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Electrochromic Eyewear:

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Abstract

Sunglasses provide comfort and safety by reducing eye strain and glare in bright daylight. Individuals needing vision correction have limited options in terms of convenient and aesthetically pleasing sunglass solutions. The current technology, photochromic lenses, activate with UV light absorption but can take over ten minutes to return to their transparent state after exposure to sunlight and do not tint while driving as windshields filter the ultraviolet light needed.

The intent of this project is to use electrochromic thin film technology to design a pair of sunglasses that can be switched between transparent and tinted states by flicking a switch. The primary design goal is to have a working prototype that switches between on and off states in milliseconds or seconds while keeping a constant tint across the radius of the lens. The difference in the transparency in the on and off-states should be greater than 30%, thereby providing adequate shading to be useful to a customer as both regular-wear glasses and sunglasses. The quick switching speed should meet a need not met by the current photochromic sunglasses, such as Transitions. As secondary and tertiary goals, the design should have controllable dimming and reduce ultraviolet light, and be aesthetically pleasing.

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Glossary of Terms and Acronyms

AFM:	Atomic force microscope
CNT:	Carbon nanotube
COMSOL:	A software package for simulation of multiphysics problems
CSA:	Canadian Safety Association
ITO:	Indium Tin Oxide
$LiClO_4$:	Lithium perchlorate
PC:	Propylene carbonate
PET:	Poly(ethylene) terephthalate
PMMA:	Poly(methyl methacrylate)
UV-Vis:	Ultraviolet and visible light spectroscopy

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1.0 Design Overview

Electrochromic materials can reversibly change their colour based on their oxidation state. Providing or removing charge to the electrochromic material by switching the applied voltage across the material and its electrolyte causes a change in oxidation state and hence a change in transparency. Many different chemicals are available for the team to use when designing for an electrochromic application. In the case of this design project, the researchers were concerned with minimizing costs and ensuring that the chemistry was relatively straightforward to avoid costly experimental difficulties, which would be expensive in terms of time and the budget.

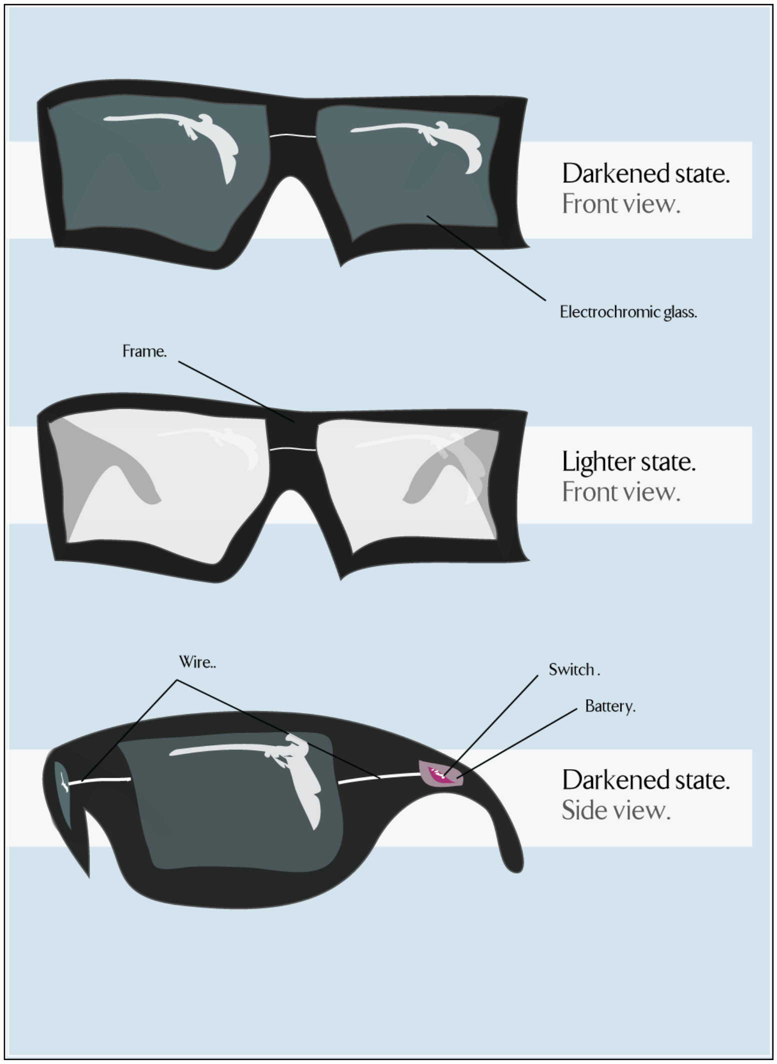

Figure 1: An illustration of a prototype of the design project

During the team's initial meetings at the beginning of the term, the group members discussed the customer requirements (Appendix A) and the challenges that the design would face. The team decided that producing a product that could compete in the marketplace would be their goal. The team envisioned that electrochromic technology if implemented in eyewear could drastically improve the switching speed and transparency that photochromic devices are presently limited by. While catering to eyewear customer of eyewear to improve a client's vision was the original goal, the team decided that sunglasses would be an easier target. The reasoning was that eyeglasses require special curvatures and additional processing techniques that sunglasses do not, so naturally the easier target was chosen for the sake of making the probability that the team could produce a workable prototype more likely.

With weekly meetings, team members gradually formed a more refined impression of how the device would be developed and a detailed project plan was generated for the development of the sunglasses (Appendix B and C). The team consulted literature and experienced professors at the University of Waterloo in an effort to determine approximations for scheduling purposes. Certain techniques, such as using polymer-based electrochromic materials were discarded on the basis that the synthesis of these materials would be too demanding on the group members who lacked the needed expertise. Half-way through the term, the team was faced with a decision between inorganic and organic electrochromic materials. The decision involved carefully weighing the benefits and drawbacks of each, and the functional specifications (Appendix D) were drafted according to how well these materials performed. Looking at the technology, the electrochromics, which made up the device, were important when drafting the specifications because the team knew that unrealistic expectations would result in disappointment when it came time to verifying components (Appendix E) during the next term. The final test plan (Appendix F) aimed to verify that the device will meet its customer requirements.

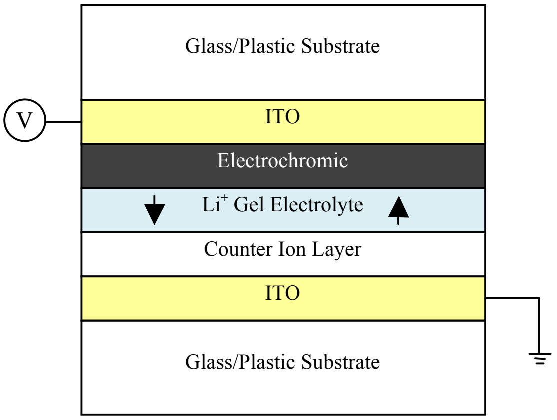

Figure 2: The layers of an electrochromic solid state device

This design report has been written to reflect the process decisions that affected each individual layer and how these decisions impacted the design project in its entirety. The components of a solid-state electrochromic device are independent of each other and their fabrication can be conducted in parallel with the expectation that the integration stage will put the components together. These components are: the substrate, the conductive layer, the electrochromic layer (and possibly the counter ion layer), and the electrolyte. Besides these physical components which are specific to this team's device, budget and purchasing decisions are elaborated. After reading each section, the authors anticipate that the reader will be convinced that the team has successfully navigated the expectations of the project, from customer requirements to its final testing and verification.

2.0 Design Story and Considerations

2.1 Substrate

2.1.1 Customer Requirements

While going through the design process, the team assessed three possibilities for our substrate material; glass, and two plastics (polyethylene terephthalate, and polycarbonate).
Looking at our primary requirements all three materials could potentially be wearable, ensure health and safety, and achieve fast switching time. The problem lies in depositing further layers (indium tin oxide) onto the two plastic substrates. If there isn't an affordable and viable technique for depositing indium tin oxide (ITO) on plastic without melting it, the device would not meet primary requirements at all.

Similarly secondary design requirements (distinct states of transparency, and limited UV exposure) are dependent on being able to deposit layers on top of the substrate successfully. Previously, the team found the advantages of glass to outweigh the disadvantages. It is the cheapest solution, it is substantially easier for deposition, it has no problems with high curing temperatures, it is proven to work for electrochromic devices, and is available with ITO already deposited (provides repeatability).

If poly(ethylene) terephthalate (PET) were used, the team could obtain slides easily, since it is a commonly available material. However, polycarbonate is not commonly available, although the team has been in contact with a few optometrists that have offered to give us samples. Then, the team would request access to G2N to deposit ITO. The team would attempt to obtain the same conductivities that the team outlined for glass by using varying thicknesses. However, the team decided to pursue use of glass sides, and if all the other layers are successfully deposited and characterized for use in a fully integrated functioning device, the team will experiment with layering on polycarbonate and PET in parallel.

It is only a tertiary requirement to have an aesthetically pleasing design that fits the customer nicely. To meet the requirement, the customer would most likely prefer a polycarbonate (like most commercial glasses) which would reduce the weight, and increase the durability and longevity.

2.1.2 Design Plan

We have previously stated that cutting glass would be a design challenge because in order to fit within a wearable frame, the shape and size of the glass will need to be adjusted. Cutting the device may impair its functioning.

The substrate has not changed our milestones throughout the progression of the design process. The initial choice was to purchase glass slides, and the team has stuck to this. However, if the team does finish ahead of schedule, an attempt at a repeat of the device synthesis on polycarbonate or PET would hopefully take less time with the gained knowledge, provided that there are not any new design challenges.

2.1.3 Design Flow

Looking at our design flow diagram, if the team does have sufficient time to repeat synthesis on a plastic, the team would start at the electrochromic/electrolyte synthesis stage, and integrate into our design.

2.1.4 Other Details

Our ensuing functional specifications, verification plan, test plan, and design specifications reiterate the importance of a design of a good base to start on. Although these documents focussed on designing and testing all of the layers on top of the substrate, the device would not meet any requirements or specifications, or pass any testing, if it does not have a reliable substrate to sit atop.

2.2 Transparent Conductive Layers

The conductive layers will be placed on various substrates beginning with glass and if successful, polycarbonate, and mylar. A major requirement of this layer is that it shows high optical transparency and low resistivity as possible. The industry standard transparent conductive oxide is indium tin oxide (ITO) and is widely used in the fabrication of electrochromic windows [**1**]. This guided the group to evaluate the effectiveness of ITO over other conductors, but other options were considered in the process such as carbon nanotubes (CNTs) and gold grid patterns.

Originally the design involved the purchase of sputter targets for depositing ITO onto various substrates such as glass, polycarbonate, and mylar. The cost of these sputter targets was quoted as approximately $800. As the group would likely not be doing large runs of ITO depositions, this was deemed an infeasible cost towards the project goals and this option was discounted. Additionally lab space and red-tape regarding sputtered depositions would be another setback.

2.2.1 Customer Requirements

For the customer requirements, the relevant requirements are fast switching, reduced UV exposure, distinct states of transparency, and a wearable prototype. The electrical characteristics of the conductive layer are important and the team wants as low resistivity as possible without making the device too opaque. This would allow for distinct transparency states. The wearable prototype requirement was considered because the deposition and usage of the conductive layers should allow for a form factor that users expect from eyewear, for example, the conductive layer's ability to be on a curved substrate, as in prescription sunglasses. At this stage the group was evaluating various options.

A newspaper article alerted the group to a company called Unidym who specialize in CNT products. Unidym was releasing a product that was marketed as a transparent conductive electrode on a plastic substrate [**2**]. This was attractive because the ability to bend the substrate into a prescription sunglass was available. However, with some research it was found that the conductivity of this sample was quite a bit lower than ITO. This alone was not the reason for discounting the use of CNT films – the main problem was finding a reliable company to give product specifications, a formal quote, or sample kit. The lack of support during the purchasing process from Unidym and similar companies led the group to believe there may be significant trouble working with such a novel transparent conductor. It was decided that the group should be safe and proceed with a technology that is heavily developed, and with which the team can find a lot of sources of help in our design. This option may become viable in the future as the technology and industry becomes more developed.

2.2.3 Project Plan and Milestones

Soon after drafting the customer requirements, the group discovered that the most effective transparent conductor option was to purchase ITO pre-deposited on a substrate. This saves time because there are no deposition hassles such as access to RF sputterers, authority to change targets, deposition time optimization. ITO can be purchased on plastic PET, mylar, and glass substrates. A source for glass substrates is Delta Technologies, although there are many companies offering ITO on various substrates. The price was also reasonable, at approximately $2.50 for a 1.5"x1.5" slide [**2**]. By purchasing the conductive layers already deposited it saves on issues of repeatability, deposition time, availability, and is actually lower in cost compared to buying a sputter target. This relates to the milestone for ordering common materials that is done very early on in the design. Test samples have been obtained which should suffice until more ITO slides need to be ordered.

