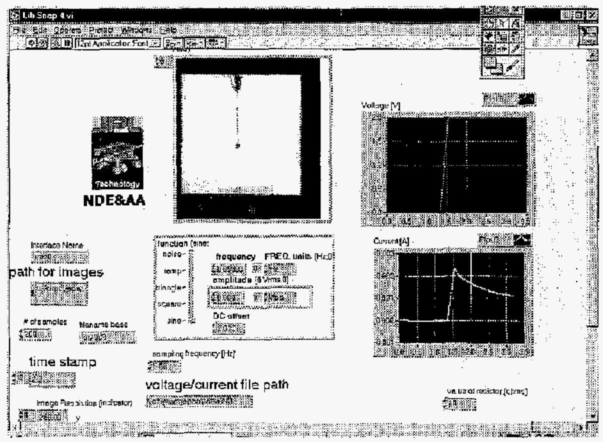


FIGURE 6: The data acquisition display. The window in the middle of the display shows tip mass mounted on the IPMC sample.

An edge detection algorithm is used to acquire at sequential-time-intervals the deformed shape of the EAP sample and then a curve fitting is used to determine the sample's geometry, slop, and curvature. Attempts to obtain consistent results were hampered by the fact that IPMC sustains irreversible shape changes under mono-polar activation (i.e., square wave). Further, the material properties are affected substantially by the material wetness, ionic constituents, temperature, off-axis deformation, dimensions of the sample as well as loading distribution and constraints. IPMC strips were loaded with a tip-mass and the curvature was determined at 0.1-Hz and image acquisition rate of 15 frames/sec.

3.5.1 Phenomenological Model And Analysis

The macro-mechanical behavior of the IPMC was modeled assuming a bending beam and the experimental data was analyzed using this model [Bhattacharya et al., 2001]. Since IPMC undergoes large deformations, traditional linear Euler-Bernoulli model does not describe very well the finite deformation of this bending. Our recent approach has been to use a nonlinear Euler-Bernoulli beam model, augmented with an eigen-strain. Consider the strip shown in the Figure 7 and assuming that initially the applied force F is zero. In response to an applied electric field, the strip bends and as observed in our experiments a pristine sample has a uniform curvature that can be traced as an arc (subjecting the sample to an electric pulse leaves permanent deformation). We call this curvature the load-free

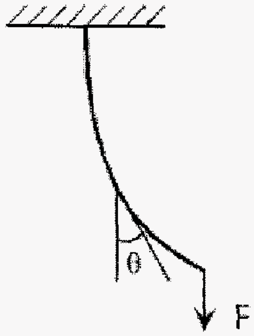


FIGURE 7: Model EAP strip/beam.

curvature, material curvature or eigen-curvature κ. If we apply a step voltage to the beam, this curvature changes with time by applying different time-dependant voltages and different forces and fitting them to the model above, we can evaluate all the material constants E, c and τ. This model can be easily adapted to many complicated loadings and applied voltages and it was used to determine the characteristics of IPMC samples and the obtained data is listed in Table 1.

TABLE 1: Measured properties for IPMC

Maximum performance			**Potential capability**		**Design properties**		
Max Bending Curvature [1/mm.V]	Max strain 10^{-3}[%]	Max Stress [MPa]	Max Mechanical Energy density [x10^4 J/m^3-V]	Max Conversion Efficiency [%]	Modulus Young Modulus [MPa]	Specific Density [g/cc]	Response Time (1/bandwidth] [Sec]
0.035-0.040	2.3 –2.6	0.2 - 0.3	0.6-1.6	0.1-0.2	70 - 140	2.5-2.9	1-12

TABLE 2: The properties that need to be characterized for EAP materials and the assumed metric.

Measurement		**Properties**	**Metric**
Mechanical		Tensile strength [Pa]	Mechanical strength of the actuator material
		Stiffness [Pa]	Required to calculate blocking stress, mechanical energy density, and mechanical loss factor/bandwidth
		Coefficient of thermal expansion [ppm/C]	Affects the thermal compatibility and residual stress
Electrical		Dielectric breakdown strength [V]	Necessary to determine limits of safe operation
		Impedance spectra [ohms and phase angle]	Provides both resistance and capacitance data. Used to calculate the electrical energy density; electrical relaxation/dissipation and equivalent circuit.
		Nonlinear Current [A]	Used in the calculation of electrical energy density; quantify nonlinear responses/driving limitations
		Sheet Resistance [ohms per square]	Used for quality assurance
Microstructure Analysis		Thickness (electrode & EAP), internal structure, uniformity and anisotropy as well as identify defects.	These are features that will require establishing standards to assure the quality of the material
Electro-active Properties	Strain	Electrically induced strain [%] or displacement [cm]	Used in calculation of 'blocking stress' and mechanical energy density
	Stress	Electrically Induced Force [g], or Charge (C)	Electrically induced force/torque or Stress induced current density
	Stiffness	Stress/strain curve	Voltage controlled stiffness
Environmental Behavior		Operation at various temperatures, humidity and pressure conditions	Determine material limitations at various conditions

4. CONCLUSION

Accurate information about the properties of EAP materials is critical to designers who are considering the construction of mechanisms or devices using these materials. In order to assess the competitiveness of EAP for specific applications there is a need for a properties matrix. This matrix needs to provide performance data that is presented in such a way that designers can scale the properties for incorporation into their models of the device under design. In addition, such a matrix needs to show the EAP material properties in such a form that allows the users to assess the usefulness of the material for specific application. This data needs to include properties and information that can be compare with the properties of other classes of actuators, including piezoelectric ceramic, shape memory alloys, hydraulic actuators, and conventional motors. The range of actuation and stress generation of the various types of EAP is quite large and the excitation field that is required for these materials can vary by 5 orders of magnitude.

Some of the macroscopic properties that can be included in the matrix are maximum strain, maximum blocking stress, response time, maximum electric and mechanical energy density as well as maximum energy efficiency. In addition, due to the mechanical interaction that is associated with the electro-reaction there is a need to characterize both the passive and electroactive properties. The properties that may be of significance when characterizing EAP are described in Table 2. While some of the properties (particularly those that are driven by polarization mechanisms) have relatively well-established methods of characterization, the ionic materials and particularly IPMC still require new techniques. These materials pose the greatest challenge to characterization methods developers due to their complex behavior. This complex response is associated with the mobility of the cations on the microscopic level, the strong dependence on the moisture content, as well as the nonlinear and the hysteresis behavior of the material. The technology related to the characterization of EAP is expected to evolve as the field is advanced and standards methods will need to be established in the coming years.

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