IMPLEMENTING PEDOT:PSS AS A CO-FILLER FOR ELECTRICALLY CONDUCTIVE ADHESIVE APPLICATIONS

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ABSTRACT
Electrically conductive adhesives (ECAs) with hybrid fillers have attracted considerable attention due to their lower processing temperature, higher conductivity, simpler processability and finer-pitch capacity. Compared with traditional soldering technology, ECAs offer an environmental-friendly bonding solution in interconnections. In this work, we demonstrated that poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) could be applied as a conductivity enhancing agent in the epoxy and silver micro flakes system to develop a hybrid nanocomposite adhesive. The electrical conductivities of the hybrid ECAs with a constant total amount of silver flakes at various PEDOT:PSS weight concentrations were investigated. It was found that adding a small amount of PEDOT:PSS (0.09 wt%) remarkably improved the electrical conductivity to 289 S/cm, which is 3 times higher than that of the conventional ECA with 60 wt% sliver flakes. The maximum conductivity of 2422 S/cm was achieved at 0.89 wt% PEDOT:PSS concentration. The adhesive strength (or shear strength) was also evaluated for increasing weight loadings to determine whether there was an adverse effect when adding PEDOT:PSS into the composite. It was determined that as the weight loading of PEDOT:PSS was increased, the shear strength variance increased with it. Furthermore, the shear strength also appeared to have slightly decreased with higher weight loading. These results were presumed to come from various sources, however, it is suspected that the main culprit is excess water molecules remaining in the mixture even after evaporation as a result of the large amounts of PEDOT:PSS solution present in the process. Overall, the incorporation of PEDOT:PSS as a co-filler has shown a good potential in ECA applications.

Keywords:
Electrically Conductive Adhesives, PEDOT:PSS, Shear Strength, Electrical Conductivity

INTRODUCTION
The demand and advancement of electronic devices have been growing rapidly within the past few years and the development for new and improved interconnection technologies is essential to fuel its continuous progression for higher performance, reliability and cost reduction [1–3]. The prevalent interconnect technology has been traditionally based on tin/lead and other metals, but recently, lead-based interconnecting materials have been deemed a major concern for the environment due to its toxicity [4–7]. As such, many works have been dedicated to finding friendlier alternatives that can one day replace lead-based solders, explaining the popularity behind the investigation of electrically conductive adhesives (ECAs) as its alternative [4–9]. ECAs offer various advantages besides environmental friendliness; in specific, it offers finer pitch, lower processing temperatures, fewer processing steps, and flexibility [4,5,10,11]. These alternatives to traditional lead-based solder can encompass multi-layered printed circuits, thin-film transistors, transparent conductive coatings, solar cells, flexible electronics, displays such as

LCD/LED/OLED and other various chip attachment techniques [5,11,12]. ECAs are primarily composed of two components: the first being a polymeric matrix, typically in the form of a thermoset such as epoxy resin thanks to its excellent thermal and mechanical stability, adhesion and cost, while the second component is a conductive filler that is usually in the form of silver flakes due to its thermal and electrical conductivities exhibited at room temperature, as well as the conductive nature of its oxide [4,5,7,9,10]. However, there are still many disadvantages to ECAs that must be conquered before fully utilizing its potential: namely its inferior electrical conductivity due to unstable contact resistances, thermal and mechanical instability upon adding conductive filler, environmental reliability and high cost of materials and processing [4,5,10–14]. In this work, we present our newly developed hybrid ECA composite material that utilizes a conductive polymer PEDOT:PSS as its co-filler.

PEDOT is among the few known conducting polymers classified as a polythiophene derivative [15–19], whereas the PSS is another polymer that has three functions: first, it acts as the medium at which PEDOT polymerizes in to become stable water-soluble [17,20]. Second, it acts as a large dopant for PEDOT allowing for the introduction of charge carriers within the polymer backbone of PEDOT [16]. Finally, PSS functions as a charge-balancing group (or counterions), providing a negative charge from the SO_3^- end-group for the positive charge seen on the thiophene group in PEDOT [17,20,21].