2.2.4 Functional Specifications

The functional specifications called for distinct changes in transparency (30-60%). This can be achieved by ITO. Looking at Appendix H, the verification data of the ITO transparency is shown to be as high as 93% in the visible range. This allows a great deal of room for the opacity to be reduced by the electrochromic layers – therefore the team is not limited on the upper limit of transparency. This will also hopefully contribute to the aesthetics of the device because a high transparency in the off state is favourable.

The UV-exposure is limited by the substrate, transparent conductor, and electrochromics. Here the tested ITO functions as a UV blocker for about 50% of the light below 400nm.

2.2.5 Verification Plan

After deciding on purchased ITO on glass as the initial substrate and conductor, it was easier to decide what characterization techniques would be used to test the ability of this layer to meet our requirements. The team decided to verify this layer by four-point-probe resistivity, ultraviolet-visible (UV-Vis) spectroscopy, and thickness measurements using atomic force microscopy (AFM) or ellipsometry. Surface techniques such as AFM may not be as useful as the other methods so they will most likely be left for last. These measurements were completed and are included in Appendix H.

2.2.6 Test Plan

The ITO becomes included in the device after integration, and is no longer independently tested. The previous analysis will however serve to help problem solve the device's issues if any errors are discovered during device testing. For example if the team finds that there is no electrochromic effect, where there was once an effect in our solution – then it will be apparent that the electrodes are not making good contact, or the circuit may not be complete. Using a digital multi-meter, or even a simple probe to test for presence of electrical connection will be useful in this testing procedure.

2.2.7 Design Specification

The ITO layer was not explicitly mentioned in the design specifications because at this stage of the design it is almost assumed to be part of the substrate. The larger problems were the electrochromic layers (whether WO_3 and Prussian blue or organometallic) as well as the gel electrolyte layer.

2.2.8 Future Problems, Design, and Chance of Success

It is still not perfectly clear whether the ITO will provide sufficient charge transfer throughout the lens without having 'fringing' effects, where the perimeter is darker than the interior (see Appendix H), due to a voltage drop from the circular electrodes to the center. Because the resistivity of the ITO is non-zero, there will most likely be a voltage drop across the electrode surface. If the center does not have sufficient potential then it may not darken as quickly as the outer edges. For this reason, the team considered depositing a grid pattern of gold by sputtering on top of the ITO contacts so that the film is conductive throughout. However, according to the analysis in Appendix H, fringing should not be an issue. There are concerns of work function differences, optical transparency decrease, as well as deposition issues such as reliability, thickness and switching time. This decision will have to be made at a later date once the initial device using solely ITO electrodes is made and tested. Reading literature, other research groups have not seemed to find this a problem, so it may not be an issue [**3**].

Given the product has already been tested and met the specifications set out originally, there is a high likely hood that this layer will be successful. It can be measured as greater than 95%.

2.3 Electrochromic

Faster switching time than comparable photochromic eyewear is a primary customer requirement for the electrochromic sunglasses. Current photochromic technology takes around 900 seconds to switch from dark to light any electrochromic material that achieves a time under this benchmark would be considered suitable. All of the electrochromic materials satisfied this requirement.

A secondary requirement was for distinct states of transparency. The expectation of having a 35% minimum difference between the visible light transmissions in the two states was arrived at by researching the capabilities of potential electrochomic materials. Additionally, the group judged what a customer would consider satisfactory for distinct states. Finally, the requirement was set so that it could be achieved with margin for error in the event that impurities for poor fabrication techniques made the electrochromic function below its ideal range.

Tungsten oxide with its Prussian blue counter-ion layer and the hybrid organic-metallic polymer electrochromic materials were selected for their fast switching time and excellent contrast between the on- and off-states. However, while their properties satisfied the customer requirements satisfactorily, it remained to be explored how their selection could align with the team's goals of low-cost, and ease of fabrication. When it was realized in October that lab space would be scarce until 2010, the team had to ensure that the choice of these electrochomics would facilitate efficient synthesis so that the device could be integrated within three months. The electrochromic materials considered included the following:

- PProDot (polymer): Required complex synthesis. This would increase the risk that the team would not have sufficient expertise and would be unable to produce the solution.
- PDot PSS with gold nanoparticles: Very expensive.
- Layer-by-layer: Colouration efficiency (change in opacity) is low (only 5-10%), although cheap deposition technique.
- **Tungsten Oxide: Cheap, extensively researched, easy, new, cheap deposition technique (electrodeposition versus expensive time consuming sputtering). Prussian blue: proven, good colour efficiency, also compare versus Ruthenium (expensive) and Ni (which cannot be electrodeposited).**
- **Hybrid organic-metallic polymer: cheap, easy to fabricate.**

The research papers for both of the chosen electrochromic species suggested a straightforward procedure with relatively little in the way of equipment. The required tools are potentiostat for deposition of the tungsten oxide, a spin coater for the hybrid organo-metallic polymer solution, and general wet chemistry equipment, like glassware, pipets, and reflux glassware.

Because of the simplicity of fabrication, it was decided that the two methods would be done in parallel by different members of the team. Because the cost of the chemicals for the electrochromics were reasonably inexpensive (under budget), the parallel development hedges the risk of failure for development of the most important layer for electrochromic sunglasses. The milestone for completion of the electrochromic synthesis reflects this optimism, providing three weeks for synthesis of both species in parallel. Two weeks were provided for the deposition of the chemicals, with the team feeling confident in the straightforwardness and minimal risks for spin coating and electrodeposition techniques.

2.3.1 Design Flow

The design flow exemplifies how the parallel development is not expected to end with both techniques completing successfully—only one must be usable. While the combined success would be a highly positive achievement for the team and would allow the team to decide which electrochomic material was superior and would possibly allow the team to integrate both types of electrochomic materials, the combined success is not considered a priority. Instead, when one group is successful, the integration of the electrolyte and the electronics will commence.

2.3.2 Functional Specifications

The two functional specifications that came from the electrochromic material decision related to the switching time and transmittance difference between the on- and off-states performance ratings. The minimum acceptable switching time would be in the millisecond range ideally, while the maximum acceptable time would be 30 seconds. While 900 seconds is the current photochromic switching time, the team was convinced that 30 seconds was achievable based on the research they had conducted on electrochromic switching time. For the transmittance, the minimum acceptable transmittance was decided as being 30% while the maximum performance was decided as being 60%. The group selected these numbers because researched showed the minimum to be attainable and the maximum was judged subjectively to be desirable by a customer.

2.3.3 Design Specifications

Because the electrochromic material needed to be tested for its transmittance and switching time, UV-Vis spectroscopy and timers were chosen as the tools for verifying that the design prototype met its specifications, and hence, the customer requirements. The UV-Vis method will only be performed after the electrochromic is integrated between two ITO on glass slides.

The design specification asked the team to use engineering theory to determine the best way to fabricate the electrochromic materials. While the team originally had a general understanding of the fabrication process, the design plan became a detailed document with the actual science of electrochromics standing behind it. A recent paper from the Nature journal became the backbone for the fabrication of the organo-metallic polymer electrochromic, while a textbook provided the foundation for the synthesis of the more traditional electrochomic, tungsten oxide. These resources strengthened the team's confidence in their design plan and forced the team to recognize that certain milestones would need to be updated according to the level of difficulty or ease that the detailed research suggested.

The test plan followed directly from the verification plan, entailing the use of the UV-Vis and timers to ensure that the electrochromic was performing within the required range.

2.4 Electrolyte

2.4.1 Electrolyte Selection

The electrolyte layer acts as both a membrane and source of positive for the device. Under an applied positive voltage pulse, they are pulled to the electrochromic layer where they trigger a change in the ionization of the electrochromic molecules leading to a darkening of the layer. This results in a tinting of the device, which is then in its on-state. When a negative voltage pulse is applied they travel back to the counter electrochromic layer which has complementary oxidation and the device returns to its off-state.

Nafion and solutions containing lithium perchlorate ($LiClO_4$) were the two options for the electrolyte layer most seriously considered. Nafion is used in fuel cell technology and is readily available to be purchased as a film. It works through the transfer of protons [**4**]. $LiClO_4$ is generally mixed with propylene carbonate (PC), and the liquid solution is injected between the electrochromic and electrode layers which are held at a set distance by the use of spacers. It works through the transfer of lithium ions (Li^+) [**5**]. The $LiClO_4$/PC solution can also be mixed with poly(methyl methacrylate) (PMMA) to create a gel [**6**].

2.4.2 Customer Requirements

Several of the customer requirements were relevant to the selection of an electrolyte layer. As can be seen in Appendix A, switching time was judged to be a primary requirement, and second in overall priority. One of the biggest drawbacks in the currently commercially available photochromic eyeglass solution is its slow transition time. Hence improving switching time could make this a more desirable alternative for consumers. Table A.1 compares the switching times of currently available photochromic glasses (900 s) and current electrochromic technology (2 s). The customer requirement was determined to be 30s, which represents a significant reduction in switching time in relation to photochromic lenses and offers a switching speed that the team believes most consumers would find tolerable.

Since hydrogen atoms are smaller than lithium ions, they are also more mobile. This would theoretically allow for faster switching times through the use of Nafion due to the quicker transport of the ions to electrochromic layer and back [**4**]. However, there are problems with using a proton based device and which are outlined later. The gelation of the $LiClO_4$/PC solution was not found to significantly reduce the conductivity of the device compared to the liquid $LiClO_4$/PC solution, suggesting that the difference in switching time between the two would be negligible [**6**].

Health and safety was also a primary requirement, and was judged third overall in priority. The liquid $LiClO_4$/PC solution could pose a risk in terms of leakage. The gelation of this solution through the addition of PMMA resolves this problem somewhat by increasing the structural integrity of the layer [**6**]. Nafion, as a solid film, should not pose any leakage risk. However, the concentration of protons in the electrochromic layer in the on state has been found to cause corrosion at the electrode-electrochromic interface due to the high acidity [**4**]. In particular, WO_3 has been found to degrade very quickly in acidic environments. Therefore, if our device uses WO_3 as the final electrode layer, it would not be commercially viable to use a Nafion electrolyte layer [**3**]. WO_3 is one of the most proven electrode layers, and has been well documented for use with an $LiClO_4$/PC electrolyte [**6**]. This could possibly cause a risk to the wearer if the electrical contacts at this interface begin to corrode since these electrodes extend outside the lenses and could cause corrosion to the electrical wiring or frames to which they are attached.

Electrochromic devices using a $LiClO_4$/PC electrolyte are also well documented in literature, unlike those using Nafion [**7**,**6**,**4**,**5**]. It is unknown whether the Nafion acidity issue could affect device performance overtime. The price of Nafion is also much higher, and the customizability in terms of ions present could be less since it would be purchased pre-made.

The third customer requirement which relates to the electrolyte layer is the distinct states of transparency. This was a secondary requirement which was judged fourth is overall priority. Lithium ions were found to provide a greater colour change than hydrogen ions [**4**].

The $LiClO_4$/PC PMMA gelated electrolyte was found to best fulfill the customer requirements. It is believed to provide a slower switching time than Nafion, but still within the maximum transition time specified. In terms of health and safety, it appears to be the safest option and should provide the greatest distinction between transition states. While the faster switching time that could be possible with Nafion is traded-off in favour of more distinct transparency states, the switching times using of $LiClO_4$/PC gel electrolytes have been proven to meet the 30 second customer requirement in literature.

2.4.3 Project Plan and Milestones and Design Flow

Since the electrolyte layer will be fabricated by the same method and with the same materials for both fabrication routes, it was decided that the electrolyte solution would be synthesized as soon as the material orders arrive. They are expected to arrive by January 4th and the electrolyte synthesis is expected to be completed by January 20th (see Appendices B and C). This accounts for the fact that during this time the group will also be synthesizing the electrochromic solution, as well as the time needed for verification and characterization.

It was also noted that the cost of preparing the $LiClO_4$/PC gel was far lower than that of purchasing Nafion. At the same time, the additional time needed for synthesis, as well as the possibility of inconsistency in success of synthesizing this gel, would require longer time to accomplish, pushing back other milestones and ultimately the completion of the project. This necessitated the parallel synthesis of the electrolyte and electrochromic layer (see Appendix C).

