**DuPont Patent US 9,587,132 B2**

**Thermoformable Polymer Thick Film Transparent Conductor and its Use in Capacitive Switch Circuits**

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1.0 Background

Electronic components are becoming a huge part of both consumer and commercial products such as cell phones, televisions, computers, and car stereos. These devices are comprised of integrated circuits or wiring boards, resistors, wiring, keyboards, touchpads, and chip packaging. A major part of these electronic parts is the conductive coating on them. These conductive coatings are applied to substrate surfaces for different reasons depending on the application, and the compositions vary as well. On March 7, 2017 DuPont received the rights to US Patent No. 9,587,132 B2, Thermoformable Polymer Thick Film Transparent Conductor and its Use in Capacitive Switch Circuits. Team 3 took on the task of modeling and optimizing this process with the use of Simulink and MATLAB softwares.

This invention solves problems with conductivity and compatibility of substances. A conductive ink is printed and dried on a substrate to produce a functioning circuit that undergoes thermoforming, subjecting it to pressure and heat that deforms the circuit to its desired three-dimensional characteristics. The PTF layer can be used on a substrate of choice or as part of a capacitive circuit stack with silver. Different embodiments of the mixture are used for different applications, but the overall composition is as follows: 10-70 wt% conductive oxide powder, 10-50 wt% of a first organic medium made of urethane resin dissolved in an organic solvent, and 10-50 wt% of a second organic medium with a polyhydroxyether resin dissolved in an organic solvent. Some embodiments contain a third organic medium, powders and printing aids to improve composition, or metals to improve conductivity.

Team 3 used Example 1 from the patent to model the mixing of the conductive ink. The final product is made by 20 wt% of a first organic medium, 50 wt% of a conductive powder, and 30 wt% of a second organic medium. Each of these components is made as an intermediate, then mixed together in the proper proportions at the end. The first organic medium is made by mixing 20 wt% of polyurethane and 80 wt% dibasic esters. This is mixed at 90o C for 1-2 hours. The second organic medium contains 27 wt% poyhydroxyether resin and 73 wt% organic solvent mixed and heated the same as the first medium. The composition was mixed for 30 minutes and subjected to several passes on a three roll-mill before being printed for circuit fabrication.



2.0 Process Control Objectives

 Main controlled, manipulated, and disturbance variables

Each process was controlled in such a way to achieve a specific value for an output of the process. For the mixing process, a specific weight percent composition of resin versus solvent was the main control objective, accompanied by secondary objective involving accuracy, performance, robustness, safety, and versatility. Controlled variables included the output flow rate, solvent flow rate, compound density, tank volume, and model set points. Manipulated variables included the input flow rate of resin to the tank, while the responding variable was the outlet flow composition in terms of flow rate. The main disturbance variable considered was variations in input flow rate, multiple classes of disturbances were analyzed including a step input, perhaps caused by inlet pipe blockage or faulty control valves, or a randomly generated variance in the flow rate, characteristic of poorly maintained equipment or leakage.

For the heating process, a specific and constant mixing temperature was the main control objective, accompanied by the same secondary objectives. This was achieved by manipulating the heat input into the tank, while keeping variables like flow rate, density, specific heat, and tank volume controlled. The main disturbance analyzed was variable temperature caused by disturbances in heat input, but could be considered as a temperature response from other sources as well. The same two classes of disturbance, a step input characteristic of a sudden rise in temperature or heat input and a randomly generated disturbance characterized by compound impurities or imperfect mixing, were analyzed graphically to assess the successful performance of the model.

3.0 Process Control Strategy

After developing a primary conceptual design of the system, transfer functions were derived from the mass and energy balances of the conceived designs(appendix a, b). These transfer functions could then be implemented into a Simulink model to represent the entire system design taking into account each class of process control equipment. For the mixing process, this included the following 4 components: (1) the composition analyzer and transmitter which measures the outlet composition and relates it to an electrical output fed to the controller, (2) the controller which analyzed the analyzes the electrical input and relates it to a designated electrical response, (3) the transducer, which translates the controller output electrical response into a pneumatic output, which would then be fed to the (4) control valve, which would respond to the pneumatic response mechanically to alter the resin flow rate, and in turn the process transfer function, the fifth transfer function which translates the resin flow rate input into a compositional response. The heating process model was developed in a similar way, though with much lesser complexity, as the only process control equipment included or necessary in the design were the thermocouple/transmitter, controller, and heating element in addition to the process transfer function which would translate heat input into a temperature response.

Without exact equipment data nor the means to test actual equipment, exact transfer functions were not possible to develop, but generic and easily modified transfer functions representing the first order response for each class of control equipment were used.

