UNIVERSIDAD DE INGENIERÍA Y TECNOLOGÍA UTEC

Mechanical Engineering

Estudio de la viabilidad de elastómeros impresos en 3D para su aplicación como Polímeros Electroactivos Dieléctricos

Study of the viability of 3D printed elastomers for their application as Dielectric Electroactive Polymers

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La tesis

Estudio de la viabilidad de elastómeros impresos en 3D para su aplicación como polímeros electroactivos dieléctricos

Ha sido aprobada

[Nombres y apellidos del Presidente de Jurado]

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Dedicatoria:

A mis padres que me dieron la educación que hizo posible esta
experiencia y el coraje para perseguirla

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SUMARRY

The present thesis studies two 3D printed materials to be used as dielectric elastomers, the Formlabs' Flexible resin and the Formlabs' Clear resin. Three properties are tested and reported: Relative Permittivity, Dielectric Strength and the Young's Modulus. In addition to the tested materials, other options for dielectric elastomers are suggested based on the relevant properties according to the electromechanical coupled Maxwell stress in 1D. Finally, a comparative method is used to determine suitability for newer materials as dielectric elastomers.

Keywords: DEA, dielectric elastomer, 3D printing, electromechanical stress.

RESUMEN

La presente tesis estudia dos materiales impresos en 3D para ser usados como elastómeros dieléctricos: La resina "Flexible" de la empresa Formlabs y la resina "Clear" de la misma empresa. Se caracterizan y reportan tres propiedades: La permitividad relativa, la rigidez dieléctrica y el módulo de Young. Adicionalmente a los materiales estudiados, se sugieren otras opciones a ser usadas como elastómeros dieléctricos basados en las propiedades relevantes según la ecuación acoplada de estrés electromecánico de Maxwell en una dimensión. Finalmente, se sugiere un método comparativo que analiza propiedades críticas del material para comparar su usabilidad como elastómero dieléctrico.

Palabras Claves: DEA, elastómero dieléctrico, impresión 3D, estrés electromecánico.

OBJECTIVES

1. Determine crucial properties in dielectric elastomers for modeling and usage as Dielectric Electroactive Actuators (DEAs) and the correlation between them.

2. Find the properties defined in objective 1 for Formlabs' Clear Resin and Formlabs' Flexible Resin and determine the suitability of the mentioned materials as dielectrics in DEAs applications.

3. Generate a fast method for comparing materials for DEA applications.

4. Propose alternative materials for further research to be used as dielectrics in DEAs.

CHAPTER I

THEORY

The present chapter includes four subsections that include the theory of: Elastomers, Capacitors, Dielectric elastomer actuators, and a novel coefficient the assess the materials properties based on the DEA requirements to determine its viability and usability as electroactive polymer for a DEA.

1. Elastomers

Elastomers are a subclass of linear polymers, characterized by its ability to withstand substantial force-induced deformation and remain in the elastic region. The name is a contraction for elastic polymers, name that summarizes one of its most outstanding feature. Ideally, an elastomer has an infinite fatigue limit and is capable of only elastic deformation. This means it should be able to recover its original shape after multiple cycles of loading and unloading.

The above-mentioned capabilities of the elastomers are caused due to its internal configuration. Engineering materials' mechanical properties, the ones related to their elastic behavior, are the result of their internal interatomic forces. As a body is pulled or compress, the distance between its atoms will change. Once the body is unloaded, the atoms will return to their original distribution, closing the elastic deformation cycle. Hooke's Law [6], describes the linear relation between the deformation and the force applied.

However, according to Black and Kohser (2012) elastomers behave differently. First of all, the elastic behavior of the elastomers is not linear. Internally, elastomers'

molecules are a linear chain-type that are found twisted or curled. Once the force is applied, the molecules untwist or uncurl. The polymer can return to it's original 'unload shape' as the bonding angles return to their original positions. As expected, this behavior cannot be modeled with Hooke's Law, and require more complex approximations. In addition, elastomers suffer of plastic deformation, due to the slide of molecular chains under the influence of a load. This creates a viscous deformation that becomes part of the plastic deformation of the elastomer.

Another characteristic behavior of elastomers is stress relaxation. It is described as viscous flow over timer for an elastomer held under a constant strain. For any given elastomer, when held in a position that requires it to stretch (with a corresponding load), it is likely to have one of the following two behaviors depending on the load set up. If the load is constant, the elastomer will start to deform further than the original deformation. If the strain is constant, the load required to maintain the mentioned strain will decrease over time. This is described as stress relaxation, and according to Black and Kohser [5,6], it's rate will depend in three factors: Material, Force and Temperature

Finally, elastomers can be found in two forms: as natural rubbers or artificial elastomers. Natural rubber comes from the latex secretions of a tree. They were first use as an engineer material when Charles Goodyear combined them with sulfur to vulcanize them. This added strength by restricting the movement of the molecular chains (they were cross-linked). In general, natural rubbers present useful mechanical properties, including good resilience, high tensile and tear strength and good electrical insulation. Even though they show good

resistant to most organic acids, they are not resistant against petroleum products (oil, gasoline, et.)

Artificial elastomers were designed taking into considerations the limitations that natural rubbers present. They also aimed to reduce the uncertainty and supply issues that a natural product can generate. Artificial elastomers can be both thermosets and thermoplastics. Their fabrication process however will differ, as making thermosets requires an irreversible vulcanization process, whereas making thermoplastics does not require any cross-links.

2. Capacitance

A capacitor is defined as "the assembly of two conductors separated by an insulator (or vacuum)" [21] with the capability of being charged. The charging is achieved by the transfer of electrons from one conductor to the other, "charging" the capacitor. Both conductors will then possess a charge equal to the one of the opposite conductor, but with the opposite sign. Even though both possess charge, as the charges are equal in magnitude, but opposite in sign, the net charge in the capacitor will remain zero.

When the capacitor is charged, and the opposite charges are established, the capacitor will have then a potential difference equal to the one of the source used to charge it. The capacitor shows a linear relationship between the charge on each conductor, and the charge density on each point, the electric field and the potential difference. Even though an increase in the magnitude of the charge on each conductor, will generate an increase in the

same rate on the mentioned properties, the ration between the charge and the potential difference, called capacitance, will remain constant.

$$C = \frac{Q}{V_{ab}}$$

The capacitance is proportional with the amount of energy a capacitor can store. "Thus, capacitance is a measure of the ability of a capacitor to store energy" [21]. The energy to be stored, depends on the geometry of the capacitor and in the material in use. Parallel-plate capacitors are a simple type of capacitor. They are made of two conductive plates with an area "A" and a distance between them "d". The field between them is uniform as well as the charge distribution on both electrodes. If a capacitor posses' flexible plates, the capacitance will change as the plate changes its geometry. Changes in the thickness of the capacitor (distance between the plates) will also affect the capacitance.