The team determined that general chemistry lab space and glassware will be needed for the electrolyte synthesis. C2-275 is expected to be used to perform the synthesis and characterization.

2.4.4 Electrolyte Considerations for Functional Specifications

The functional specifications that pertain to the electrolyte layer include distinct transparency states, switching time, and fine transparency control, which were rated with priorities of one, four, and seven out of ten, respectively (see Table 1.D).

The team determined that the minimum on-state transparency to visible light should be 30% while the maximum off-state transparency should be 60%. The minimum required transparency change is therefore 30%. According to literature, $LiClO_4$/PC gel has been shown to provide this transparency change when used in combination with Prussian blue electrochromic layer. Therefore only $LiClO_4$/PC gel meets this functional specification.

In terms of switching time, the functional specification specified a maximum transition time of 30 seconds from off-state to on-state or vice versa. Both electrolytes discussed were found to be capable of meeting this specification according to literature.

Finally, due to the slower migration of lithium ions compared to hydrogen ions, it was hypothesized that fine transparency control may be more feasible using $LiClO_4$/PC gel than Nafion since it would be less likely that all the ions would move across in a single voltage pulse.

By this point the team had determined with fairly high confidence that the $LiClO_4$/PC gel was the best option for the electrolyte layer.

2.4.5 Verification and Test Plans

Of the verification techniques presented in Appendix E, UV-Vis spectrometry, cyclic voltammetry, thin film characterization techniques, and timers will be used for the verification of the electrolyte layer. UV-Vis spectrometry will be used to measure the transmittance of the lens in the on and off-states in order to determine if the minimum opacity change has been achieved. This verification can only be done after all layers have been deposited.

In order to initially test and characterize the conductivity of the electrolyte gel, cyclic voltammetry using a potentiostat will be used. Timers with a precision of milliseconds will be used to time the transition between on and off-states to verify that it is below the 30 second requirement set out by the functional specifications.

The number of moles of lithium per volume needed will also be determined through literature research in order to yield the desired colour change. Given the estimated electrochromic layer thickness determined through previous verification and the known lens size, the volume of electrolyte solution to be injected will be found and the necessary number of lithium ions will be determined.

The thickness of the electrolyte layer after it has been sandwiched between the two slides will be determined. From this value and the surface area of the slide, the volume of $LiClO_4$/PC gel sandwiched can be determined to aid in repeatability of results when scaling up to larger slides and possibly lenses (see Appendix C, section C.3).

Finally, the sandwiched-layers will be visually inspected for leakage of electrolyte gel after the edges have been sealed.

2.4.6 Design Specifications

The team knew the electrolyte member had to be ionically conductive as well as electrically insulating. It also required a high degree of optical transparency so that it does not significantly reduce the transparency of the device in its off-state. It must not leak from the device. Finally, it must not oxidize the electrochromic or ITO layers, causing degradation.

From the above analysis, it became clear to the team that LiClO4/PC PMMA gel represented the best fit to the above specifications, while still meeting our customer requirements and design specifications. Section G.2 of Appendix G outlines a procedure for the synthesis of this material.

2.4.7 Verification and Chances of Success

Significant literature research support the successful use of $LiClO_4$/PC as the electrolyte layer in electrochromic devices [**7**,**6**,**4**]. In particular $LiClO_4$ has been proved to work in conjunction with the other materials which the team finds promising, namely ITO, WO_3, and Prussian blue.

Given the vast amount of literature supporting the synthesis and efficacy of this material, the team is fairly confident in its ability to successfully synthesize this material. It is also reassuring that even a liquid $LiClO_4$/PC solution has been proven successful in many electrochromic devices. Therefore, even if the team cannot get the solution to gel, this should not cause the design to fail. Since leakage could pose an issue in this case and no member of the team has ever performed a similar synthesis, the confidence level is approximately 80%.

2.5 Integration

The integration phase of this project is the last step. It starts with the completion of the EC device. Once the layers are prepared and deposited, they need to be assembled. First the two EC layers and their respective substrates will be sandwiched together, with a piece of tape on their perimeter to separate them. The gap will then be filled with the electrolyte gel. Upon the successful completion of testing the device, it will be integrated into frames. This process will first entail the sealing of the perimeter of the device to ensure that the gel layer does not leak. The semi-assembled device will then be fitted into the frames, and held into place with sealant. Once rigid, a power supply and switch will be affixed to the frame.

2.5.1 Customer Requirements

The integration of the design has changed very little from our original plan to fulfill the customer requirements. The design consisted of the EC device being integrated into a frame, with a battery and switching mechanism present. The requirements for the integration design is that it must be physically wearable, safe to wear, have a dimming switch, and look aesthetically pleasing.

2.5.2 Project Plan and Milestones

As the team moved into the project plan and milestones phase, the team had to decide on quantitative values for our requirements. After doing a bit of research, our team decided that the device should not weigh more than 150g, have dimensions larger that 20cmX16cmX5cm, and meet all CSA safety standards. To assess the aesthetics of the integrated device, a survey is to be given out to subjects who wear glasses. This survey will hopefully give us the feedback the team will need on whether or not the device is something that an individual would wear.

2.5.3 Design Flow and Verification and Other Considerations

The overall integration plan was unchanged in the design flow and functional specification stages. However, considerable thought was put into the device during the development of the verification and test plans. The integrated design was incrementally changed and fortified to stand up to the tests that it would go through. First, the electrical set-up of the device must be verified and tested. To do this probes must be attached to the EC device and a bias must be put across it. Appendix H shows a simulation that has been done to ensure that applying a bias to only the outer edge of the device will ensure that the entire device switches. After evaluating the COMSOL model, it was determined that the parameters the team chose for our design are sufficient and the integrated design would not be changed.

During these stages, it was also determined that the integrated device would need to have a good seal between the two sandwiched substrates. This adaptation to the design was made to ensure that the device is safe for the wearer.
The integration for the design was unchanged in the design specifications and verification of data sections. The verification of the COMSOL EC model increased the team's confidence that the integration method of having the electrical contacts on the perimeter of the lens was justified.

This is mostly due to this being the final stage of the project. A majority of our resources are currently being allocated to getting the EC layers to change transparencies. With this being said, our confidence that this stage will be completed is highly dependent on the completion to the preceding steps. By evaluating how far along the team is in the design process, the team only has a 70% confidence that there will be time to complete the integration process.

3.0 Purchasing Considerations

The two options that were available to us for purchasing chemicals and equipment were either through the University of Waterloo or directly through the supplier.

When purchasing through the university there is usually a longer wait time, higher possibility of our orders getting misplaced (from previous experiences), and tighter guidelines. At the same time, there is a safe location to deliver chemicals to, such as ChemStores or labs of professors and consultants. Whereas when the team order on our own, it is harder to pass clearances and shipment acceptances when not affiliated with an institution, however, the team would most likely be able to receive our orders quicker with less paper work and procedures along the way. Purchasing on our own would require our own funds to be used, and then reimbursed later by the school, as per our budget proposal and receipts and forms as proof.

In the end the team decided to purchase through the school, as this was a much safer procedure and was better tracked on record, and there would not be as much hassle over delivery and storage, especially with some of our materials possibly coming in during the holiday break between terms.

Once the team decided the exact materials that the team were buying, the team compared different chemical companies to order from. Prices were compared on an item by item basis, and the team tried to stick with one company to make ordering simpler. The team also compared percent quality of the material, and overall Alpha was our top choice over Sigma Aldrich.

4.0 Current Status

We hope to begin synthesis by the start of January, provided that all the necessary materials have arrived. Materials are to arrive during the exam period. Since the two design methods will run independent of each other, the possible late delivery of some chemicals for one design route will not hinder the start of the other.

Realistically, the team believes that it is under very tight deadlines, having only two months to create a fully integrated and functioning device. A lot of our design challenges are likely to come into play, and the contingency planning shown in our design flow diagram will be put to the test.

Taking this into consideration, the group hopes to spend 6 to 12 hours per week in the lab, per team member, during the first three weeks of the winter 2010 term. Knowing that there will be a very heavy work load during February and later, the team will devote as many hours as possible towards synthesis of the device early on. The team set our milestones accordingly to this at the beginning of our design process, and are adhering to them to date. Shown below is the group's current progress with its milestones.

Table 1: Milestone Completion Status

Milestone	Work Begins	Milestone Date	Completion Status
Order common experimental materials.	October 1, 2009	October 5, 2009	Complete
Research complete.	September 28, 2009	November 15, 2009	Complete
Order materials for selected fabrication method.	November 20, 2009	December 15, 2009	Semi-Complete
Synthesize electrolyte solution.	January 4, 2010	January 20, 2010	-
Synthesize two different electrochromic solutions.	January 4, 2010	January 20, 2010	-
Deposit electrochomic solution on electrode.	January 15, 2010	January 30, 2009	-
Commit to a single electrochromic material.	January 10, 2009	January 30, 2009	-
Sandwich electrodes with electrolyte in between.	January 25, 2010	February 1, 2010	-
Electrochromic glass prototype built and operational on bench.	February 1, 2010	February 15, 2010	-

Verification of key requirements.	February 12, 2010	February 18, 2010	-
Optimization.	February 15 , 2010	February 20, 2010	-
Integration.	February 15, 2010	March 5, 2010	-
Presentation material developed.	March 1, 2010	March 13, 2010	-
Design symposium presentation.	March 15, 2010	March 19, 2010	-
Final report due.	March 1, 2010	March 12, 2010	-

We have also included a probability matrix, on our confidence in each design step functioning, as defined by design specifications.

Table 2: Confidence Levels

Aspect of Design	Confidence of Successful Functioning
Substrate	95%
Transparent Conductor	95%
Electrochromic	85%
Electroylte	80%
Integration	70%
Confidence for Benchtop Device	61%
Confidence for Complete Device	**43%**

All confidence levels were based on existence of repeatability of previous methods, as found in literature, and then decreased to create a worst case scenario. Each confidence defines the chances of successful functioning of the device component the very first time; this is not to say that this is the probability of each component functioning after troubleshooting (which would be much higher). To obtain a total confidence of the device functioning the first time through, as of our current design status, the team treated each step as a mutually exclusive event and multiplied all of them together.

The substrate and transparent conductor have been proven to work thousands of times over, independently and in conjunction with each other. However, because it’s the first time our group has attempted these procedures, the team predict a slightly lower chance of success. The procedure for a successful electrochromic deposition has been well outlined and repeated several times in our previously referenced literature, but provides greater risk than the first two layers. Likewise the synthesis of the electrolyte is well documented, and the procedure is easy to follow step by step. The short time frame significantly decreases the chances of reaching integration, and reduces the overall chance of success. However, if the team is able to meet their milestones on time, the full design will be realized successfully. The benchtop design also has a high chance of completion given our current confidence levels.

References

[1] A. Pennisi, "Preliminary test of a large electrochromic window," *Electrochim. Acta*, vol. 44, pp. 3237-4300, 1999.

[2] Unidym. (2009, Dec.) Carbon for Electronics. [Online]. www.unidym.com/files/unidym_03_final10.pdf

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[4] R. J. M. a. D. R. R. P.M.S. Monk, *Electrochromism and Electrochromic Devices*, 1st ed. New York, United States of America: Cambridge University Press, 2007.

[5] C. M. e. al, "Flexible electrochromic device based on poly(3,4-(2,2-dimethylpropylenedioxy)thiophene)," *Electrochimica Acta*, vol. 54, p. 598–605, 2008.

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[7] L. Su, "All solid-state electrochromic device with PMMA gel electrolyte," *Materials Chemistry and Physics*, vol. 52, pp. 180-183, 1998.

[8] R. Rauh, "Elecrtonic Windows: An Overview," *Electrochimica Acta*, pp. 3165-3176, 1999.

Appendix A - Customer Requirements

Introduction

A customer for this design is defined as an individual who is seeking user-controllable opacity change in their eye wear. This encompasses users who are looking for an opacity change from clear to darkened (i.e. in prescription glasses) as well as users who are looking to vary the intensity of darkness of their eye wear (i.e. varying intensity based on how cloudy or sunny it is on a particular day). A customer is also someone who is on the cutting edge of style and technology, who is looking for the newest gadget to wow their colleagues. A customer is frustrated with their current Transitions® eye wear as they hate walking into a dark building from a sunny day and not being about to see in front of them.

The following sections 1 through 7 represent the customer requirements and are ranked from highest priority level (1) to lowest (7). The minimum performance standard requires that all the requirements laid out in priority levels 1 through 3 are met by the design.

A.1 Primary Requirements:

A.1.1 Wearable Prototype

The customer requires a wearable pair of sunglasses for which he or she can manually change the opacity of the two lenses, as seen in Figure A.1 below. The two lenses will be embedded in a supporting frame. Embedded in the frame will be a switch for the user to control whether the lenses are in a darkened or lightened state. The customer can visually verify all of these requirements.