It has been a few decades since Shirakawa et al, discovered the very first conductive polymers in 1977, but now, there are a handful available in the market as outlined by R. Balint et al [16]. Conductive polymers are divided into three organic groups: sulfur-containing heteroatoms (thiophenes and sulfides), nitrogen-containing heteroatoms (polymerized pyrrole, aniline, etc), and non-heteroatom containing polymers with numerous alkenes. In specific, PEDOT:PSS was first produced by Bayer AG research laboratories in Germany [17,19] since the 1980s and has since been the topic of great research and industrial interest, being hailed as one of the most successful conductive polymers used in commercial use today [18,19,22,23]. There are many examples of where PEDOT:PSS has already been applied such as energy-based PV technologies [18–22,24,25], electronic semiconductor devices that require excellent electron/hole transport such as field-effect transistors, LCDs, LED based technologies, anti-static bags [17,18,20–23,26], and finally, bio-sensing/bioengineering applications [16,25].

PEDOT:PSS films contain work functions ranging from 5-5.2 eV and induces charge transfer at very high kinetic rates [19]. Its conductivity comes from a variety of factors, however, only two will be mentioned in this work. The first is its conjugated backbone that forms alternating double bonds. The presence of weakly localized π-bonds that overlap with each other, giving rise to the possibility for electrons to become delocalized and move or "jump" through the polymer backbone and as a result, move between groups of atoms [16].The second factor that makes conductivity possible is the presence of dopant(s) that act as counterions that keep the conducting polymer stable, but at the same time is a charge carrier source that is responsible for adding/removing electrons from the polymer chains to become what are known as polarons or bipolarons [16]. These polarons/bipolarons move through the polymer chain as an electric potential is applied onto the system, which in turn allows for charges to pass through the backbone resulting in electrical conductivity. A more comprehensive explanation on what polarons/bipolarons are can be found from Bredas and Steer, whom also explain the entire conducting mechanism of conductive polymers from a chemist's point of view, as well as explains the possible miscommunications that can arise as a consequence of using the term "dopants" to explain the conductivity mechanism in conductive polymers [27], whereas good diagrams and cartoons that explain this mechanism in a simpler manner are found in Balint et al's work [16]. To add to this, Crispin et al presents a chart illustrating the conductive evolution of PEDOT:PSS as a function of weight percentage with the addition of DEG [20]. Their chart shows a percolation curve between 0.1 and 1 weight percent showing synonymous trends to the percolation curves for conventional ECAs. As such, it was a natural link that inspired this work to explore the use of PEDOT:PSS as a co-filler within the conventional ECA in hopes of being able to either improve the conductivity limits of the conventional ECA, or at least reduce the silver content required to decrease material costs. In specific, this work will explore the electrical conductivity of a hybrid ECA that uses PEDOT:PSS as its co-filler, as well as investigate how adding increasing amounts of

PEDOT:PSS will affect the mechanical properties (in this case, adhesion strength by means of evaluating shear strength). To the knowledge of the author, no work regarding the use of PEDOT:PSS in composite systems for the sake of improving electrical conductivity has been undertaken, as most current work utilizes PEDOT:PSS as a film for varying applications rather than a solution-based component. Therefore, this work will be primarily act as a preliminary study to determine its feasibility as a solution-based co-filler in hopes of opening a new avenue for modifying and improving the performance of conventional ECAS.

EXPERIMENTAL SECTION

Preparation of conductive composites

Diglycidyl ether of Bisphenol-A liquid epoxy resin (D.E.R. 331) and Triethylenetetramine (TETA) epoxy hardener (D.E.H. 24) was supplied by DOW Chemical Company and were used as the resin base and the hardener respectively. A high conductive grade of Poly(3,4-ethylenedioxythiophene)-Poly(styrenesulfonate) (PEDOT:PSS 1.1% in water), was purchased from Sigma-Aldrich and used as the conductive filler material for the composite. Methanol HPLC grade was purchased from Sigma-Aldrich and used as a common solvent between the water and the resin to assist with dispersion and mixing, as well as to wash out the PSS content in the solution. Silver micro-flakes (density of 10.49 g/cm^3, 10 µm particle size) were purchased and received to act as the principle conductive material of the composite.

The procedure for making the conductive composites begins by adding the epoxy resin with both PEDOT:PSS solution, and methanol solvent [21]. The resulting mixture is first mixed vigorously using a vortex mixer to ensure that the aqueous PEDOT:PSS is dispersed in the epoxy resin. Once complete, silver flakes are added into the mixture and then mixed further at 2000 RPM for 5 minutes using a Thinky Mixer (ARE 310). The mixture is then placed into the oven at 90 °C in order to allow both the methanol and water content to evaporate. Finally, epoxy hardener TETA is added into the system and again mixed at 2000 RPM for 5 minutes. The mixture is then placed into either a pre-cleaned 7 mm x 7 mm mold made of a glass slide, thermally resistant tape and a copper sheet (to ensure that the sample thickness remains constant) or a shear test coupon that is explained in an upcoming section. The samples are then placed into an oven to be baked at 150 °C for 2 hours. Figure 1 is a schematic that illustrates and summarizes the entire procedure.