In between the two conductive layers, capacitors usually include a nonconducting material called dielectric. According to Sears, Zemansky, Young and Freedman [19], capacitors include this dielectric for three reasons:

- Provides support to the structure, solving the building problem of keeping to conductive plates at a very small distance from each other without touching.
- The dielectric increases the maximum possible voltage to be applied to the capacitor as long as the dielectric possess a higher dielectric breakdown than the air. This also means that the capacitor will be able to handle higher charges.

- The capacitor's capacitance will increase as well as the capacitors are able to handle higher charges.

The dielectric materials add an extra variable, called the dielectric constant. This is defined as the ratio between the new capacitance and the original capacitance of the capacitor

$$K = \frac{C}{C_0}$$

The value of a capacitor's capacitance will be affected by the presence of a dielectric. The new capacitance can be expressed as:

$$C = K\epsilon_0 \frac{A}{d}$$

Rewriting the first equation, we obtain that the Voltage of a capacitor with a dielectric material in between the two conductive plates is:

$$V = \frac{Q}{C} = \frac{Qd}{K\epsilon_0 A}$$

3. **Dielectric Breakdown**

According to Young, Freedman and Ford [19], capacitors' operating voltage is limited by the dielectric strength of the dielectric material. This is a material property that limits the maximum electric field that it can withstand without becoming a conductor. The dielectric strength is reported as a function of the thickness of the material. The voltage at which a

material of a given thickness fail is called voltage breakdown, and is the product of the dielectric strength by the material thickness.

As stated before, a dielectric material can become a conductor if the applied voltage is higher than the voltage breakdown. The reason for this is because of the interaction between the electrons and the electric field. When the electric field reaches a certain level, the electrons on the atoms are forced to move to other atoms, moving more electrons in their way. This generates an arc discharge or a spark in the material, melting a hole through the dielectric material. The hole becomes a conducting path, a short circuit, between the conductors, leaving the capacitor useless and converting it into a simple cable.

The dielectric strength of the material is susceptible to changes in temperature, impurities, irregularities in the surfaces, and multiple other factors. All these variables make the dielectric strength a particularly difficult property to control. Due to this, dielectric strength is reported within a range in addition to the value.

4. Dielectric Elastomer Actuators

The "Dielectric Elastomer Actuators" (DEA) are considered integrated structures (Sommer-Larsen, Hooker, West, Benslimane, & Gravesen, 2001) that consist of two parts: A relatively soft dielectric elastomer or more generally a polymer, and a couple of compliant electrodes. [12, 15, 16, 17, 18] It is also called compliant capacitor, as the two compliant electrodes sandwich a passive dielectric elastomer film between them [15,18].

The DEA are electric field driven [16]. This means that they require an electric potential difference to be applied between the two compliant electrodes in order to be actuated. The free charges on the electrodes generate electrostatic forces proportionally to the square of the electric field [15]. When both electrodes are charged, attraction due to Coulomb forces occur among them, compressing the dielectric elastomer inner layer [12]. For any isotropic polymer working as dielectric, the induced stress and strain by the electrostatic field have two components: The Maxwell stress and Electrostriction. The first one can be defined as "coulombic interaction between charges on electrodes needed for establishment of the electric field" (Krakovsky, Romijn, & Posthuma de Boer, 1999) and can be derived from a simple electrostatic model. It is given as an effective pressure, which is a function of the applied voltage and the initial thickness of the film [14]. The second one is the result of the change in the dielectric properties of the material as the ions rearrange and stretch in the direction of the electric field. [11]. However, the elastomer material used in DEAs is not required to show an electroactive response by itself. This allows it to be differentiated from a "true" electrostrictive polymer. [16]

As a result of the mechanical compression, the dielectric elastomer will stretch in length and width (in the film plane direction) as they contract in the thickness direction. [14, 15, 18]. It is important to mention that due to the insulating behavior of the dielectric elastomer, the material deformation will be caused by the electrostatic mechanical pressure that the electrodes exert in the thickness direction, and not due to the electric field [18].

The compliance of the electrodes refers to the required ability of the electrode to deform as the dielectric elastomer deforms. Due to the reduction in thickness, the elastomer

will increase its width and length and consecutively, its area. In order to allow the film to expand, the electrode needs to be flexible and compliant. Furthermore, the compliance also means that the charges within each electrode will also generate further mechanical deformation. The reason for this is that like-charges will generate repulsion within each other [15] generating "charged particles to move relative to one another" (Suo, 2010) [17]. In addition, if the electrodes are compliant, the stress of the dielectric elastomer will be double the stress on each electrode, and the deformation will occur under volume conservation, for materials with a Poisson ratio close to 0.5. [16]. Under these conditions, a reduction in the thickness of the film will generate a proportional expansion in the in-plane x-y direction [12].

Electroactive polymers require elastic conductive polymers to act as compliant electrodes. Kaneto (Research Trends of Soft Actuators based on Electroactive Polymers and Conducting Polymers, 2016) list multiple research trends related with conductive elastic polymers and the processes for their fabrication. These methods have been studied for more than 10 years and are a real possible source of materials for the compliant elastic electrodes [9]. Ideally, the electrode should present the least possible resistance for the expansion of the film by possessing the smallest possible Young's modulus. [15]

Perline et all. provides an interesting analysis based on the above-mentioned behavior of the dielectric elastomer when subjected to an electrostatic field. DEAs can be understood as actuators that convert the electrical energy (taken from the electric field) into mechanical work (electric field pressure on the material and load). The DEAs then, from an electrical point of view "bring opposite charges closer together and like charges further

apart as the film contracts in thickness and expands in area. These changes reduce the stored electrical energy and the difference is converted to mechanical work." (Pelrine, et all., 2001) [13].   Performance improvement in the actuation motion range can be achieved by pre-stretching the film. Some materials can be pre-stretched up to five times it's nominal dimension. [18]

Dielectric Elastomer Actuators present multiple benefits due to their construction and operation method. Some characteristics benefits are their high strains, high actuation velocity and pressure and high specific energy densities, [15, 18] no noise when actuated, light weight, and relatively low cost [10]. Another important benefit is the high efficiency they can theoretically achieve (around 80% to 90% considering charge recovery) [15, 18]. DEAs are also very versatile, as their effective pressure can be varied without varying the operating voltage. This can be achieved by using high-dielectric polymers, as the pressure on the elastomer is directly proportional to the dielectric constant of the film. [15]. In comparison with their air-filled counterparts, the electrostatic devices, the electric fields that DEAs can withstand is potentially larger for a device with the same thickness, outperforming them in the maximum electrostatic pressure achievable [15].