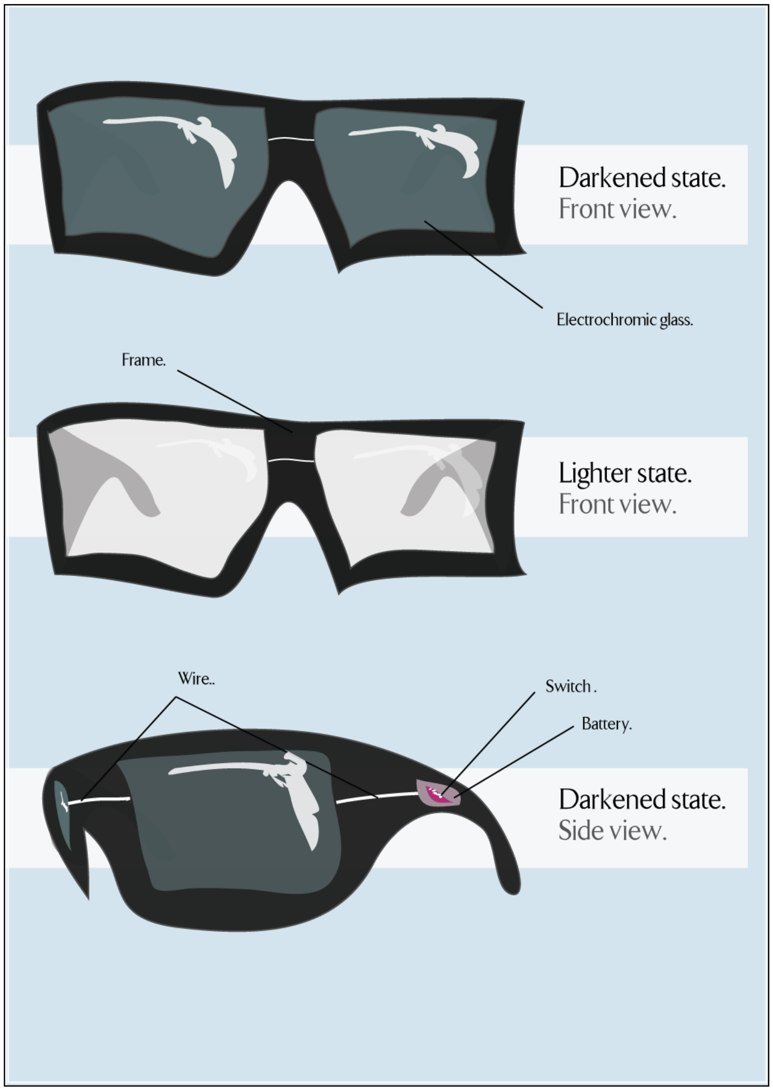


Figure A.1: The electrochromic sunglass customer features.

A.1.2 Faster Switching

The design should offer a significantly faster switching time from darkened to lightened states than traditional dimming lenses, namely photochromic lenses, often known commercially as Transitions®. The switching times from darkened to lightened states for Transitions® lenses, current electrochromic lenses, and the minimum tolerable switching time for this design are shown in Table A.1 below.

Table A.1: Switching times of various technologies.

Technology	Switching Time (Dark-to-Light)
Photochromic (Transitions®)	900 s [A1-1]

Current Electrochromic Technology	2s [A1-2]
Design Project: EC eyewear	30s

The customer will determine if the design meets this requirement by timing the switching time to go from the lenses darkened state to a lightened state.

A.1.3 Health and Safety

The prototype must demonstrate that the design will be safe to be worn by consumers. There should not be any exposed electrical wiring nor any sharp glass or plastic edges that could harm the wearer. Any chemicals used in achieving the transparency change must be sealed within the sunglasses in such a matter that they cannot leak out and harm the wearer. In the event of the frames or lenses breaking, these chemicals must be benign enough that they do not pose an immediate serious health risk to the wearer or the public.

The customer will verify that the design has met these requirements by visually examining the prototype for exposed wiring, sharp edges, and leakage. The Material Safety Data Sheets for any chemicals employed in the design to determine if they pose a risk to the public in the event that they become exposed.

A.2 Secondary Requirements:

A.2.1 Distinct States of Transparency

A visually perceptible difference will be expected from the sunglasses between its lightened and darkened state. The customer expects a 35% minimum difference in visible light transmission between the darkened and lightened states. This can be tested by the customer using Ultraviolet-Visible (UV-Vis) spectroscopy to analyze and compare the transmission of light in the two states.

A.2.2 Limit Ultraviolet Ray Exposure

The design should limit the eyes' exposure to ultraviolet (UV) rays. In both the light and dark states, the glasses should filter out at least 90% of UVA and UVB light incident on the lenses.

This requirement will be confirmed using a UV-Vis spectrophotometer with the incident light in the UVA through UVB range of 280 to 400nm.

A.3 Tertiary Requirements:

A.3.1 Dimming Switch

A dimming slide switch which would allow for incremental darkening or lightening of the lenses should be a component of the design, as seen in Figure 1. The switch should slide back and forth on a portion of the frame near the ear hook or could alternately be implemented as a spinning dial. Switching times between each stage should conform to the *Faster Switching* requirement outlined previously. The customer will also verify this requirement by checking if the change in opacity between each increment is visible to the naked eye and noticeable to the wearer. This requirement will be verified by the customer through marketing research which will ask potential consumers whether or not they can perceive the opacity change between incremental switches.

A.3.2 Aesthetics

The integrated switchable lenses and frames must not look larger, or bulkier then current publicly accepted eye wear, nor should it weigh any more or feel awkward on the customer's face. Having a product that does not meet this requirement will affect the customer demand in a negative way as customers will opt for more a more aesthetically pleasing solution to their problem. This requirement will be verified by the customer through marketing research groups where this product will be put side by side with the competition and evaluated by randomly selected potential consumers.

Appendix A - References

[A.1] Transitions®, Transitions® VI Advanced Performance for Healthy Sight, 2007, http://en-ca.transitions.com/Transitions_MCMS/flash/splashflash/flashSplash.html

[A.2] Chao Ma, Minoru Taya, "Smart Sunglasses and Goggles Based on Electrochromic Polymers," Center of Intelligent Materials and Systems, University of Washington, Available at: http://uwnews.washington.edu/ni/relatedcontent/2007/March/rc_parentID31522_thisID31528.pdf.

Appendix B - Project Plan and Milestones

In this appendix, the design project's milestones are listed along with detailed descriptions and deadlines. Challenges that the team expects are mentioned along with ways to mitigate these issues. While the members of the team has a similar educational background, certain individuals have displayed a particular preference or aptitude for this project's tasks. The role of each member in the team is discussed in this section. Finally, the resources that the team requires including lab space, software, and the cost of materials and equipment are documented in this appendix.

B.1 Milestones:

Table B.1: Milestone timeline

Milestone	Description	Work Begins	Milestone Date
Order common experimental materials.	Chemicals and equipment which are used regardless of the chosen fabrication technique will be purchased. ITO on a substrate will be on item purchased as it will be needed for all methods being evaluated.	October 1, 2009	October 5, 2009
Research complete.	Perform research on device fabrication, determining costs, fabrication times, characterization techniques, and performance expectations. Because there are several different approaches to the experimental design of electrochromic solid-state devices, it is important to choose a method that reduces the project's cost and offers the best combination of ease of fabrication, performance and aesthetics. This will be completed through a decision matrix, evaluating all aspects of each method.	September 28, 2009	November 15, 2009

Order materials for selected fabrication method.	After a fabrication method has been settled upon, the chemicals and equipment needed to synthesize the different layers of the electro-chromic glass will be purchased.	November 20, 2009	December 15, 2009
Synthesize electrolyte solution.	Use the chemicals purchased, the labs scheduled, and the methods researched to synthesize an electrolyte solution for electrochromism. Nature of solution depends on electrochromic method chosen.	January 4, 2010	January 20, 2010
Synthesize two different electrochromic solutions.	Use the chemicals purchased, the labs scheduled, and the methods researched to synthesize an electrochromic solution for electrochromism. Nature of solution depends on electrochromic method chosen.	January 4, 2010	January 20, 2010
Deposit electrochomic solution on electrode.	Deposit the electrochromic solution on the substrate covered with an ITO electrode layer. Nature of the deposition method depends on the solution chosen. This will result in a thin film layer.	January 15, 2010	January 30, 2009
Commit to a single electrochromic material.	The parallel development of the two electrochromic materials will result in one being superior to the other. Its progress will be continued while the other technique will be ended.	January 10, 2009	January 30, 2009
Sandwich electrodes with electrolyte in between.	The electrolyte solution will be injected between the two sets of films outlines in the last two milestones. This will result in an electrochromic device.	January 25, 2010	February 1, 2010
Electrochromic glass prototype built and operational on bench.	At this point, the device will operate as expected when connected to a power	February 1, 2010	February 15, 2010

	source. That is, its opacity will switch according to the voltage applied to the glass' electrodes.		
Verification of key requirements.	Requirements for safety, UV transmission, visible light transmission, and degree of distinction between the on and off states of the device will be verified at this point.	February 12, 2010	February 18, 2010
Optimization.	Depending on the difficulty of synthesizing the device layers, optimization may include synthesizing different layers to improve performance or to change the colours of the glasses to improve aesthetics.	February 15 , 2010	February 20, 2010
Integration.	This step involves connecting the fabricated electro-chromic glass to a switch. This switch will need to be embedded in an eye wear frame which will require machine cutting. The switch will need to be able to control the voltage and current which affects the electro-chromic device.	February 15, 2010	March 5, 2010
Presentation material developed.	A poster and video will need to be developed to showcase the invention. An oral presentation will need to be prepared	March 1, 2010	March 13, 2010
Design symposium presentation.	This presentation is a requirement for the nanotechnology design project course.	March 15, 2010	March 19, 2010
Final report due.	This report is a requirement for the nanotechnology design project course.	March 1, 2010	March 12, 2010

B.2 Design Challenges

B.2.1 Chemicals Take Too Long to Ship

Without experience in ordering the chemicals required for this project, it is difficult to estimate how long a supplier will take to deliver their products. If a supplier's delivery estimate would not be compatible with our milestone dates, a different chemical and fabrication technique would need to be selected. To mitigate this challenge, the team will be ordering chemicals as soon as the team know the team need them.

B.2.2 Synthesizing Polymers and Inorganic Chemicals

The team expects that fabricating polymers will require a great deal of trial and error in order to reproduce the molecular weight requirements presented in reference papers. To mitigate this challenge, the team will work in parallel on the various layers to maximize productivity. Additionally, a generous amount of time has been allotted for the synthesizing of the materials. Finally, additional time has been allotted over the exam and holiday period for the emergency case where the fabrication method must be switched to an easier method (although at a cost of performance) because the polymers cannot be synthesized.

B.2.3 Device Does Not Switch

There is a possibility that the fabricated device does not perform its intended function of changing transparencies. To mitigate this challenge, it will be necessary to investigate the deficiencies in the materials synthesized and used in the device. This would involve characterization using microscopy and likely further scientific research into how to improve our chemical processes.

B.2.4 Dangerous Chemicals

The chemicals used in the fabrication of the different layers of the device can be explosive, toxic, and highly reactive. To mitigate this challenge, additional supervision will likely be required when using these substances. The team must ensure that a supervisor is present by arranging an appointment before the date of the lab work.

B.2.5 Cutting the Glass

In order to fit within a wearable frame, the shape and size of the glass will need to be adjusted. Cutting the device may impair its functioning and force the group to fabricate the glass using the appropriate initial size. This would slow the team's fabrication down because two lenses will take twice as long, in comparison to using a single sheet of glass and cutting two lenses from that. However, while the time to fabricate will increase, it should not delay reaching any milestones; it would simply inconvenience the team. To mitigate this challenge, facilities that frequently cut optical glass are being contacted to assist.

B.3 Areas of Responsibility

All team members will be highly involved in the synthesizing steps because of their shared backgrounds in polymer science. One of Mike's roles in the team is to secure lab space because of his great familiarity with the University of Waterloo's technology. For the synthesizing, integration, validation, and optimization steps, Mike will be a leader in contacting professors and booking equipment. Mitch and Budd will play important roles in integrating the electro-chromic glass with the wearable frame. Mitch will be a leader in the design of the presentation poster and video because of his expertise with multimedia. Goran and Anne will be vital in characterizing the materials that are synthesized as well as validating the requirements. For other milestones, all group members will be expected to contribute an approximately equal share of their time and effort.