Characterization Methods

The electrical sheet-resistance values of the cured composites were measured using a four-point probe setup that includes a probe fixture (Cascade Microtech Inc.) and a micro-ohm meter (Keithley 2440 5A Source Meter, Keithley Instruments Inc.). The sheet resistance R_s obtained can then be inserted into the bulk resistivity equation and then converted into conductivity with the following equations [10,28] (Amoli Ag NP, Binley 1996):

Bulk Resistance

$$\rho = R_s \cdot t = F \cdot t \left(\frac{\pi}{\ln 2}\right)\frac{V}{I} \quad (1)$$

Conductivity

$$\sigma = \frac{1}{\rho} \quad (2)$$

Combined Equation

$$\sigma = \frac{1}{F \cdot t}\left(\frac{\ln 2}{\pi}\right)\frac{I}{V} \quad (3)$$

Where t represents the thickness of the sample, while I and V represent the applied current and measured voltage from which the sheet resistance value is based upon.

The lap-shear strength values of the cured composites were measured using a Universal Material Tester (UMT) Tribological Test Equipment (CETR Campbell) equipped with tensile wedge grips (G1061-2, Mark-10 Corp). The test procedure followed ASTM D1002 with the exception of reducing the contact area to 12.7 mm x 12.7 mm in order to accommodate the load cell used in the experiment [29]. 1/16 inch thick double sided FR-4 boards were used as lap shear coupons and were purchased from MG Chemicals. The boards were machined to meet the dimensions specified in Figure 2.

RESULTS AND DISCUSSION

Electrical Properties of the Hybrid ECA composite

The electrical conductivity of pristine PEDOT:PSS was characterized by a standard four-point probe setup in solution cast films. It was found that the electrical conductivity of pure PEDOT:PSS was about 15 S/cm.

In order to investigate how PEDOT:PSS influences the overall electrical conductivity of their polymer composites, we used conventional ECAs containing only silver flakes at various weight fractions as a control. For all hybrid ECA samples with PEDOT:PSS co-fillers, the absolute amount of sliver flakes was kept constant, whereas the amount of PEDOT:PSS solution was increased. The following formulations used for testing are summarized in Table 1.

Table 1: Summary of different sample compositions and the masses of the conductive filler material

Sample	Composition	PEDOT:PSS water mass (mg)	PEDOT:PSS mass (mg)
A	Epoxy + Ag 60 wt%	0	0
B	Epoxy + Ag 60 wt% + PEDOT:PSS 0.1 wt%	33.9	0.339
C	Epoxy + Ag 60 wt% + PEDOT:PSS 0.2 wt%	67.9	0.679
D	Epoxy + Ag 60 wt% + PEDOT:PSS 0.5 wt%	170.3	1.703
E	Epoxy + Ag 60 wt% + PEDOT:PSS 1 wt%	342.4	3.424

Figure 3a illustrates the expected conductivity of conventional ECAs that typically contain only silver flakes as its main conductive filler as a function of weight percent loading. It is seen that past a certain point in the curve known as percolation threshold (within the range of 40 wt% loading), the composite transitioned from being an insulating material into a conductive material. Furthermore, by looking at the higher weight loadings, it is seen that adding more silver flakes into the epoxy matrix did not significantly improve electrical conductivity. In contrast, the electrical conductivities of the hybrid ECAs with a constant total amount of silver flakes at various PEDOT:PSS weight concentrations were plotted in Figure 3b. It was found that adding a small amount of PEDOT:PSS (0.1 wt%) remarkably improved the electrical conductivity to 289 S/cm, which is 3 times higher than that of the conventional ECA with 60 wt% sliver flakes. The maximum conductivity of 2422 S/cm was achieved at a weight loading of 0.89 wt% PEDOT:PSS showing that the combination of PEDOT:PSS and conventional ECAs produces highly conductive composites. It is further determined that like conventional ECAs, adding more PEDOT:PSS into the composite past a certain concentration yields reduced improvement to electrical conductivity. This decrease in electrical conductivity may be attributed to the fact that increasing the amount of PEDOT:PSS into the system also increases the potential of residual water molecules from the PEDOT:PSS solution thereby decreasing the crosslinking density of the epoxy as it is being cured leading to fewer metallurgic interconnections between the silver flakes [9,30]. Thermal studies can be done to further investigate and verify this behavior.