Dielectric Elastomers' application has been discussed in several papers [12, 13, 15, 18]; however, they can be group in three categories: Actuators, Sensors and Generators. Each application will be discussed in the following lines.

As actuators, some of the mentioned applications include: Microrobot actuators, sound generators, display injects, smart skins [15], mobile mini and microrobots,

micropumps and microvalves, micro air vehicles, disk drives, prosthetic devices, and flat panel loudspeakers [18]. All these applications relay on two main characteristics of the DEAs: the high actuation speed that these solid-state actuators can achieve, and the usage of constrain that enables the construction of stretched-film actuators by varying the actuated and non-actuated zones [15]. This in-plane expansion is constrained to convert it into motion or forces in different directions [18]

As generators, the working principle is very similar to the actuators, but instead of converting electrical energy into mechanical work, it converts mechanical work into electrical energy. In order to use a dielectric elastomer as a generator, an initial voltage difference between the electrodes is required. However, instead of increasing the voltage to deform the material, a mechanical pressure needs to be applied in order to compress the set-up. The electrostatic field pressure will work against the mechanical work, elevating the voltage of the charge, and therefore, generating electrical energy [13]. By this, the dielectric elastomers prove their capability not only to convert mechanical actuation as a response to an applied electric field, but also prove they are capable of harvesting electrical energy based on mechanical pressure and movement [12].

Finally, the sensing capabilities are discussed in Newell, Garcia, Krtuz and Harmeyer's paper about the industrial sensing application of DEAs [12]. They use the DEA integrated structure as a parallel plate capacitor. The capacitance and overall sensitivity is determined by the overall geometry, which include changes in thickness (or relative distance between the conductive plates), changes in the area and changes in the parallelism of the two electrodes. Also, changes in material properties, both physical properties and

electric properties influence the capacitance of the set up. The discussed applications include hydraulic hose sensor, tire sensors and seal sensors. Finally, they mention that even though this set up would perform as a sensor, the dielectric elastomers in a DEA set up, provide means of actuation to the sensed structure [12].

CHAPTER II

DEA COEFFICIENT

As stated on the previous chapter, the Maxwell stress is responsible of a DEA deformation under the influence of an electrical potential differential. The derivation of this equation is derived in multiple articles, but [4D] summarizes the process in a very friendly and easy to understand way. It starts with the assumption that an elastomer is very likely to have a Poisson ratio close to 0.5, meaning that the volume of the elastomer remains constant

$$Az = constant$$

A = Area

Z = Film thickness

They define the effective pressure "p" of the actuator as the change in electrostatic energy per unit area per unit displacement of the film in the thickness direction

$$p = \left(\frac{1}{A}\right)\frac{dU}{dz}$$

U = Electrostatic Energy

Then, the stored electrostatic energy U of a film with opposite charges Q and -Q placed on its surfaces can be written as

$$U = \frac{0.5Q^2}{C} = \frac{0.5Q^2 z}{\epsilon_0 \epsilon A}$$

$$C = \frac{\epsilon \epsilon_0 A}{z} \text{ is the capacitance,}$$

$$\epsilon = \text{relative dielectric constant (As}$$

relative to the polymer density, does

not vary)

$$\epsilon_0 = \text{Free space permittivity}$$

The change in stored electrostatic energy dU for a change dz in thickness and dA in area can be derived:

$$dU = \left(\frac{0.5Q^2}{\epsilon \epsilon_0 A} \right) dz - \left(\frac{0.5Q^2 z}{\epsilon \epsilon_0 A} \right) \frac{dA}{A}$$

By applying the constrain (Az = constant → Adz + zdA = 0):

$$\frac{dA}{A} = -\frac{dz}{z}$$

We can rewrite the previous equation in the following form:

$$dU = \left(\frac{Q^2}{\epsilon \epsilon_0 A} \right) dz$$

Combining this equation with the effective pressure one, we obtain:

$$p = \left(\frac{1}{A} \right) \frac{dU}{dz} = \left(\frac{Q^2}{\epsilon \epsilon_0 A^2} \right) dz$$

Finally, they consider that the electric field E is given by $E = \frac{Q}{\epsilon\epsilon_0 A}$, and express the effective pressure as:

$$p = \epsilon\epsilon_0 E^2$$

$$E = \frac{V}{z}$$

This equation can be further study and paired with a mechanical response. The simple linear elastic model that Hooke's law described is used. This 1D model will use the stress generated by the electric field and the attraction between the electrodes to generate a strain based on the Young's Modulus of the material

The final effective pressure equation is rewritten as follows:

$$\epsilon\epsilon_0 \left(\frac{V}{z}\right)^2 = p = \sigma = \varepsilon Y$$

Y = Young's Modulus

ε = Strain

The strain that will be occur due to the pressure created by the attraction of the electrodes, can be then modeled in 1D with the following equation:

$$\varepsilon = \frac{\epsilon\epsilon_0}{Y}\left(\frac{V}{z}\right)^2$$

The equation shows shown above relates the thickness of the dielectric film with the voltage applied, the elastic modulus and the relative permittivity in order to obtain a strain.

Calculating the deformation is a proper measurable way to compare two materials. However, it will be affected by the geometry of the material and how big the assembly used to test the properties or build the DEA is. For this reason, a combination of purely properties based on the electromechanical coupled equation is proposed as a method for analyzing the viability and usability of a material as an electroactive polymer for a DEA. The proposed coefficient is a dimensionless number that will be used to compare a material with another one by the comparison of their numbers. It does not hold a value on its own.

The coefficient formula is very similar to the electromechanical coupling formula, but with a small difference. It shares with the electromechanical coupling formula the Young's modulus and the relative permittivity, but uses the dielectric strength as the maximum possible ratio between the voltage and the thickness of the material. In other words, it presents the best possible situation in which the material is subjected to the maximum possible voltage before breaking. The coefficient formula then looks like this:

$$Coef_{DEA} = \frac{Relative\ Permitivity}{Young's\ Modulus\ [MPa]} \left(Dielectric\ Strength \left[\frac{MV}{m} \right] \right)^2$$

In order to avoid numbers with multiple powers of 10, the Young's modulus is reported in megapascals and the dielectric strength in megavolts per meter.

CHAPTER III

PROCEDURES

1. Dielectric Breakdown

The aim of the test is to determine the breakdown voltage and dielectric strength of the insulating material tested. For this, five samples of each material are prepared, measured and tested in accordance with the ASTM D3775 standard [2]. An accurate summary of the test is given in the standard and included in this thesis:

"The specimen, held in a properly designed electrode system, is electrically stressed by the application of an increasing direct voltage until internal breakdown occurs. The test voltage is applied at a uniform rate of increase. The direct voltage is obtained from a high-voltage supply of adequate current capacity and regulation, reasonably ripple-free, with facilities for measuring and controlling the output voltage."