B.4 Requirements

B.4.1 Laboratory Space and Equipment

B.4.1.1 Lab Space

- **General chemistry lab space** complete with benchtops and fumehood space. This is proposed to be in C2-275, or 168 based on the permission of Rob Donkers and Jake Fisher.
- **AFM, SEM, and XRD space** for surface topography characterization based on the permission of Rossi Ivanova, Randy Fagan in DC metrology suite.

- **Electrical lab** space for analysis on low voltage power supplies (<10V) in E2 labs. Permission to be asked from Rasoul Keshavarzi-Valdani.
- **Engineering Machine Shop.** Required training and permission to be asked at the facility.

B.4.2 Equipment

B.4.2.1 In General chemistry lab space

- UV-Vis spectrophotometer (C2-168) - determining optical transmittance curves
- Sonicating bath (C2-275) - solubilization and creation of various chemicals
- Sonic dismembrator with titanium horn (C2-275) - creation of nanoparticles
- Potentiostat (C2-275) - deposition of electrochromic polymers and inorganics
- Optical microscope (C2-275 and DWE-3509) - general use
- Spin coater (Available in Ting Tsui's lab, the fourth year nano lab space, DC undergraduate clean room) - for deposition of polymeric layers
- Finally, miscellaneous chemistry equipment (pipets, glassware, etc.) purchasable from ChemStores or borrowed from Chemistry labs in C2.

B.4.2.2 AFM, SEM, and XRD space (DC Metrology)

- Ellipsometer - *for measuring film thickness*
- Profilometer - *for measuring film thickness*
- Atomic Force Microscope - *determining surface topology*
- Scanning Electron Microscope - *determining surface topology and composition by EDX*
- X-ray diffractometer - *determining the presence crystal structure of the layers*

B.4.2.3 Electrical labs

- DC power supply (ECE labs in E2)
- Alternatively a Keithley Semiconductor Analyzer in DC3702 could be used for more detailed IV characteristic curves

B.4.2.4 Engineering Machine Shop

- General tools for production of integrated frame and lenses (clamps, drills, wires, solder, switches, and circuit components)

B.5 Software Needs

The team will make use of Microsoft Word, Excel and Powerpoint in order to prepare reports and presentations which are required by the course. Adobe Illustrator will be used to prepare poster graphics. COMSOL may be used to model ion transfer in the electro-chromic layers in order to explain the phenomenon and to simulate the effect various chemical changes could have on the device's performance.

B.6 Budget

B.6.1 Standard Costs

- Lithium perchlorate gel solution: $100
- ITO on glass substrates x100 slides: $200
- ITO on plastic substrate: $100
- Frames: $25
- Electrical components: $25

B.6.2 Polymer+Gold nanoparticle approach:

- PEDOT-PSS: $175 for 25 grams
- Gold nanoparticle materials: ~$300 *This amount is not exact. May be more or less.

B.6.3 Layer-by-layer approach, NiCoCd nanoparticles:

• Ni(NO3)2*6H20	$26/100g
• Co(NO3)2*6H20	$50/100g
• Cd(NO3)2*4H20	$50/100g
• KOH	Borrow
• Ammonium Hydroxide	$45/500mL
• PDDA med. MW	$35/1L 20% weight

B.6.4 LBL approach, Ruthenium Purple:

- $FeCl_3$: $35/100g
- KCl: $50/500g
- KCN: $50/100g

- $K(RuCl_5)$: \$250/5g *This compound is not exact. May be more expensive.
- 2-acrylamido-2-methylpropanesulfonic acid (AMPS) \$12/50g
- Polyaniline: \$60/5g

B.6.5 Tungsten oxide approach:

- WO_3:

B.6.6 Organic-metallic hybrid approach:

- Polymer:
- Metal:
- Solvent:

Table B.2: Estimated Dollar Comparison of Different Methods

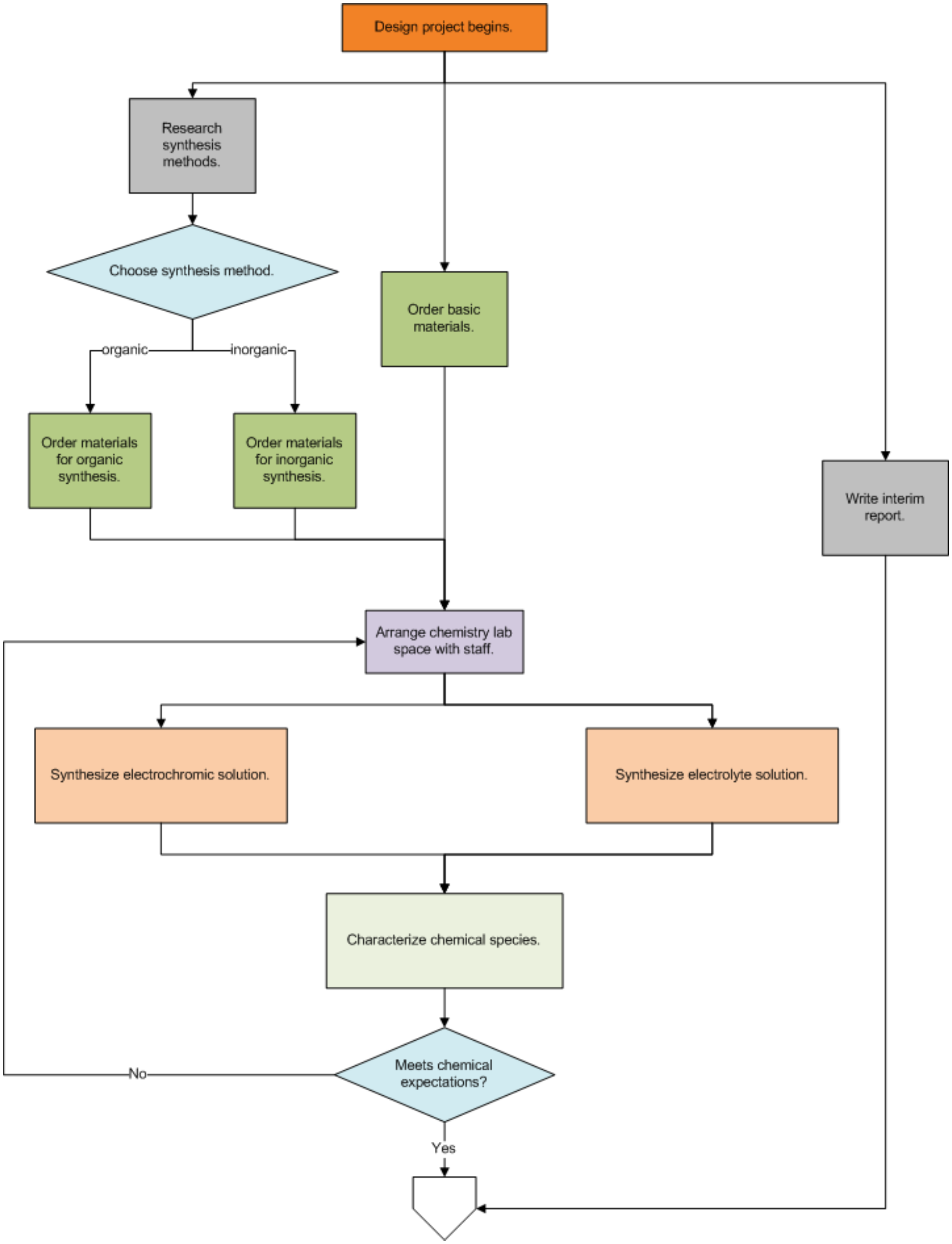
Method	Estimated Budget	Total (Est * 150%)
Polymer+gold nanoparticle approach	$925	$1387
Layer-by-layer approach NiCoCd nanoparticles	$656	$984
Lbl approach Ruthenium Purple	$907	$1360
Tungsten Oxide	$	$
Organic-metallic hybrid	$	$

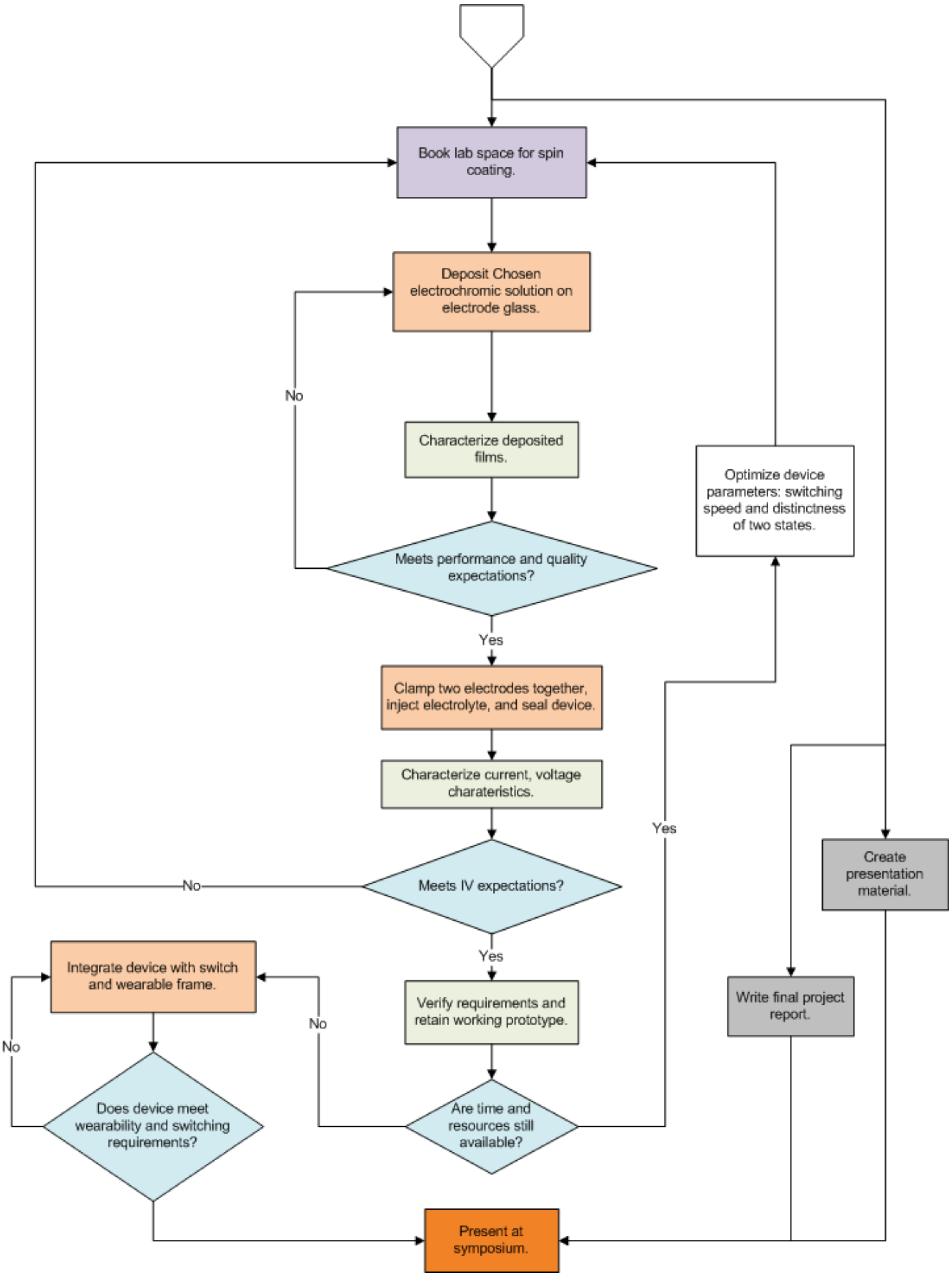
Appendix B - References

[B.1] Transitions®, Transitions® VI Advanced Performance for Healthy Sight, 2007, http://en-ca.transitions.com/Transitions_MCMS/flash/splashflash/flashSplash.html

[B.2] Chao Ma, Minoru Taya, “Smart Sunglasses and Goggles Based on Electrochromic Polymers,” Center of Intelligent Materials and Systems, University of Washington, Available at: http://uwnews.washington.edu/ni/relatedcontent/2007/March/rc_parentID31522_thisID31528.pdf.

Appendix C – Design Flow Diagram




Book lab space for spin coating.
Deposit Chosen electrochromic solution on electrode glass.
No
Characterize deposited films.
Optimize device parameters: switching speed and distinctness of two states.
Meets performance and quality expectations?
Yes
Clamp two electrodes together, inject electrolyte, and seal device.
Characterize current, voltage charateristics.
Yes
Create presentation material.
No
Meets IV expectations?
Yes
Integrate device with switch and wearable frame.
Verify requirements and retain working prototype.
No
Write final project report.
No
Does device meet wearability and switching requirements?
Are time and resources still available?
Present at symposium.