Once these models were developed, controller tuning remained the final step in the process control strategy. For the mixing process, a PI controller was chosen to control the flow rate of the input resin. Derivative controllers are not typically used in flow control processes, and the mixing process did not inherently have a risk associated with it that constituted a more complicated controller than a PI. With the proportional and integral control components, the controller would be able to respond directly to instantaneous error in the process as well as to the integral error present in the system over time. For the heating process, where error in the system has a higher risk outcome, a PID controller was chosen. With the addition of the derivative control component, the model would be capable of adjusting the manipulated heat input variable in prediction of future behavior based on the slope of the output vs time graph.

Using the derived proportional and integral gains found in chapter 12 of *Process Dynamics and Control*, controller elements could be optimized for both gain and time delay of each element. An additional redundancy mechanism was built into each model which allowed for direct graphical representation and analysis of multiple trials of gains or time delay deviations from the direct synthesis examples available in *Process Dynamics and Control*, greatly improving the method for controller tuning. All variable were structured as exactly that, variables in the given model, and capable of being manipulated in the same command window using the Model Initialization Callback Function for model parameters. This method resulted in the following values for the arbitrary equipment transfer functions modeled in the mixing system:

Kc=4.1 𝛕I =1.1

And in the heating system:

Kc=5 𝛕I =1 𝛕D =5

The resulting tuned models for each mechanisms responded exceptionally to various disturbances in their corresponding process, both of which still attaining steady state behavior and set point values within 10-20 seconds of controller initialization, a negligible amount of time relative to the process duration.

4.0 Safety

 Safety is a priority in any manufacturing process, so there are several precautions that must be taken during the making of these polymer films. First of all, a Pre-Start-Up-Safety Review (PSSR) was conducted to make sure that the all the equipment in the facility meets all the design and safety parameters. Once the facility is approved, it can be cleaned and energized and then chemicals could be introduced. This process involves heating elements, so one safety component that can be implemented is a feature that will shut off the system if a certain temperature is met. This will eliminate the possibility of the system overheating. Additionally, there is the risk of the pressure in the system being too high. Adding safety valves will help relieve the pressure and control this problem. Fail open and fail close valves could be included throughout the system so that if power or air is lost, the valves will be in the safest possible position. Another potential problem that could occur is the tank overfilling. An overflow would obviously make a mess a result in dangerous working conditions. In order to remedy this, a level indicator could be placed on the tank which would monitor the amount of liquid inside. This would ensure that excess liquid is not accidentally added to the tank if levels are already relatively high.

5.0 Conclusion

Throughout the project, the team had many learnings and takeaways.Upon receiving the task, the team worked to understand the patent from DuPont. Patents are very important in industry, as they exclusively give rights to an entity for an invention. Getting exposure to this patent in particular gave the students insight to designing processes to produce and transform chemicals. Additionally, as Chemical Engineers in training, the students were able to apply concepts from their Chemical Process Control, Modeling and Simulation class to the real world. This course taught all about the application of mathematical functions to model processes in real time.

Further development of the project would include fine-tuning the transfer functions, modeling the process for the conductive powder, and optimization. Obtaining specifications for the equipment and instrumentation would make the model even more accurate because the variables would be realistic instead of assumptions. Also, the team could model manufacture of the Indium Tin Oxide (ITO) powder, or at least include a transfer function to include the parameters of the component to determine how it affects the final product. Finally, process optimization is necessary to minimize cost and maximize throughput and efficiency. The project could definitely be advanced given more time and resources.

6.0 References

Desmocoll 540 Polyurethane SDS: <http://www.productsafetyfirst.covestro.com/sdssearchpage/sds?SearchDigit=&SearchName=Desmocoll+540&SelectedLanguage=EN&SelectedCountry=US>

Dow Oxygenated Solvents:

<http://msdssearch.dow.com/PublishedLiteratureDOWCOM/dh_096d/0901b8038096dc0d.pdf?filepath=oxysolvents/pdfs/noreg/327-00001.pdf&fromPage=GetDoc>

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Dimethyl Succinate MSDS:

<https://cameochemicals.noaa.gov/chris/DSE.pdf>

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Phenoxy PKHH Resins:

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<http://www.brenntag.com/media/documents/bsi/product_data_sheets/material_science/gabriel_phenoxy_resins/inchem_product_brochure.pdf>

ITO Powder:

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<http://www.cen.iitb.ac.in/chemical_approval/msds/318_msds.pdf>

<http://www.us-nano.com/inc/sdetail/364>

ATO Powder:

<http://www.us-nano.com/inc/sdetail/278>

<http://www.reade.com/products/antimony-tin-oxide-ato-powder>

7.0 Appendices

7.1 Appendix A - Mixing Point Transfer Function Derivation

$ρ\frac{dV}{dt}=w\_{1}+w\_{2}+w$ *mass balance*

$ρ\frac{d(Vx)}{dt}=w\_{1}x\_{1}+w\_{2}x\_{2}+w$ *mole balance*

$x\_{1}=x\_{2}=1$

$ρ\frac{d(Vx)}{dt}=w\_{1}+w\_{2}+w$

$ρ\frac{d(Vx)}{dt}=ρx\frac{dV}{dt}+ρV\frac{dx}{dt}$ *chain rule*

$ρV\frac{dx}{dt}+(w\_{1}+w\_{2}+w)x=w\_{1}x\_{1}+w\_{2}x\_{2}+w$ *substitution*

$ρ\frac{dV}{dt}=w\_{1}+w\_{2}-w$

$ρV\frac{dx}{dt}=w\_{1}(1-x)+w\_{2}(1-x)$

$$\frac{dx}{dt}=f(x,w\_{1})$$

$ρV\frac{dx'}{dt}=[\frac{∂f}{∂x}]\_{s }x'+[\frac{∂f}{∂w\_{1}}]\_{s }w\_{1}'$ *linearized differential equation*

$ρV\frac{dx'}{dt}=(-\overline{w\_{1}}-\overline{w\_{2}})(x-\overline{x\_{}})+(1-\overline{x\_{}})(w\_{1}-\overline{w\_{1}})$

$ρV\frac{dx'}{dt}=(-\overline{w\_{}}) x'+(1-\overline{x\_{}}) w\_{1}'$

$ρVsX'(s)=(-\overline{w\_{}}) X'(s)+(1-\overline{x\_{}}) W\_{1}'(s)$

$(\frac{ρV}{\overline{w\_{}}}s+1)X'(s)= \frac{1-\overline{x\_{}}}{\overline{w\_{}}} W\_{1}'(s)$ *Transfer Function*

$$\frac{X'(s)}{W\_{1}'(s)}=\frac{ \frac{1-\overline{x\_{}}}{\overline{w\_{}}}}{\frac{ρV}{\overline{w\_{}}}s+1}=\frac{K}{τs+1} K= \frac{1-\overline{x\_{}}}{\overline{w\_{}}} τ=\frac{ρV}{\overline{w\_{}}}$$

7.2 Appendix B - Heating Process Transfer Function Derivation

$$mC\frac{dT}{dt}=wC(T\_{i}-T)+h\_{e}A\_{e}(T\_{e}-T)$$

$0=wC(\overline{T\_{i}}-\overline{T})+h\_{e}A\_{e}(\overline{T\_{e}}-\overline{T})$ steady state

$$mC\frac{dT}{dt}=wC[(T\_{i}-\overline{T\_{i}})-(T-\overline{T})]+h\_{e}A\_{e}[(T\_{e}-\overline{T\_{e}})-(T-\overline{T})]$$

$\frac{m}{w}\frac{dT'}{dt}=-(T'-T'\_{i})+\frac{h\_{e}A\_{e}}{wC}(T'\_{e}-T')$ deviation variables

$(\frac{m}{w}s+1+\frac{h\_{e}A\_{e}}{wC})T'(s)=T'\_{i}(s)+\frac{h\_{e}A\_{e}}{wC}T'\_{e}(s)$ Laplace transform (a)

$$m\_{e}C\_{e}\frac{dT}{dt}= Q-h\_{e}A\_{e}(T\_{e}-T)$$

 $m\_{e}C\_{e}=0$ negligible thermal capacitance

$$0= Q-h\_{e}A\_{e}(T\_{e}-T)$$

$$0= \overline{Q}-h\_{e}A\_{e}(\overline{T\_{e}}-\overline{T})$$

$0=(Q-\overline{Q}$)-$h\_{e}A\_{e}[(T\_{e}-\overline{T\_{e}})-(T-\overline{T})]$

$0= \frac{Q'}{h\_{e}A\_{e}}-(T'\_{e}-T')$ deviation variables

$T'\_{e}(s)= \frac{Q'(s)}{h\_{e}A\_{e}}+T'(s)$ Laplace transform (b)

$(\frac{m}{w}s+1)T'\_{}(s)=T'\_{i}(s)+\frac{1}{wC}Q'(s)$ combine (a) and (b)

$(\frac{m}{w}s+1)T'\_{}(s)=\frac{1}{wC}Q'(s)$ Input temperature constant

$\frac{T'(s)}{Q'(s)}$= $\frac{1/wC}{\frac{m}{w}s+1}$= $\frac{K}{τs+1} K= \frac{1}{wC} τ=\frac{m}{w}$ Transfer Function