1.1. Specimens

The specimens are made from the 3D printed materials: Formlabs' Flexible Resin and Formlabs' Clear Resin. The specimen is required to have a nominal diameter of 50mm and a nominal thickness of 0.5mm. The specimen cross section dimensions are presented in Figure 1 - *Specimen cross section for dielectric breakdown*.

50mm

5mm
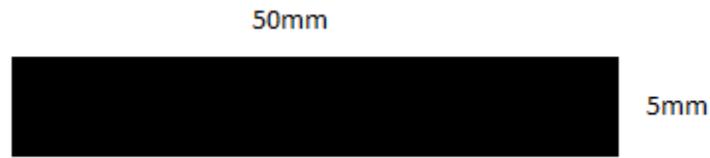
Figure 1 - Specimen cross section for dielectric breakdown

1.2. Electrodes

The electrodes are made of conductive grease (MG Chemicals's Carbon Conductive Grease) with a thin strip of copper. The copper strip is placed between the specimen and the grease. It is used as a connection port to attach the terminals of the power source to the assembly. A conductive grease is selected instead of a solid conductor due to its ability to stretch at the same rate than the specimen. The specimen materials are selected due to its capability to be used as dielectric materials in DEAs. Due to this, they will present a deformation when voltage is applied in the current set up, requiring an electrode with the same capability to stretch without creating an additional stress on the specimen. In addition, as the material stretches, the voltage is applied to the same material, keeping the portion of the mass of the specimen tested constant. Each sample is covered with a conductive grease following a template. The template can be seeing in Figure 2 - *Grease application layout* The grease needs to be applied evenly in both faces, which are 180° rotated mirrors of each other. A minimum of 5 specimens per material is required.
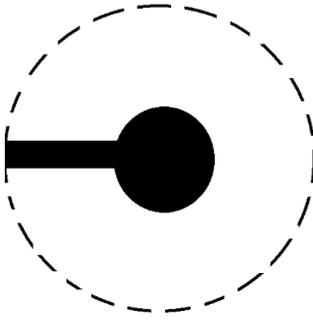
Figure 2 - Grease application layout

As a safety measurement the upper side of the specimen will be the positive side whereas the bottom of the specimen will be the negative one. (In case the specimen is not laying horizontally, but vertically, the side facing the user should be the positive one).

1.3. Test equipment

For this test, a variable high voltage supply with a voltage display is required. In case the voltage source does not display the voltage, an additional measuring tool will be required. The source used is the Trek's model 610E. It was paired with two crocodile connectors attached to the copper strips on each sample.

As a safety precaution, a connection between the source ground port and the building ground is recommended due to the high voltages will be applied. It is important to keep in mind that a flexible capacitor is being charged while doing this test, so giving time to discharge or connecting it to the building ground in case the test needs to be interrupted is important. Another recommended safety precaution is to use a low current fuse or a voltage source with a thermal fuse to avoid the high current peak when the material breaks.

1.4. Test Set up

- Place the holder next to the power supply.

  - It is important to make sure that the specimen and the holder are not between the user and the power supply. As the power supply requires constant manipulation, placing the holder in between implies a big unnecessary risk.

- Place the specimen on top of the holder, leaving both copper strips free on the sides.

  - As mentioned before, the upper side of the specimen is the one to be connected to the positive end of the power source, whereas the lower side is the one to be connected with the negative end of the power source.

- Connect the building ground terminal with the power supply ground electrode.

- Connect the positive and ground connector of the power supply to the electrodes.

1.5. Procedures

This procedure is specific for the Trek's model 610E high voltage power supply, and it is based on the ASTM D3775 standard and the power supply user's manual.

For the first test of a new material:

1.5.1. Procedure "A"

1. Check that all switches are turned "Off" or placed as presented in figure____.
2. Check that the switch for the high voltage dial selects the 0-1000V.

3. Turn "on" the On/Off switch and wait for 30 seconds for the power supply to charge.

4. Change the HV switch to "On"

5. Turn the dial to increase the voltage at a rate of 50V/s 1

    a. If the material does not break, then turn the dial down at the same speed and then turn off the source. Then, wait for 20 seconds to give time to the capacitor to discharge and proceed with the procedure "b".

    b. If the material break, record the voltage at which it broke.

1.5.2. Procedure "b"

1. Check that all switches are turned "Off" or placed as presented in figure_____.

2. Check that the switch for the high voltage dial selects the 0-10KV.

3. Turn "on" the On/Off switch and wait for 30 seconds for the power supply to charge.

4. Change the HV switch to "On"

5. Turn the dial to increase the voltage at a rate of 500V/s 2

6. Record the voltage at which the material breaks. In case it does not break, try with thinner specimen.

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[1] This rate is given to make sure the maximum voltage is reached at 20 seconds.
[2] This rate is suggested in the ASTM D149 standard

Once the breakdown voltage for the material has been determined by the first iteration, replace the voltage increase rate with the one obtained from on Equation 1 - *New voltage increase rate*:

$$New\ rate\left[\frac{Volts}{Sec}\right] = \frac{Voltage\ at\ breakdown}{20\ seconds}$$

Equation 1 - New voltage increase rate

1.6. Report

Before the test, the following information must be recorded:

1. Date of the test

2. Specimen's material

3. Test number (relative to the other specimens made from the same material)

4. Measured thickness [mm]

5. Temperature [°C]

6. Voltage increase rate [V/sec]

After the test, the following information must be recorded:

7. Voltage at breakdown [V]

8. Test time [s]

9. Location of failure (center of electrode, edge or outside)

With the recorded information, the Dielectric strength can be calculated with Equation 2 - *Dielectric Strength calculation*

$$Dielectric\ Strength\ \left[\frac{V}{mm}\right] = \frac{Voltage_{failure}\ [V]}{thickness\ [mm]}$$

Equation 2 - Dielectric Strength calculation

After the completion of this test, the report should include the following information:

1. Material name

2. Temperature of test [C]

3. Voltage increase rate [V/s]

4. Average thickness [mm]

5. Mean dielectric strength [V/mm]

6. Max / Min dielectric strength [V/mm]

7. Standard deviation

8. Coefficient of variation [%]

2. Traction

The aim of the test is to determine the tensile properties of the material tested. For this, five samples of each material are prepared, measured and tested in accordance with the ASTM 638 standard [3]. The specimens are tested under defined temperature, pretreatment and testing machine speed conditions.