Appendix D – Functional Specifications

This device is designed to be a wearable pair of sunglasses which can have the opacity of its lenses controlled by the user using a switch. The technical parameters of this device are listed in Table below.

Table D.1: Device Specification Sheet

Priority	Specification	Description	Performance Min.	Performance Max.	Units
1	Distinct transparent states	Transmittance difference between on and off state[1]	30	60	%
2	Wearable	Weight	70	150	g
3	Wearable	Dimensions[2]	17*x*14*x*3	20*x*16*x*5	(L*x*W*x*H) cm
4	Switching Time	Time delay between on and off states	0	30	s
5	Limits UV exposure	UV transmittance	0	10	%
6	Aesthetically pleasing	Population sample average rating	40	100	%
7	Fine transparency control	Granularity in change of transmittance	2	20	%
8	Product safety	Are there sharp edges?	No	-	Yes/No
9	Product safety	Do chemicals leak from device?	No	-	Yes/No
10	Product safety	Are electrical sources and wires insulated?	Yes	-	Yes/No

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[1] See an example of an ideal voltage-transmittance relationship under Specification Notes

[2] See a diagram of the device's dimensions under Specification Notes

D.1 Specification Notes

D.1.1 Distinct Transparency States

With the users' manual control via a switch, they should be able to change the transparency from a low transparency state to a high transparency state. These will be defined as ON and OFF states, respectively. Transparency, or transmittance, is inversely related to absorbance; high absorbance means the glasses will have low transparency. These relations are shown in Table D.2 below.

Table D.2: Definition of ON and OFF states

	Transparency	**Absorbance**	**Qualitative**
ON	Low	High	Dark
OFF	High	Low	Clear

In terms of specific numbers for ON and OFF transparency, each lens darkening technique has its own limitations therefore the group feels the *range* of transparency is more important. For example, an OFF state of 70% transmittance and an ON state of 30% transmittance would be deemed acceptable. This corresponds to a 40% difference in transparency.

Our minimum specification goal is 30% transparency difference in ON/OFF states and our maximum would be as high as possible, or 60%.

Figure D.1 below demonstrates a changing voltage in the lens, and the corresponding absorbance. This is a sample curve from an electrochromic technology similar to our design specification.

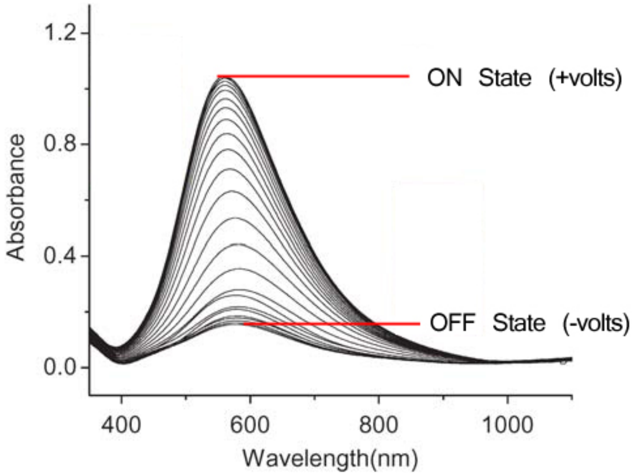


Figure D.1: Idealized wavelength-absorbance curve for varying applied voltages. Based on a bench-top electrochromic piece of glass [1].

D.1.2 Wearibility

This section covers the limiting specifications in order for the glasses to be considered "wearable" by a customer. The wearability specifications are that the total weight of product (power supply inclusive) will not exceed 150g, nor will they exceed dimensions of 20 cm x 16 cm x 5 cm. These specifications are based on weights and measurements taken by the design team, using their own instruments and samples.

Figure D.2: Diagram of glasses with dimensions

In order to leave room for error, measurements were rounded up a few percents of their total weight, and upon summation, the total weight was rounded up. Glasses weighing 100g are still deemed to be light weight, and comfortable.

Table D.3: Mass breakdown of electrochromic eyewear device

Item	Mass
Battery	5g
Frame	50g
Electronics	5g
Lenses	10g
Thin Film & Sealent	5g
TOTAL	75 g

D.1.3 Switching Time

The speed which the device switches from on to off and off to on should be within an acceptable range. This range is defined by the performance of current technologies, seen in the below table. For this device, the switching time specification will be set at 30 seconds or less for switching in both directions.

The parameter will be measured with a stop watch. The start and stop queues for the measurement will be triggered when the device reaches the 'on' and 'off' transmittance defined in the above section.

Table D.4: Comparison of switching times

Technology	Switching Time (Tint-to-Bleach)
Photochromic (Transitions)	900 s [2]
Current electrochromic technology	2 s [3]
Design Project: EC eyewear	30 s

D.1.4 Limit UV exposure

Figure D.3 shows the transmittance spectrum of 3.2 mm thick polycarbonate 150nm to 2500nm. As can be seen from the figure, transmittance in the visible range should be above 90% in the visible range. It then drops off suddenly in the UV range (10-400 nm) to nearly 0%.

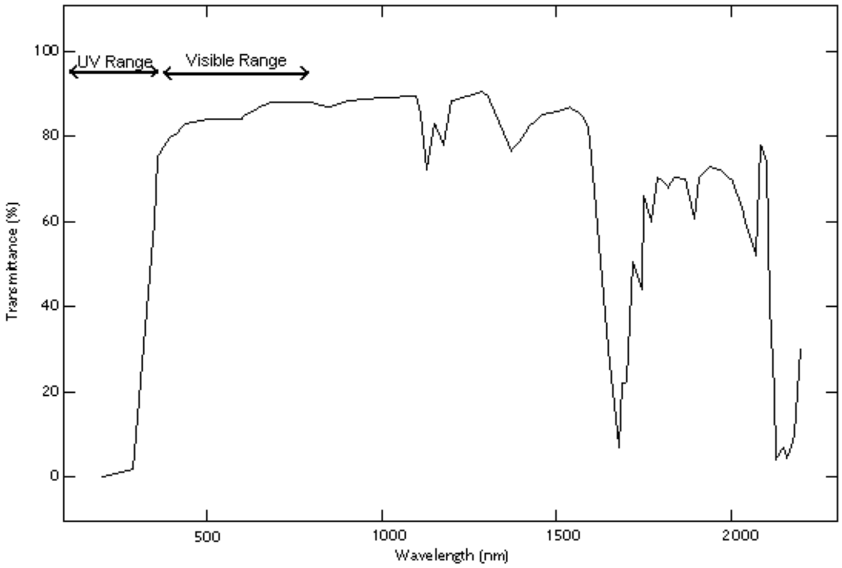


Figure D.3: Transmittance spectrum of polycarbonate [4.D].

Thus by employing a polycarbonate substrate, the design should limit the UV exposure to the eyes of the wearer to below 10% transmittance in the UV range. This means that 90% of all UVA and UVB light incident on the lenses should be reflected or absorbed.

Additionally if polycarbonate cannot be used the group will investigate further coatings and thin films that reduce UV transmittance while maintaining visible light transparency.

D.1.5 Fine Transparency Control

Instead of a simple on/off switch that lets the user change from dark/light modes, it would be more functional to have granularity in the dimming process. This may be accomplished by a dimming switch and circuit that controls the voltage (therefore current) provided to the device for a given amount of time.

It would be ideal to change the transparency in units of 2% based on this dimming switch; however it will be acceptable for this secondary goal to achieve dimmable states of 20%.

D.1.6 Aesthetics

The integrated switchable lenses and frames must not look larger, or bulkier then current publicly accepted eye wear, nor should it weigh any more or feel awkward on the customer's face. To measure the aesthetics of this device the team will gather data through a survey.

The survey will take use a random sample of the eye glass wearing population and ask them if they would wear the device for functional purposes in public. Other data will be collected through this survey, but the intent of the survey will be to gain a metric of what percent of the population will wear the device due to its aesthetic appeal.

Our specification for the aesthetics is to have 40% or more of the eye glass wearing population wanting to wear the device for functional purposes.

D.1.7 Product Safety

In order for the device to marketable, it must stand up to CSA standards. This means that the device should not leak any harmful liquids, discharge any electrical charge to the wearer, have any sharp edges capable of poking or cutting the wearer, and the MSDS for the chemicals in the device should be provided to the wearer.

In terms of specifications; when leaving the device to sit for 8 hours, no liquid should leak out. When running a ground probe over the surface of the device, there should not be any electrical shorting. When running a kimwipe over the surface of the device, then should be no snags that tear the delicate tissue. These three specifications are pass/fail, not quantitative. As for the MSDS sheet, it should be available with the device upon consumer purchasing.

Appendix D - References

[D.1] Jain, Vaibhav, and Rabindra Sahoo. "High contrast solid state electrochromic devices based on Ruthenium Purple nanocomposites fabricated by layer-by-layer assembly." Chemical Communications (2008): 3663-665.

[D.2] http://en-ca.transitions.com/Transitions_MCMS/flash/splashflash/flashSplash.html.

[D.3] Smart Sunglasses and Goggles Based on Electrochromic Polymers. Chao Ma, Minoru Taya. University of Washington.

[D.4] Fresnel Technologies, "Transmittance of polycarbonate as a function of wavelength," 2003, http://www.fresneltech.com/graphs/polycarbonate_graph.htm.

Appendix E - Verification Plan for the Paper Design

E.1 SUMMARY OF ANALYSIS AND DEVELOPMENT TECHNIQUES

1. **UV-Vis spectrometry** for verifying the percentage transmittance and UV absorbance of individual device layers as well as the final product
2. **Resistivity measurements** using a four point probe for validating the resistivity of the transparent conductive oxide layers
3. **Cyclic voltammetry** using a potentiostat for both the deposition of electrochromic and counter electrodes as well as the characterization and operation of the layers in a test cell
4. **Thin film characterization techniques** using profilometry, ellipsometery for thickness verification of the deposited layers as well as SEM and AFM for visual characterization
5. **Circuit analysis** using a variety of tools such as power supplies, function generators, and multimeters to develop and verify the control electronics
6. **Timers** with a precision in the milliseconds will be used for various uses such as the time between on and off states.

E.2 DEVICE SCHEMATIC

The electrochromic (EC) eyewear project will consist of the following layers deposited onto glass or plastic lenses. The ITO electrodes will be connected externally to the voltage source. In forward bias the Li ions will migrate to the EC layer from the gel electrolyte. If the voltage source switches polarity the Li^+ ions will be moved from the EC layer through the electrolyte to the counter electrode. Depending on the location of the Li ions, the lens will appear in different levels of opacity.

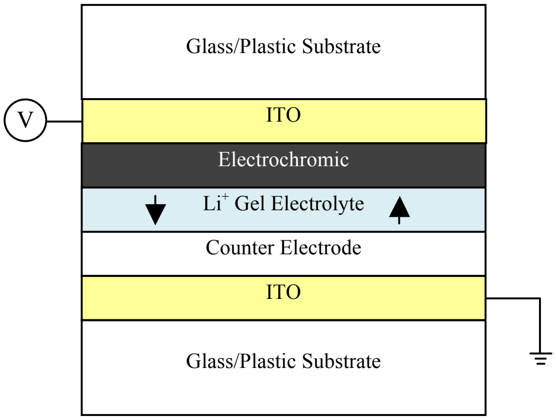


Figure E.3 - Electrochromic eyewear device

E.3 VERIFICATION STEPS

E.3.1 VERIFICATION OF SOLUTION BASED ELECTROCHROMIC ASSEMBLY

The purchased electrochromic will be preliminarily tested to determine if it does change in opacity. A layer will be deposited on a small ITO glass slide and then submerged in a beaker filled with a lithium perchlorate/propylene carbonate solution. The probes of a potentiostat will be put in the solution and a voltage applied. The electrochromic layer should exhibit a colour change.

E.3.1.1- Thickness versus opacity

The electrochromic layer should be deposited with different layer thicknesses on several ITO glass slides. The degree of opacity change for different thicknesses will then be judged visually to determine the approximate thickness needed to yield the desired opacity change (~30%). The thickness of the deposited layers will be measured by profilometer, elipsometer, or AFM.

E.3.1.2 - Switching time

Using a stop watch, the switching time from light to dark will be measured to verify it is below 30s. Although in actual design, diffusion of lithium ions will likely take longer, this step will give some indication if the switching time requirement is attainable with this electrochromic at a given thickness. If necessary, the layer thickness could be increased to speed the switching time.