Mechanical Properties of the Hybrid ECA composite

The mechanical property that was observed was the shear strength of the adhesive in order to determine if the composite remains functional as a joining material after adding co-filler PEDOT:PSS into the system. The shear strength was investigated using (CETR UMT) universal mechanical tester with a load cell of 100 Kg and Mark-10 wedge grips G1061-2 with gripping force of 1-9 kN, while following ASTM D1002 with exceptions that were specified in a previous section. The shear strength as a function of PEDOT:PSS weight loading was investigated with the results summarized in figure 4.

Overall, it was observed that addition of PEDOT:PSS into the conventional ECA (where the weight loading of silver flakes was kept constant) shows both an increase in variance with an increase in PEDOT:PSS, as well as an apparent decrease in adhesive strength by 11%. One possible explanation for this is that the addition of another component into the epoxy matrix resulted in more variability in the system (whether it comes from change in dispersion efficiency, addition of processing steps giving room for error, contamination issues or thermal instability) leading to weakened adhesion. However, it is important to note that the change in shear strength is only 11% when compared to the increase in electrical conductivity of the similar weight loading of PEDOT:PSS.

It is important however, to examine the reason for why the shear strength tends to decrease. One explanation must take into consideration that the PEDOT:PSS comes as an aqueous dispersion at a concentration of ~1%, whereas the rest of the solution is primarily water based. As such, it is highly likely that the evaporation step that is intended to release all of the water that is introduced after adding PEDOT:PSS does not release all of the water in

the system, leaving trapped water molecules within the resin.

It is known that the presence of water in epoxy resin results in the compromising of its crosslinking density [31,32], thus leading to the degradation of both electrical conductivity and mechanical strength. With this in mind, it is very likely that the variability of how much water molecules are evaporated out of the system is directly related to both the bulk strength of the material, as well as its electrical conductivity. It is intuitive to know how increasing the crosslinking density results in better mechanical strength, however, the reason that crosslinking density is related to the bulk resistivity of the system needs to be explained through the concept of increasing shrinkage in epoxy. Increased shrinkage within the thermoset leads to better intimate contact between silver flakes, resulting in the formation of more conducting pathways [5,9,12,33–35]. Two avenues that were not explored in this study is the effect of methanol on the crosslinking density of the epoxy. Firstly, no explicit mention that methanol has a negative effect on crosslinking density of epoxy was found, however, it is suspected to be a potential contributing factor when it comes to the incomplete curing of the epoxy, or even the formation of air bubbles during evaporation. Second, dispersion issues could lead to problems in the utilization of PEDOT:PSS as it is not well known whether the actual active component is aggregated or isolated in small pockets within the composite system upon curing. Furthermore, poor dispersion is suspected to also act as potential weakness in the structure of the cured composite, leading to variance in its mechanical strength.

CONCLUSIONS

PEDOT:PSS was successfully introduced into a conventional ECA formulation; its electrical conductivity and shear strength was investigated. The electrical conductivity results from the 4-point probe test have shown that the incorporation of PEDOT:PSS improves the conductivity of conventional ECAs to 289 S/cm, which is three times more conductive than without the co-filler. The maximum conductivity found in this investigation when incorporating 0.89 wt% PEDOT:PSS into the conventional formulation yields a conductivity of 2422 S/cm. The co-filler system is successful at reaching high conductivity values without increasing the silver content within the ECA, and not requiring an extra 10-20 wt% of silver to achieve the same results.

The mechanical strength of the hybrid ECA was also investigated by closely following the ASTM D1002 standard but with some modifications to the testing area. By varying only the weight loading of PEDOT:PSS, it was found that the increase of the co-filler increased the variance of the shear strength of the system, as well as a slight decrease of 11% in shear strength (1 MPa at worst case). It has been explained that the increase in variance could be from a variety of factors, however, it is suspected that the introduction of PEDOT:PSS at higher weight loadings resulted in excess water molecules that were trapped in the composite because of insufficient evaporation, leading to the varying shear values. Overall, it has been found that the incorporation of PEDOT:PSS on the point of view of both electrical conductivity and mechanical strength that is positive. It increases the electrical conductivity of the conventional ECA while simultaneously reducing the required amount of silver to be functional, as well as exhibits negligible impact on its shear strength. This newfound use of PEDOT:PSS as a co-filler in conventional ECAs can lead to a variety of innovative applications in solder mount technologies, as well as flexible electronic joining materials.