2.1. Specimens

The specimens are made from the two 3D printed materials: Formlabs' Flexible Resin and Formlabs' Clear Resin. The specimen is shaped is the type IV from the ASTM standard. The specimen cross section dimensions are presented in Figure 1 - Specimen cross section for dielectric breakdown.
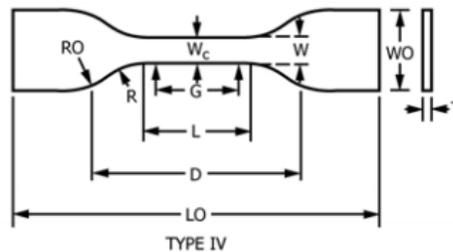
Specimen Dimensions for Thickness, T, mm (in.)[A]		
Dimensions (see drawings)	4 (0.16) or under	Tolerances
	Type IV[B]	
W—Width of narrow section[E,F]	6 (0.25)	±0.5 (±0.02)[B,C]
L—Length of narrow section	33 (1.30)	±0.5 (±0.02)[C]
WO—Width overall, min[G]	19 (0.75)	+ 6.4 (+ 0.25)
WO—Width overall, min[G]	...	+ 3.18 (+ 0.125)
LO—Length overall, min[H]	115 (4.5)	no max (no max)
G—Gage length[I]	...	±0.25 (±0.010)[C]
G—Gage length[I]	25 (1.00)	±0.13 (±0.005)
D—Distance between grips	65 (2.5)[J]	±5 (±0.2)
R—Radius of fillet	14 (0.56)	±1 (±0.04)[C]
RO—Outer radius (Type IV)	25 (1.00)	±1 (±0.04)

Figure 3 - Specimen dimensions for tensile test – Adapted from the ASTM D638 standard

After the specimens are 3D printed, the next steps need to be follow:

1. Manually remove the excess resin

2. Place the specimen is in an isopropyl alcohol bath for 5 minutes.

3. Rinse the specimen with water, to clean any residue or resin on the surface.

4. Dry the specimen with a dry paper towel

5. Place the specimen in a clean surface and left in laboratory conditions of temperature and relative humidity for two days before the test.

    a. As the specimens are susceptible to UV light, they are kept inside the fabrication laboratory and covered when transported to the laboratory for test.

6. Any additional post printing process takes place after drying the material.

2.2. Test equipment

For this test, the used equipment is the following: a tensile machine, a load cell, two grippers, a PC interface for controlling the tensile machine and a micrometer. An external extensometer can be used to determine the elongation. Due to availability, the equipment used for this particular case was:

- MTS Insight – Electromechanical – 100KN Standard Length tensile machine paired with A 2000 pounds load cell
- A 0 to 1 inches Mitutoyo micrometer

2.3. Test Set up

1. Turn on the PC and the testing machine

2. Open the program and load the ASTMD638 Tensile test pre-defined test

3. Introduce the cross-section area of the specimen

4. Check the load cell mounted on the machine is the correct one

5. Introduce the separation rate for the specimen. This separation rate depends on the specimen type and nominal strain rate at failure. For this case, a 5mm/min is used.

2.4. Procedure

1. Insert one specimen between the grippers.

2. Using an Allen key, twist the screw at the top grippers to tide the specimen in between the two opposite faces.

3. Align the bottom gripper to the top one so that the specimen remains aligned.

4. Use the Allen key to secure both grippers so that the specimen does not fall.

5. Using the caliper, measure the separation distance between the grippers

6. Insert the measured initial separation distance

7. Start the test

8. Once the test is finished, bring the two grippers to the original position

9. Unscrew the Allen screws to take the parts of the specimen out of the gripper.

2.5. Report

Before the test, the following information must be recorded:

10. Date of the test

11. Specimen's material

12. Test number (relative to the other specimens made from the same material)

13. Cross-sectional area [m2]

14. Temperature [°C]

15. Separation rate [mm/min]

After the test, the following information must be recorded:

1. Initial Separation

2. Final Separation

3. Test time [s]

4. Location of failure

With the recorded information, this particular equipment already calculates the elasticity modulus and plots the engineering curves of stress and strain for the material. After the completion of this test, the report should include the following information:

1. Material information

    a. Name

    b. Grade

    c. Color

    d. Manufacturer

2. Specimen information

    a. Thickness

    b. Width

    c. Cross section Area

3. Speed of testing

4. Stress and Strain at break

5. Percentage of elongation at break

6. Modulus of elasticity

7. Date of testing

3. Capacitance

The aim of the test is to determine the capacitance of the specimen and relative permittivity of the dielectric material tested. For this, six samples of each material are prepared, measured and tested in accordance with the ASTM D150-11 standard [1]. The specimen is placed between two electrodes and the capacitance is measured for different frequencies. A dissipation factor is also reported.

3.1. Specimens

The specimens are made from the two 3D printed materials: Formlabs' Flexible Resin and Formlabs' Clear Resin. The specimen is required to have a nominal diameter of 50mm and a nominal thickness of 0.5mm. Both are printed with the default settings for the material. The specimen cross section dimensions are presented in Figure 1 - Specimen cross section for *dielectric breakdown*.

50mm

0.5mm
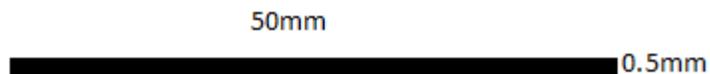
Figure 4 - Specimen cross section for relative permittivity test

Once the specimen is printed, it is rinsed in Isopropyl alcohol to remove the excess of resin and clear any particle of unwanted material. The specimen is left on the IPA bath for a time of five minutes and then washed with water. After this process, the specimen is

dried with paper towels and left in laboratory conditions of humidity, light and temperature for two days before the test. Due to production limitations, the samples printed from Formlabs' Clear resin were printed one day before the samples of Formlabs' Flexible resin.

The specimens require thin film electrodes to be attached to it. The two thin electrodes are made out of a thin sheet of stainless steel of 0.001in thick and cut to possess the same diameter as the specimens.

3.2. Test equipment

For this test, a LCR meter and a compatible capacitance measuring module are required. The specific test equipment used are the Agilent 4263B LCR meter and the Agilent 16451B capacitance measuring module. In addition, a micrometer is used to measure the specimen thickness and a caliper is used to measure the diameter of the specimen.