E.3.2 VERIFICATION FOR ELECTROLYTE SOLUTION

An electrolyte solution made up of lithium perchlorate, propylene carbonate, and PMMA will be synthesized and tested. Verify that the solution forms a gel.

Note: PMMA and propylene carbonate are simply used to form a gel and solubilise the lithium perchlorate. It is not necessary to verify their exact chemical compositions as it is not crucial for operation, except in so far as a gel is formed.

E.3.2.1 Verify gel conductivity

Using a multimeter with the probes immersed in the solution, verify the conductivity of the gel.

E.3.2.2- Determine needed moles of lithium

The number of lithium ions needed per volume of electrochromic layer needed in order to yield the desired colour change will be determined from literature research. Given the estimated thickness found in A.1 and an estimate of the lens size, the volume of the electrochromic layer will be found and the corresponding number of lithium ions will be calculated. This will ensure that the number of lithium atoms present is in excess.

E.3.3 VERIFICATION OF SLIDE/ELECTROLYTE SANDWICH

The electrolyte will be poured onto an ITO glass slide and sandwiched with another glass slide. The slides will then be clamped together.

E.3.3.1- Verify conductivity

Before sandwiching slides, add in conductive contacts that will be hooked up to a multimeter in order to check the conductivity.

E.3.3.2 – Verify transparency

Using UV-Vis, determine if transparency in visible range is high enough that, given the estimated transparencies of the subsequent layers (from literature research) the overall transparency will be high enough in off-state to ensure distant states of opacity (distinct change in opacity from on-state to off-state).

E.3.3.3- Determine thickness of sandwiched electrolyte

Increasing volumes of electrolyte will be poured and sandwiched, to determine the volume needed to complete cover the slides. This will be determined as the minimum volume for which a minimum amount of electrolyte seeps out of edges when sandwiched. The volume to use is directly related to the surface area of the slide, and so this can be used to calculate the needed volume for any size lens.

Once the needed volume of solution is known, the known needed moles of lithium perchlorate calculated in B.2 will be used to find the concentration of lithium perchlorate that should be used in the electrolyte gel given a particle lens size (concentration = moles/volume).

E.3.3.4- Verify electrolyte can be sealed

Verify the electrolyte can be sealed by sealing by some method. Check for leakage visually over a number of days. Also, check for differences in conductivity using multimeter that could indicate minute leakages.

E.3.4 VERIFICATION OF LAYER DEPOSITIONS

There are three layer of concern –indium tin oxide, an electrochromic active layer and a counter electrode. The ITO will be purchased as a deposited layer on plastic or glass. As with any item being purchased this will have to be verified to see that it meets to specifications. The layers actually being deposited are the EC layer and the counter electrode. One layer is deposited on one piece of glass/ITO substrate and the other is deposited on a second glass/ITO substrate. The two may have different deposition methods (i.e. electrodeposition, spin coating, layer-by-layer deposition) and as such each layer must be verified separately for its thickness. Thickness is an important parameter because it determines transparency, ion conductivity, and electrical conductivity.

E.3.4.1- ITO on glass or plastic slides

There are three parameters that must be verified for the ITO thin film: transparency, sheet resistance, and thickness. The three are interrelated and the first two are more important than the thickness. As the thickness increases the conductivity increases but the transparency decreases.

Therefore the group must verify UV-Vis transparency from 300-700 nm wavelength. It should be within a range of 80-90%. This transparency relates to a sheet resistance of 10-200 Ω/sq. The sheet resistance cannot be much lower because based on electrochromic devices created in literature the conductivity will not be sufficient.

The sheet resistance will be verified using a four-point probe in standard operation. The transparency in the visible range can be measured using a UV-Vis photospectrometer. The slide is simply inserted where the traditional cuvette port exists. For film thickness there are a variety of methods the group can use such as profilometry, ellipsometry or atomic force microscopy. This last check will simply be to verify the manufacturers quoted film thickness. This does provide a good reference if the electrodes are to be deposited by the group if the team need better control over the ITO thickness.

E.3.4.2- Calculate layer thickness needed

Depending on the deposition method, use literature values and estimates determine in A.1, choose electrochromic layer thickness for correct opacity (not too dark to see, but dark enough to meet minimum opacity change between on and off-states.

The ranges of these layers need to be in the few hundred nanometers range.

For the lithium gel electrolyte it is difficult to control the thickness due to the sandwich and clamp technique that will be used.

Note: Deposition technique may be sol gel, spin coat, dip coat, electrodeposition.

E.3.4.3 Check desired thickness was obtained

Again these deposited layers will be characterized and verified by thickness measurement techniques such as profilometry, ellipsometry, or AFM.

E.3.5 VERIFICATION OF GLASS SLIDE ASSEMBLY

Figure E.2 below shows the test set-up for these verification steps. The tips of two glass slides will be taped to prevent any deposition in those areas. An electrochromic layer will then be deposited on one slide and a counter-electrode on the other. The tape will then be removed and electrical contacts will be attached to the exposed ITO. The electrolyte solution will be poured onto one slide and the two slides will be sandwiched together and clamped.

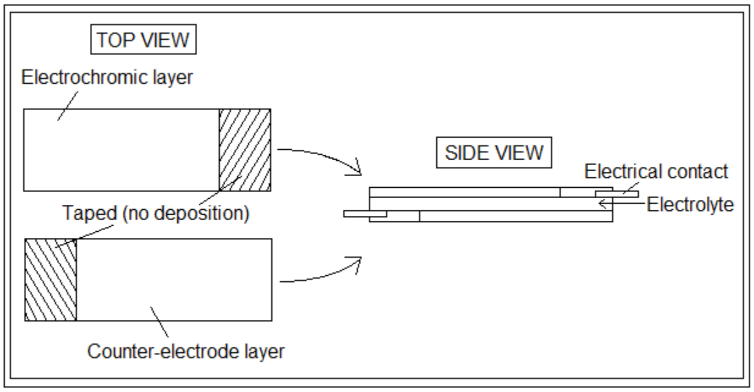


Figure E.2: Test set-up for glass slide assembly testing

E.3.5.1-Verify thickness dimension

Using callipers, the thickness of the sandwich will be measured to determine if the thickness is below the maximum thickness of 3cm

E.3.5.2- Verify electrochromic transition

A potentiostat will be attached to the electrical contacts and a small positive voltage will be applied and it will be visually determined whether or not the slide becomes more opaque. A small negative will then be applied and it will be visually determined whether or not the slide returns to its former transparency. If no change is seen, the positive and negative voltages used will be incrementally increased.

E.3.5.3- Verify minimum opacity change

Using a UV-Vis spectrometer, the opacity change between on and off-state in the visible range (~450-700nm) will be determined to verify it is over 30%. For the on-state, it is necessary that the slide assembly be attached to a potentiostat while the reading is taken. This will be facilitated by small electrical connections and long wires to the potentiostat. Applied voltage from the potentiostat can be increased until the necessary change is seen.

E.3.5.4-Verify UV light blockage

Again using the UV-Vis spectrometer, the transmittance in the UV range (below 300 nm) will be determined to verify the transmittance in the UV range is below 10%. Verify in on and off-states.

E.3.5.5-Verify switching time

Using a stop watch, determine if switching time is below 30s. Initially, the minimum applied voltage needed in D.2 to facilitate a 30% opacity change will be used. If this voltage does not cause an opacity change in 30s, it will be increased. When the required minimum voltage is known, record the corresponding current running through the device using the potentiostat.

For this verification, the time at which the on-state (maximum opacity change) is reached will simply be determined visually.

E.3.5.6- Verified fine opacity control

Using increasing voltages on the potentiostat, it will be determined if the degree of opacity can be controlled incrementally (if states between maximum transparency and maximum opacity can be obtained by incrementally increasing the applied voltage). Whether or not two states are distinct will be determined visually.

E.3.5.7- Determine power requirements

Using the potentiostat, the minimum power requirements will be determined. This is the power needed to apply a voltage which satisfies both D.2 (the minimum opacity change) and D.5 (the maximum satisfactory switching time), given the current recorded in D.5.

A battery that can provide this power and corresponding voltage and current will then be selected.

E.3.5.8- Verify use of glass lenses will conform to wearability

The battery selected in D.7 will be weighed to determine if it poses a risk to the weight specification imposed by the wearability requirement of the device (< 150g total), given the estimated weight of the frame, and the weight of the glass lenses. If the mass will be too high, polycarbonate lenses may have to be used.

E.3.6 INTEGRATION VERIFICATION

Actual lenses and frames conforming to the maximum dimensions and weight set-forth by the wearibility requirement (17 cm wide and 14 cm deep, 150g) will be selected. The lenses will be cut from ITO glass or ITO polycarbonate, depending on the results of D.8. Layer deposition will be performed. For even opacity change, the conductive electrical contact will surround the circumference of the front of each lens as seen in Figure E.3. The electrolyte will then be poured on, and the lenses sandwiched and sealed as determined previously.

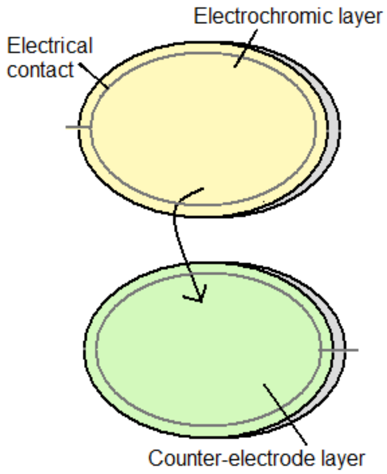


Figure E.3 - Electrical contacts for lens geometry.

E.3.6.1- Electrical set-up verification

Wire on/off switch to battery. Using a multimeter, test that the on/off switch does control the output voltage of the battery.

Wire the battery to the electrical contacts and visually test if an opacity change is seen as the switch is turned on and off.

Time permitting, a dial or slider may be used in place of a switch to provide different voltages and thus different degrees of opacity change. A multimeter will be used to test if the dial or slider does incrementally change the output of the battery. Once connected to the electrical contacts, the existence of distinct states of opacity will be tested visually.

E.3.6.2- Final device verification

Completely verify the entire device for all parameters included in the functional specifications. This includes the following tests:

1. Measure distinct transparency states via spectroscopy
2. Measure physical dimensions of the device (weight, height, length, width)
3. Time the duration of switching between modes
4. Determine amount of UV absorbance via spectroscopy
5. Test for granular opacity states using power supply and control circuit
6. Evaluate product for all safety requirements (leakage, edges, electrical insulation)

E.3.6.3- Aesthetic verification

Aesthetic verification will be tested by polling a random group of consumers and gauging their opinion on the aesthetics of the final integrated device versus other commercial lenses. Adjustments may then be made according to their suggestions, time permitting.

Table E.1: Pertinent verification steps

Priority	**Specification**	**Description**	**Performance Range**	**Pertinent Verification Steps**
1	Distinct transparent states	Transmittance difference between on and off state[1]	30 - 60%	D1, E3, F2
2	Wearable	Weight	70 – 150g	
3	Wearable	Dimensions[2]	17*x*14*x*3 -20*x*16*x*5 cm	
4	Switching Time	Time delay between on and off states	0-30s	

5	Limits UV exposure	UV transmittance	0-10%	
6	Aesthetically pleasing	Population sample average rating	40-100%	
7	Fine transparency control	Granularity in change of transmittance	2-20%	E6, F2
8	Product safety	Are there sharp edges?	No	
9	Product safety	Do chemicals leak from device?	No	
10	Product safety	Are electrical sources and wires insulated?	Yes	

A detailed plan showing how the design at the concept and structural abstraction levels will be verified to meet all the performance specifications before prototype (hardware and/or software) construction is started. The plan must show which analysis techniques or specification development techniques will be used to verify the design, reducing the number of design oversights or invalid design assumptions, before detailed design and/or prototype construction start.

Basically, the plan should show how, via some analysis or technique, that the functional specification and the design specification meet the customer requirements. It should also, via some analysis or technique, confirm that if all the pieces in the block diagram, be they those the team will design or those the team will acquire, work according to their specification, the combined function will meet the customer requirements. Basically, the design concept works and meets the customer requirements. If it does, verify the structural design abstraction works and meets the customer requirements. The earlier in the design cycle design oversights or incorrect assumptions are discovered the less painful is the correction.

Appendix F - Test Plan for the Constructed Design Prototype

Performance Specifications to be Tested

The designed device ***prototype*** will be tested on a variety of performance parameters. These parameters are summarized in Table F.1.