FUTURE WORK

The incorporation of PEDOT:PSS into conventional ECAs has been shown to be effective at improving conductivity at little cost to its mechanical properties. However, future work should be undertaken to answer the question of whether dispersion had a significant effect on the shear strength variance experienced by the hybrid ECA. More investigation as to whether methanol has negative effects on the shear strength should also be examined and this can be done by further investigation into thermal stability and curing mechanics, in hopes for a better understanding on exactly how solvents are influencing the properties of the hybrid ECA. Finally, more work should be done to perfect the procedure of finding better dispersion agents that will assist in preparing the composites in a more efficient and effective manner.

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BIOGRAPHIES

Josh Trinidad is a Chemical Engineering-Nanotechnology MASc student working in the Surface Science and Bionanomaterials Laboratory at the University of Waterloo. He obtained his BASc in Nanotechnology Engineering in 2013 from the University of Waterloo. His research interests include electrically conductive polymers with a focus on improving the processing and synthesis, micro and nanocomposites, nanomaterials such as graphene and silica nanoparticles, adhesives and adhesion mechanics, mechanical strength studies (tensile, shear, indentation tests on a variety of materials) and mechanical designing.

Dr. Wei Zhang is a Chemical Engineering-Nanotechnology Ph.D graduate currently working as a Post Doctoral Researcher in the Surface Science and Bionanomaterials Laboratory at the University of Waterloo. He obtained his MASc in Chemistry in 2011 from the University of Waterloo, and his BSc in Materials Science from Huaqiao University in China in 2009. His research interests include but are not limited to synthesis and functionalization of bio-inspired materials, chemical modification of electrically conductive polymers and adhesives, contact mechanics studies (such as adhesion, friction and wetting behavior of surfaces) of synthetic biomimetic materials, and interfacial phenomena in the nanoscale.

Alex Chen is a technology development engineer at Celestica Inc. His primary responsibilities include process development, technology implementation and manufacturing site qualification. He has experience in electronics failure analysis for the past decade and is recently working with customers to develop reliable performance evaluation methodologies. Alex is a MASc graduate from McMaster University and has been working in IBM and Celestica for over 20 years.

Dr. Boxin Zhao is an associate professor in the Chemical Engineering Department at the University of Waterloo. Dr. Zhao is a Chemical Engineering Ph.D from McMaster University since 2004 and afterwards worked as a Post Doctoral Researcher at the University of California in Santa Barbara from 2005 to 2008. Since then, he has joined the University of Waterloo and established a Surface Science and Bio-nanomaterials Laboratory at the University of Waterloo, where he focuses on both academic and applied research to advance the ever growing field of bio-nanotechnology. Dr. Zhao is working on developing multi-functional, green and smart materials as well as clean-processing and additive manufacturing and 3D printing. He holds the Early Researcher Award for the Province of Ontario in 2012 and is frequently invited to deliver presentations, lectures and talks to Celestica, Xerox, Magna and other conferences both in the national and international levels.

Robert Lyn is the founder and CTO of Microbonds Inc. Robert holds a BA Sc degree in Mechanical Engineering from the University of Toronto, and has over 20 years of experience in the microelectronics industry working with Microbonds, IBM Microelectronics and Celestica Inc. Prior to co-founding Microbonds Inc., Robert was a senior staff engineer in the advanced packaging development division of IBM and subsequently Celestica where he led a team that introduced a number of strategic microelectronics packaging technologies into the manufacturing process, including Chip-on-Board wire bonding technology, Flip Chip technology, Ball Grid Array chip carriers, Multi-chip modules, and single chip packaging. These technologies have supported product offerings, such as; PCMCIA Notebook cards, SIMM memory cards, RF/Wireless cards, Networking cards, Power Technology, PC motherboards, Workstations, Mainframe computers and Internet routing systems

John Persic is the founder and V.P. of Research and Development of Microbonds Inc. John holds a BA Sc degree in Material Science from the University of Toronto, as well as a MASc degree in Applied Science from the University of

Toronto, specializing in the area of Metallurgy and Material Science. John has nearly 10 years of experience in the microelectronics industry. At Celestica, he served as a staff engineer in the process engineering and development division working on process improvements, new product development, and troubleshooting. As a project leader he was responsible for numerous microelectronics packaging and assembly projects, such as wirebonding improvements, encapsulation, contamination control, lead-free solder, conductive adhesives, flip chip/chip scale package development, fluxless soldering, wave-soldering, SMT improvements, chip carrier design, and microvia qualification and testing. John has served as the ISO 9000 Quality representative for Process Engineering and performed ISO and SCC audits, resolved non-conformances and facilitated ISO 9000 training courses.