The Agilent 16451B can be paired with multiple options for electrodes. The user manual [10] includes a chart that describes the electrodes and their applications. Figure 5 - Available Electrodes with the Agilent 16451B module shows the list included in the user manual. From the possible options for the electrodes, the electrode "type C" where the chosen one for the test, as they can be used with thin film electrodes and allows the material tested to be thin as well. The configuration of electrode "C" can be observed in Figure 6 - Electrode C and Electrode D configuration

Measurement Method	Contacting Electrode Method (used with Rigid Metal Electorde)	Contacting Electrode Method (used with Thin Film Electorde)	Non-contacting Electrode Method (Air Gap Method)
Electrode* Structure	Rigid Metal Electrodes	Thin Film Electrode	
Operation	Simple → Complex		
Applecable Test Material	○ Thick material ○ Smooth material	Materials on which thin film electrode can be applied without changing its characteristics	○ Including contacting Method's applicable test Materials ○ Highly compressible material ○ Soft material
Electrodes of 16451B	Electrode-A Electrode-B	Electrode-C Electrode-D	Electrode-A Electrode-B
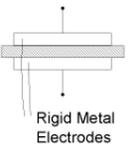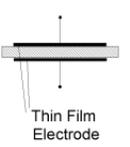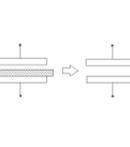
Figure 5 - Available Electrodes with the Agilent 16451B module. Extracted from Agilent
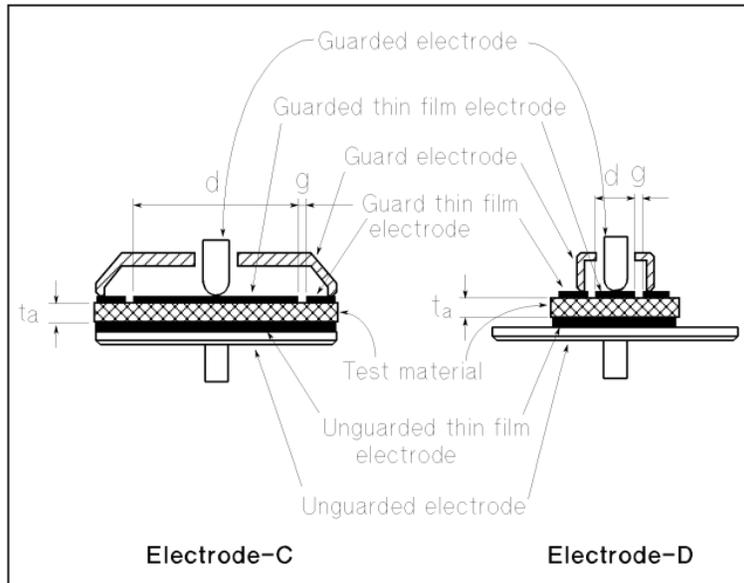
16451B user manual

Figure 6 - Electrode C and Electrode D configuration. Extracted from Agilent 16451B user

manual

3.3. Test Set up

This test uses the type "C" electrodes for the capacitance measuring module. For instance, the guarded electrode must be placed on the Agilent 16451B module, replacing the top electrode. In order to change the electrode, the included Allen tool is necessary. A screw on top of the 16451B module holds the guarded electrode in place. To unscrew it, it is first necessary to hold the guarded electrode with one hand and then unscrew the screw with the Allen screwdriver. Then replace the electrode with the type "C" electrode and hold it against the top of the equipment. Then, softly twist the screw until the new electrode can hang in the air. Finally, hold the electrode type "C" and secure the screw.

Other additional procedures that are required to set up the equipment include:

6. Connect the capacitance measuring module to the LCR meter and secure the connection

7. Distance the two electrodes

8. Turn on the LCR meter

9. Place the "sandwich of thin electrodes and the dielectric material" in between the guarded electrodes of the Agilent 16451B.

3.4. Procedures

This procedure is specific for the Agilent's model 4263B LCR meter paired with an Agilent's 16451B Capacitance measuring module, and it is based on the ASTM D150-11 standard and the equipment user's manual.

1. With the machine already set up, proceed to select the option to display capacitance and dissipation.

2. Twist the micrometer until the guarded electrodes are in contact with the thin film electrodes

3. Use the wrench dial on top of the micrometer to reach the required pressure.

4. Register the value of the Capacitance and the Dissipation Factor.

3.5. Report

Before the test, the following information must be recorded:

1. Date of the test

2. Specimen's material

3. Test number (relative to the other specimens made from the same material)

4. Measured thickness [mm]

5. Temperature [°C]

6. Frequency [Hz]

After the test, the following information must be recorded:

1. Capacitance [V]

2. Dissipation Factor [s]

With the recorded information, the relative permittivity strength can be calculated with Equation 2 - Dielectric Strength calculation

$$\varepsilon_r = \frac{t_a\,[m]\ x\ C_p[F]}{A[m^2]\ x\ \varepsilon_0\ \left[\frac{F}{m}\right]}$$

Equation 3 – Relative permittivity

Where:

$$t_a\ = Thickness$$

$$C_p = Capacitance$$

$$A\ \ = Area$$

$$\varepsilon_0\ = 8.854\ x\ 10^{-12}$$

After the completion of this test, the report should include the following information:

1. Material information

    a. Name

    b. Grade

    c. Color

    d. Manufacturer

2. Shape of the specimen

3. Dimensions of the specimen

4. Electrode information

    a. Type of electrode

    b. Dimensions of the electrode

5. Test Conditions

    a. Temperature of test [°C]

       b.   Frequency [Hz]

6.   Capacitance [F]

7.   Relative permittivity of the dielectric material

CHAPTER IV

RESULTS AND DISCUSSION

The results for each test are presented in the following sections. All the results values are compared with a reference material: 3M VHB. This material was chosen as a reference due to its repeated appearances in academic literature and because it is a standard when comparing materials for DEAs applications. The properties used to compare the VHB with the Formlabs' Clear and Flexible resin are dielectric breakdown, elongation at break, Young's modulus and dielectric strength. The mentioned properties are listed in Bozlar, et al. paper [7] and presented in the *Table 1 - VHB's properties*

MATERIAL	YOUNG'S MODULUS [MPA]	ELONGATION AT BREAK [%]	RELATIVE PERMITTIVITY	DIELECTRIC STRENGTH [MV/M]
3M'S VHB	0.220	860	3.21 @ 1kHz	65

Table 1 - VHB's properties

1. Dielectric Breakdown

The obtained results for the Dielectric Breakdown are presented in Table 2 - *Dielectric Breakdown Results* The dielectric strength is calculated by dividing the voltage breakdown by the thickness of the film. The result is given in mega Volts per meter of thickness. The average dielectric strength for the Formlabs' Flexible resin is 12.526 MV/m and the average dielectric strength for the Formalb's Clear resin is 10.6575 MV/m.

There is a correlation between the presence of air bubbles in the specimen and the dielectric strength. For both materials, the sample with the lowest dielectric strength happened to be the sample with the highest presence of air bubbles.