Figures

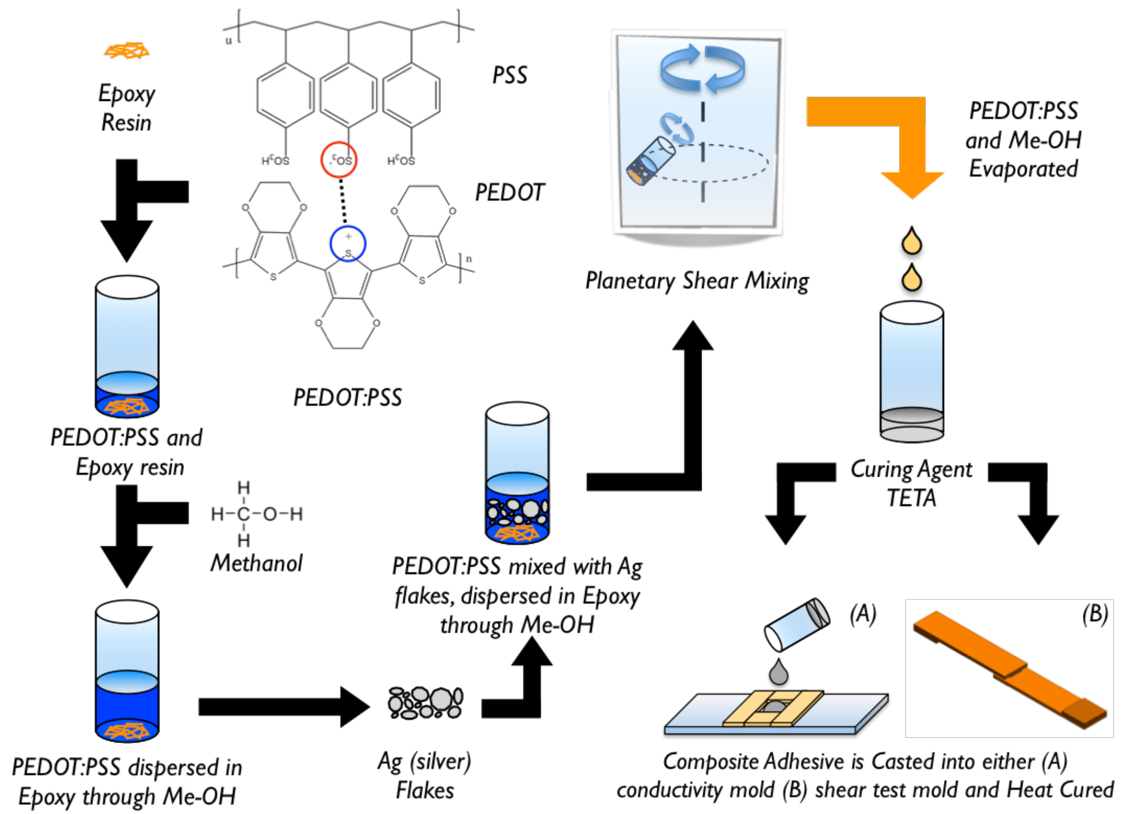


Figure 1. Schematic diagram of PEDOT:PSS as it is incorporated into conventional ECAs**.**

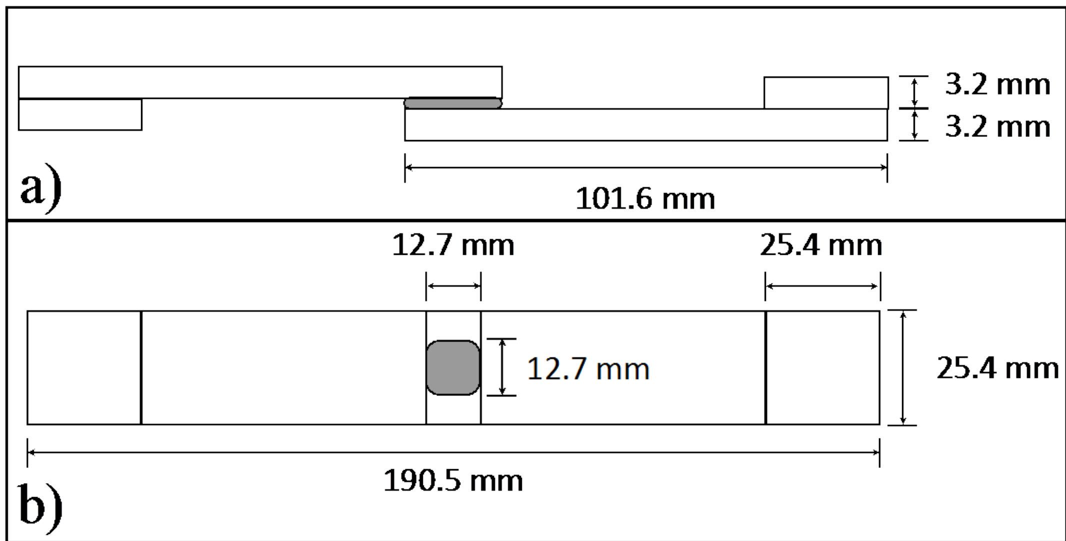
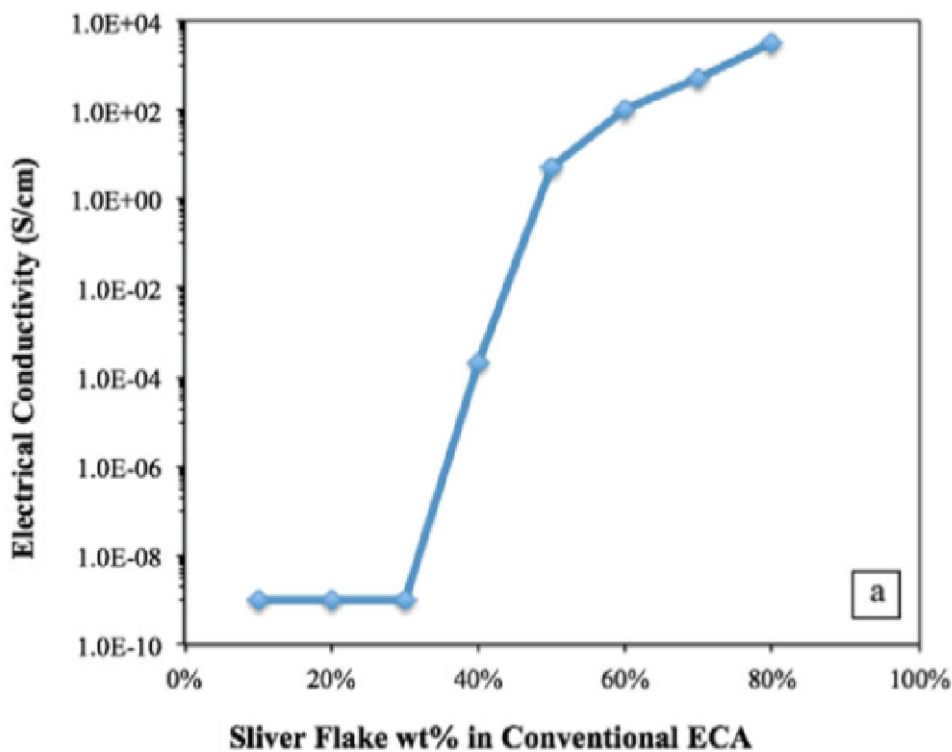


Figure 2. (a) Top view of shear test coupon showing the modification of the paste area with measured values; (b) Side view of shear test coupon with measured values.