Code	Material	Thickness [m]	Area [in^2]	Voltage Breakdown [V]	Dielectric Strength [MV/m]
1FC	Flexible	0.000711	0.0020	7800	10.96738
2FC	Flexible	0.000711	0.0020	8350	11.74072
3FC	Flexible	0.000528	0.0020	7480	14.15809
4FC	Flexible	0.000767	0.0020	8110	10.57256
5FC	Flexible	0.000559	0.0020	7620	13.63636
6FC	Flexible	0.000564	0.0020	7940	14.08101
1CC	Clear	0.000798	0.0020	8200	10.28136
2CC	Clear	0.000782	0.0020	8790	11.23581
3CC	Clear	0.0322	0.0020	9210	11.26082
4CC	Clear	0.0300	0.0020	8480	11.12861
5CC	Clear	0.0372	0.0020	9010	9.535602
6CC	Clear	0.0328	0.0020	8750	10.50269

Table 2 - Dielectric Breakdown Results

2. Tensile Test

The tensile test results are presented in the Table 3 - *Tensile Test Results* The elongation

break and the elastic modulus are reported with other specifications of the test and

characteristics of the specimen. Two different speed tests were used, resulting in a small

difference in the elastic modulus due to the viscoelastic dampening effect in the flexible.

For the Formlabs' flexible resin, the average value at 5mm/min is 9.62MPa and an

elongation at break of approximately 29.24%, whereas at the higher speed of 50 mm/min,

the perceived average elastic modulus was 11.32MPa and the average elongation at break

was 40.94%. The other material, the Formlabs' clear resin, presents a stiffer behavior. The average elastic modulus for a separation speed of 5mm/min was 787.35MPa in average, and the average elongation at break was 7%. When the test speed is increased, the elongation at break drops to 3.86% whereas the elastic modulus increases to 1.063GPa.

From the mentioned results, it was particularly interesting that for the flexible resin. the average elongation at break at higher speeds was bigger than the elongation at break at lower speeds. The influence of speed in the mechanical properties is not under the scope of this thesis. However, it was considered relevant analyze if there was a viscoelastic effect on the materials for future work.

Code	Material	Width [in]	Thickness [in]	Cross Section Area [in2]	Speed of testing	Elongation at break [%]	Elastic Modulus [MPa]
1F	Flexible	0.2482	0.0393	0.0098	5mm/min	22.20%	9.62
2F	Flexible	0.2555	0.0415	0.0106	5mm/min	25.90%	9.75
3F	Flexible	0.2502	0.0399	0.0100	50mm/min	41.50%	11.59
4F	Flexible	0.2518	0.0398	0.0100	50mm/min	41.20%	11.94
5F	Flexible	0.2604	0.0420	0.0109	50mm/min	28.60%	10.43
6F	Flexible	0.2585	0.0412	0.0107	5mm/min	35.80%	9.52
7F	Flexible	0.2486	0.0438	0.0109	5mm/min	33.90%	9.63
8F	Flexible	0.2550	0.0460	0.0117	50mm/min	42.50%	11.16
9F	Flexible	0.2543	0.0465	0.0118	50mm/min	46.40%	12.89
10F	Flexible	0.2566	0.0395	0.0101	5mm/min	28.40%	9.56
11F	Flexible	0.2642	0.0412	0.0109	50mm/min	43.90%	10.68

12F	Flexible	0.2606	0.0399	0.0104	50mm/min	42.50%	10.51
1C	Clear	0.2581	0.0273	0.0070	5mm/min	7.60%	630.90
2C	Clear	0.2476	0.0223	0.0055	5mm/min	13.50%	Failure
3C	Clear	0.2635	0.0280	0.0074	5mm/min	13.80%	772.76
4C	Clear	0.2583	0.0255	0.0066	50mm/min	4.60%	924.84
5C	Clear	0.2656	0.0283	0.0075	5mm/min	2.30%	881.35
6C	Clear	0.2645	0.0274	0.0072	5mm/min	3.50%	877.81
7C	Clear	0.2698	0.0297	0.0080	50mm/min	2.50%	1192.93
8C	Clear	0.2524	0.0273	0.0069	50mm/min	4.80%	1060.04
9C	Clear	0.2438	0.0227	0.0055	5mm/min	8.40%	617.50
10C	Clear	0.2591	0.0285	0.0074	50mm/min	5.00%	1116.44
11C	Clear	0.2592	0.0259	0.0067	50mm/min	2.40%	1022.88

Table 3 - Tensile Test Results

3. **Capacitance**

The capacitance of the thin electrodes sandwiched with the dielectric elastomer was measured at one kilohertz. The relative permittivity was calculated based on the area, thickness and the measured capacitance. For the Formlabs' Flexible resin, the average relative permittivity value was 5.051 at one kilohertz. The average relative permittivity for the Formlabs' Clear resin was 5.201 at one kilohertz as well. The capacitance depended on the geometry of each sample, but as most of them share similar dimensions the average capacitance obtained with a Formlabs' flexible dielectric setup was 141.0267 picofarads. In the case of the clear resin, the average capacitance was 113.33 picofarads.

Code	Material	Thickness [in]	Area [m²]	Capacitance [pF]	ε_r @ 1khz
1FC	Flexible	0.0280	0.0020	142.82	5.660098
2FC	Flexible	0.0280	0.0020	150.76	5.974768
3FC	Flexible	0.0208	0.0020	139.62	4.110436
4FC	Flexible	0.0302	0.0020	142.94	6.109949
5FC	Flexible	0.0220	0.0020	125.12	3.896066
6FC	Flexible	0.0222	0.0020	144.9	4.553006
1CC	Clear	0.0314	0.0020	94.08	4.181228
2CC	Clear	0.0308	0.0020	112.92	4.922645
3CC	Clear	0.0322	0.0020	135.44	6.172765
4CC	Clear	0.0300	0.0020	106.04	4.502647
5CC	Clear	0.0372	0.0020	115.9	6.102436
6CC	Clear	0.0328	0.0020	115.62	5.367644

Table 4 - Capacitance Test Results

4. Coefficients

The above measured properties can be plugged into the coefficient equation described in Chapter 3. This equation comes from the pairing of Maxwell stress equation with a simple linear model that describes the deformation of an elastic material.

$$Coef_{DEA} = \frac{Relative\ Permitivity}{Young's\ Modulus\ [MPa]}\left(Dielectric\ Strength\left[\frac{MV}{m}\right]\right)^2$$

For the two materials tested and the 3M VHB, the coefficients are presented in the *Table 5 - Coefficients* As expected, the VHB material scored a high coefficient with 61 646.59 whereas the Formlabs' flexible resin, scored an 82.38. Finally, the Formlabs' Clear resin scored a 0.751 approximately. As mentioned before, this number as a standalone does not provide information. When compared with the scores other materials obtained, it provides a comparative position on how much better a material will perform in comparison to the other one.

MATERIAL	RELATIVE PERMITIVITY []	DIELECTRIC STRENGTH[MV/M]	YOUNG'S MODULUS [MPA]	COEF
VHB	3.21	65	0.22	61 646.59
FORMLABS' FLEXIBLE	5.051	12.526	9.62	82.381
FORMLABS' CLEAR	5.201	10.66	787.35	0.75064

Table 5 - Coefficients

5. Other Materials

The software GRANTA CES EduPack 2017, provided access to an enormous database of material, among which we were able to search for the one that will be likely to score higher than the 3D printed materials from FormLabs. In order to filter the more than 3700 materials available as thermoplastic polymers, four requirements were set:

- Minimum a dielectric constant of 2

- Minimum a dielectric strength of 5MV/m

- Minimum an elongation at break of 200%

- Maximum a Young' module of 0.001 GPa

The software suggested 10 materials that reach the minimum requirements. Those materials are listed in the *Table 6 - EduPack Materials*, the coefficient is also included for comparison analysis. Even though no material's coefficient is at the magnitude than the one from the VHB, there are some materials that reached interesting scores, such as the PVC – Elastomer (Shore A35), Ethylene Acrylic Rubber, Acrylic Rubber and Acrylic Rubber (ACM). In the *Figure 7 - Materials sorted by EduPack* the materials are sorted and placed

with respect to their Young's modulus and their Dielectric Strength. The graphic

representation allows the user to understand where does the materials stand with respect to

the others.

| MATERIAL	RELATIVE PERMITIVITY []	DIELECTRIC STRENGTH [MV/M]	YOUNG'S MODULUS [MPA]	COEF
VHB	3.21	65.00	0.22	61,646.59
PVC - ELASTOMER (SHORE A35)	6.00	29.55	0.77	6,808.60
ETHYLENE ACRYLIC RUBBER	4.50	31.00	1.17	3,712.02
ACRYLIC RUBBER (ACM)	4.00	31.00	1.22	3,163.79
ETHYLENE VINYL ACETATE RUBBER	3.60	24.50	1.17	1,854.85
SILICONE	2.70	18.00	0.21	4,165.71
ETHYLENE PROPYLENE (DIENE)	2.60	29.50	1.17	1,933.89
SIS (SHORE A45)	2.65	20.00	1.23	862.49
SIS (SHORE A30)	2.65	20.00	0.50	2,120.00
BUTYL HALOBUTYL RUBBER	2.35	20.00	1.20	783.33
SEBS (SHOREA35)	2.20	29.20	0.77	2,437.70

Table 6 - EduPack Materials
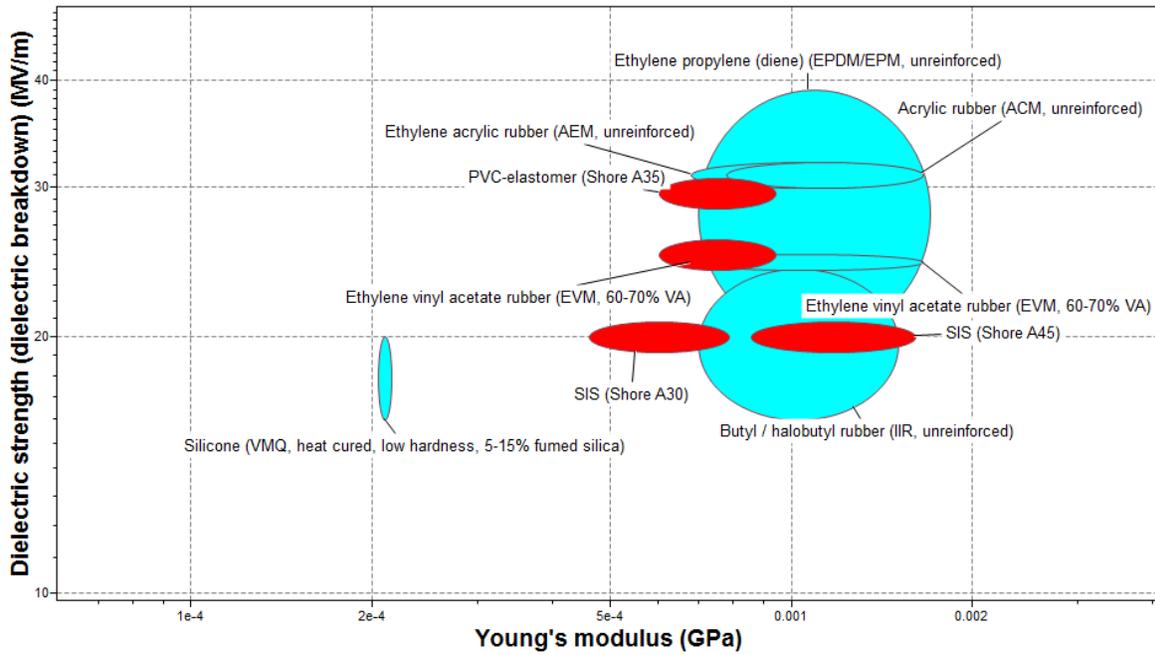
Figure 7 - Materials sorted by EduPack

CHAPTER V

CONCLUSIONS

1. Four properties can be considered crucial when studying a possible dielectric elastomer: Relative Permittivity, Dielectric Strength, Young's modulus and Elongation at break. Based on those properties, much of the material behavior and response can be modeled.

2. The mathematical model presented is limited by its 1D analysis of the phenomena, future work will include a 3D approach to the electromechanical coupling.

3. The Formlabs' flexible resin presents a relative permittivity of 5.05, a dielectric strength of 12.53MV/m, a Young's modulus of 9.62MPa and an elongation at break of 36.07%.

4. The Formlabs' clear resin presents a relative permittivity of 5.20, a dielectric strength of 10.66MV/m, a Young's modulus of 787.35 MPa and an elongation at break of 6.22%.

5. Neither the Formlabs' Flexible Resin or the Formlabs' Clear Resin are suitable to be used as dielectric elastomer due to the following reasons. First, the elongation at break is considerably lower than the one of a conventional elastomer. Second, none of the materials were able to pass the 100% elongation. Third, the materials dielectric breakdown is several times inferior than the VHB's one. Four, the materials are not capable of pre-stretching to enhance deformation under load. As a result of this, they scored very low in the coefficient.

6. There are some other alternatives of thermoplastics that present good properties and good potential to be used as dielectric elastomer. Four worth of highlight are PVC –

elastomer (Shore A35), Ethylene Acrylic Rubber, Silicone, and Acrylic Rubbe (ACM). As all the mentioned materials are thermoplastics, it is possible to develop an additive manufacturing technique for them.

7. The suggested coefficient can be used as a reference for selecting materials. However, it does not provide information as a stand-alone value and requires to be compared with other for different materials. Further study of the involved parameters can be included in future work.

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