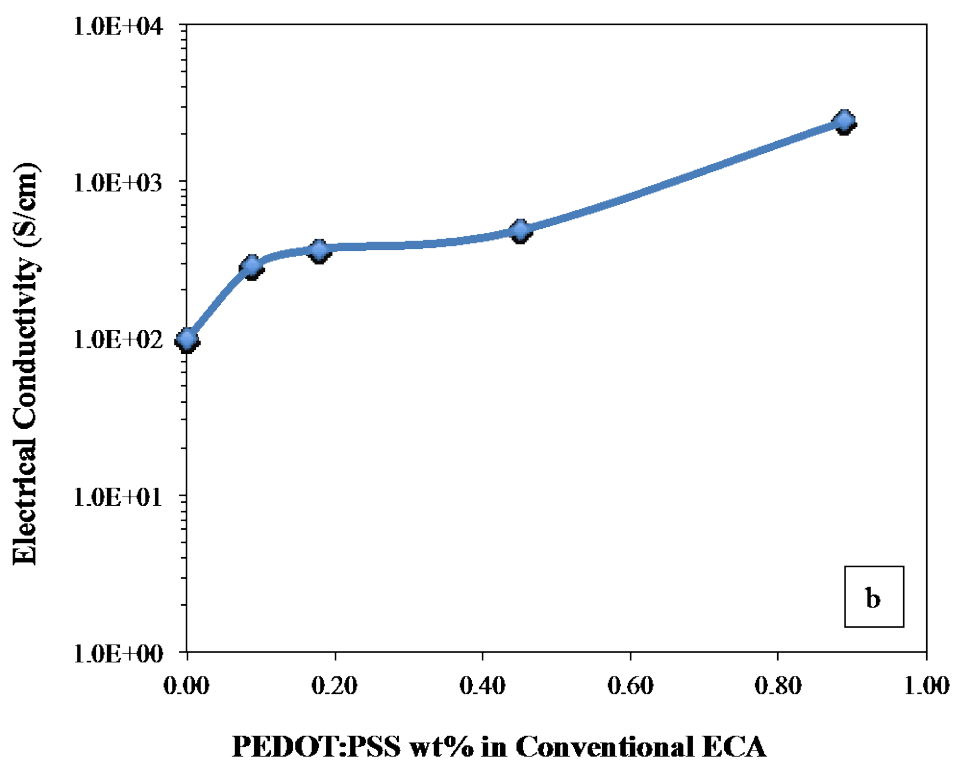


Figure 3. Electrical conductivity as a function of filler content silver flakes (a) and PEDOT:PSS (b) in weight % for the hybrid ECAs in log scale.

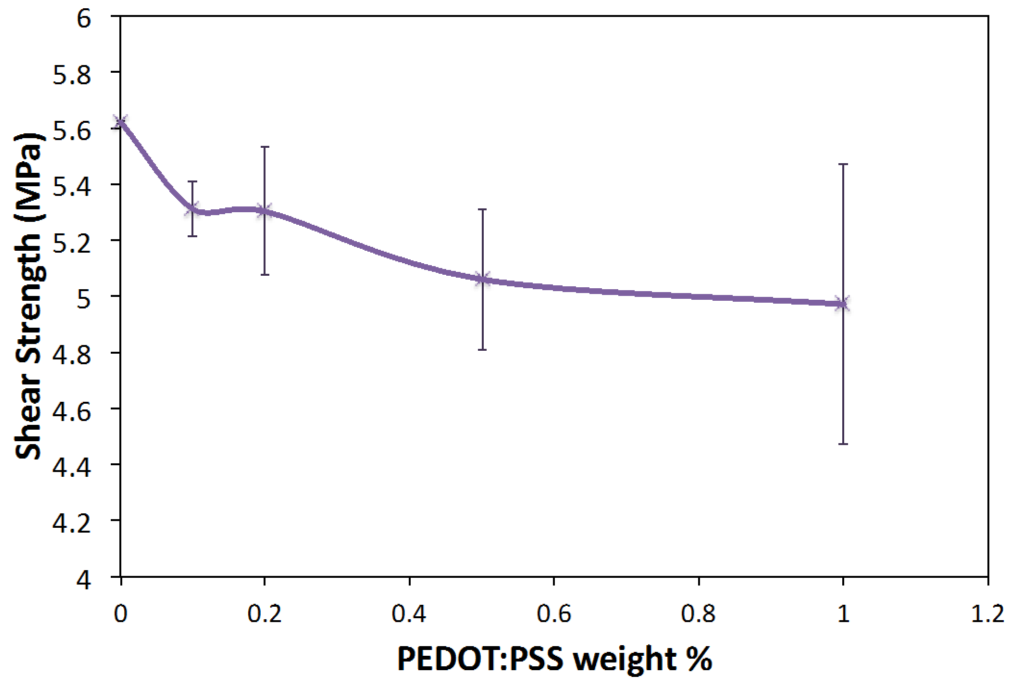


Figure 4. Shear Strength as a function of PEDOT:PSS fraction for the hybrid ECAs.