Table F.1: Device Specification Sheet

Specification	Performance Min.	Performance Max.	Units
1. Distinct transparent states	30	60	%
2. Weight	70	150	g
3. Dimensions	17*x*14*x*3	20*x*16*x*5	(L*x*W*x*H) cm
4. Switching Time	0	30	s
5. UV Tranmittance	0	10	%
6. Aesthetics	40	100	%
7. Transparency control	2	20	%
8. Sharp Edges	No	-	Yes/No
9. Leaks	No	-	Yes/No
10. Risk of Electric Shock	Yes	-	Yes/No

F.1 Distinct transparent state

The performance requirement for this specification is that the transmittance difference between the on and off states is between 30% and 60%. To test this specification, a spectrometer will be used to measure the transmittance of the device in the on and off states.

The spectrometer that will be used is one that not only supports the testing of typical cuvette samples, but solid samples as well. The device's lens will be placed in the spectrometer, positioned so that the path of visible light is orthogonal to its surface. A reading will be taken for both the on state and off state. The connections to power supply or potentiostat will lead into the spectrometer in order to control this on/off state. The resulting spectroscopic plots will be compared in the 400 nm to 750 nm visible light range to determine the percentage of transmittance difference.

F.2 Weight

The overall weight of the integrated device must be comparable with current eye wear on the market. The specification set for this device is set in the range of 70 grams to 150 grams.

To test the weight of the prototype, a standard analytical balance will be used. The device comprised of the electrochromic lenses, frame, power source, switching mechanism, and supporting wiring will be place on a zeroed balance. The reading will determine the weight of the prototype.

In the event that a power source and switching mechanism cannot be integrated into a stand-alone prototype, the device minus these features will be weighed in the same manner described above.

F.3 Dimensions

The prototype's dimensions are to be between 17 cm x 14 cm x 3 cm and 20 cm x 16 cm x 5 cm in length x width x height. These specifications are based on measurements taken by the design team, using their own instruments and samples.

The testing of this specification will be done using standard calipers for measuring centimeter or millimeter length scales.

F.4 Switching Time

The speed in which the device switches from on to off and off to on should be with in an acceptable range. Through research, it was found that the transition from on to off takes much more time then off to on, and will be the focus of the testing. This transition time should take less than 30 seconds.

The apparatuses that will be used for the testing of this specification are a stop watch and a spectrometer. The sample will be loaded into the spectrometer as described in section 1. A time dependent collection of data at a constant wavelength (600 nm was chosen as it is a midrange visible wavelength) will be collected. The device will be turned on using a power supply (potentiostat for development, and the integrated power supply for final product) then the spectrometer will start to collect data. The device will then be turned off, and a stop watch will start time keeping. Once the data from the spectrometer shows that the device has reached a constant 'off' transmittance, the stop watch will stop the time.

F.5 UV Transmittance

In the interest of protecting the wearer's eyes from UVA and UVB rays, a specified UV transmittance of 10% or less was set. This parameter will be tested the same way as visible light was tested; with a spectrometer.

The sample will be loaded into the spectrometer in the same manor outlined above. A spectroscopic plot will be collected at both the 'on' and 'off' states. The transmittances at the UV wavelength (230-365 nm) will be observed and recorded.

F.6 Aesthetics

The integrated switchable lenses and frames must not look larger, or bulkier then current publicly accepted eye wear, nor should it weigh any more or feel awkward on the customer's face. To measure the aesthetics of this device the team will gather data through a survey.

The survey will use a random sample of the eye glass wearing population and ask them if they would wear the device for functional purposes in public. Other feedback data will be collected through this survey, but the intent of the survey will be to gain a metric of what percent of the population will wear the device due to its aesthetic appeal.

Our specification for the aesthetics is to have 40% or more of the eye glass wearing population wanting to wear the device for functional purposes.

F.7 Transparency Control

Upon the creation of a controlled dimming mechanism, it must be tested to verify the percent transmittance change during each step of dimming. To do this, the spectrometer will once again be used. A plot will be generated before and after the controlled step in transparency. The visible light transmittance will be compared for each of the steps, and the percent change will be logged.

F.8 Sharp Edges

To meet CSA standards, the prototype must meet certain safety benchmarks. First off, the prototype must not have any sharp edges capable of poking or cutting the wearer. The test to ensure that there are no sharp edges will be done with a kimwipe and a finger. When running a kimwipe over the surface of the device, there should be no snags that tear the delicate tissue.

F.9 Leaks

Another safety standard required the device not to leak any of the electrolyte solution from it. To test this, after the lens is sealed and integrated into the frame it will be left to sit over night. If within a 12 hour period of sitting, no liquid leaks out, the prototype passes the leak test.

F.10 Risk of Electric Shock

The final safety element that will be tested is the potential for the prototype to shock the wearer. To test for areas that could shock you, a ground probe will be used. When running a ground probe over the surface of the device, there should not be any electrical shorting. If no shorting occurs, the prototype passes the safety test.

Appendix G - Design Specifications

The following appendix translates functional specifications into detailed design specifications, and identifies all key aspects of the design of electrochromic eyewear that need to be carried out and the (engineering) theory used therein.

Sections have been split up logically according to device layering. Each section first presents the high level system design specification before the low level detailed procedure and design.

The big picture of the device being designed is an a layered electrochromic cell that will be comprised of a substrate, a tungsten oxide layer, an electrolyte membrane, a layer of Prussian blue, and an organic-metallic polymer. These layers will be integrated with electrical components that.

G.1 - Tungsten Oxide Layer

The Tungsten Oxide Layer will be fabricated to lay between layers of ITO and Electrolyte membrane. The Tungsten oxide layer will also be integrated to the negative terminal of an applied voltage (with the positive terminal being attached to the Prussian Blue layer)[G.1][G.2].

There are several methods to deposit Tungsten Oxide; including vacuum evaporation, electrodeposition, and from a powder form [G.3]. The procedure outlined in this design is that of electrodepositing Tungsten Oxide using the chemical reaction below:

Na2WO4 + H2SO4 + H2O2 --> WO3 + Na2 SO4 + H2O + H2O2 (1) [G.3]

OR

W2O11 2- + 2 H+ --> 2 WO3 + 2 O2 + H2O (2) [G.2]

By applying a cathodic potential to a cleaned substrate a Tungsten Oxide film is deposited on an electrical conducting glass surface, outlined below using equation 1 (note that equation 1 represents the procedural method of choice, based on results and availability of materials):

1) Cathodic electrodeposition from an aqueous solution of Na2WO4 (0.2 M)
2) 30% hydrogen peroxide (0.13 M) and H2SO4 (to pH 1) are added.
3) Deposition is performed in a transparent electrochemical cell with a counter electrode at a constant cathode current density of about 1 mA/cm2
4) Deposition is performed at room temperature (about 23-25 deg C)
5) The Tungsten Oxide films are deposited onto a transparent current-conducting glass substrate with an ITO layer.

In past practices, uniformity was achieved by using statistical process control methods [G.2], but doesn't need to be performed in future work since synthesis methods have been optimized by previous experimenters.

Alternatively, if materials are not available, results are not sufficient, or any other road blocks occur with the outlined method above, there exists an alternative (yet equally as reputable and well tested) method for Tungsten Oxide film deposition from peroxy-tungstic acid electrolyte, as outlined below [G.3]:

1) Dissolve 1.8 g of tungsten powder in 50 mL of hydrogen peroxide (takes approximately 24 hours).
2) Decompose the excess hydrogen peroxide by adding small amount of platinum black.
3) Dilute the solution to 50 mM by adding about 150 mL of 50/50 v/v water/2-propanol (pH value of 1.92)
4) Add varying amounts of sulfuric acid to adjust the pH value of the electrolytes.
5) Electrodeposit solution using a potentiostat (provided by the NE lab). A three-electrode configuration with platinum as a counter electrode and Ag/AgC as a reference electrode was employed to deposit the films in literature [G.4]. However, ITO-coated glass will be used as the working electrode, after being cleaned with water and acetone.
6) Calcine the synthesized films in air up to 450 °C for 10 min.

Surface morphology, particle size and crystalline structure of the films can be analyzed with XRD, TEM, and SEM, so as the check the effectiveness of the Tungsten Oxide deposition procedure.

G.2 - Electrolyte membrane

The electrolyte membrane must be electrically insulating while remaining ionically conductive [G.5]. It is the source of Li^+ ions in the device. In a standard electrochemical cell the membrane would be embodied in the electrolyte bath in which the electrodes are submerged. There are a few requirements that are needed for this membrane to function properly.

1. High degree of optical transparency
2. A non-liquid to stop leaking and enhance safety of device in case of accidental breakage
3. Sufficient concentration of lithium ions for inducing color change in EC layers
4. As neutral pH as possible so that it does not oxidize EC or ITO layers

From literature and patent searches, there are many materials that fit all three requirements. Much research has been done for lithium electrolyte membranes not from electrochromic devices, but for lithium ion batteries. The most widely used component system is a polymer (PMMA, polyacetonitrile, PVB etc.) and propylene carbonate containing lithium ion salts. Again, there are many variations, but the following chemical procedure allows for a gel-type membrane which is ionically conductive. The polymer matrix serves as a way to provide solid support for the lithium ions (which diffuse easily through the macromolecules). The propylene carbonate serves as a

Some papers use a mixture of the three components and then polymerize the methacrylate monomers after mixing [G.6]. This will take too much time so instead a simple mixture will be sufficient.

1. Prepare a 20mL solution of lithium perchlorate ($LiClO_4$) in reagent grade propylene carbonate (PC). The concentration of lithium perchlorate should be within the range of 0.25M to 0.1M. Typically 1M is used.

2. Slowly add PMMA under stirring to the $LiClO_4$+PC solution. PMMA should have a molecular weight of roughly 10^6 g/mol.
3. Add amount of PMMA to achieve desired viscosity/transparency combination.
4. The final product should be a viscous, transparency gel electrolyte. Conductivity of 2mS/cm and transparency of 88%.

Another paper gives a procedure in terms of mass: 7g PMMA, 20mL PC, and 3g of LiClO4.

This electrolyte exhibited good performance of 50,000 cycles (on-off-on states) in several papers as tested against many other polymeric electrolyte layers. The electrolyte would be injected between two layers of ITO coated glass with the electrochromic and counter layer. This will then be sealed around the edges with UV-curable epoxy.

An interesting avenue of research would be to use Nafion as a hydrogen electron transport layer. Nafion is heavily studied, but modern electrochromic research has been mainly done using lithium as an ion source. It would be expected that hydrogen, a smaller molecule, would offer faster diffusion into the electrochromic layer – enhancing device performance. Perhaps the low pH of the solution using this method would be detrimental to the device.

G.3 - In-organic electro-chromic materials: Prussian blue

Metal hexacynometallates change colour electrochromically by intervalent charge-transfer [G.7]. Prussian blue (ferric ferrocyanide, or iron (III) hexacyanoferrate (II)) will be used as the electrochromic layer for the lenses.

Prussian blue is readily available commercially. Once purchased, it will be analysed by x-ray powder diffraction (XRD) to confirm its structure.

Prussian blue thin films can be deposited by several methods including sacrificial anode methods and extensive redox cycling of hexacyanoferrate (II)-containing solutions [G.7]. The group intends on performing the deposition by electrochemical deposition, which is the most widely used technique for this process.

Electrochemical deposition of Prussian blue occurs through reduction of iron (III) hexacyanoferrate (III), which is the main electron transfer process. It follows the following equilibrium [G.7]:

$$Fe^{3+} + [Fe^{III}(CN)_6]^{3-} = [Fe^{III}Fe^{III}\ (CN)_6]^0$$

For electrodeposition onto ITO electrodes, absorbance has been shown to be proportional to the charge passed [G.7]. This indicates that using this method has been shown in practice to the capability of being used in the fabrication of lenses that incrementally shift in opacity as higher switching voltages are applied, meeting design specifications.

The Prussian blue film switching between a faint, translucent yellow off-state to a much darker, translucent black-blue colour in the on-state. Prussian blue and tungsten oxide (WO_3) can be used in a single electrochromic device as their electrochromic reaction are complementary as seen below [G.7]:

$$\underset{blue}{[Fe^{III}Fe^{II}(CN)_6]^-} + e^- \longrightarrow \underset{transparent}{[Fe^{III}Fe^{II}(CN)_6]^{2-}}$$

$$\underset{transparent}{WO_3 + x(M^+ + e^-)} \longrightarrow \underset{blue}{M_xW^{VI}_{(1-x)}W^{V}_{x}O_3}$$

where M in the group’s device is lithium, as discussed above.

The layer thickness determines the transparency of the Prussian blue layer in the off-state, whereas the applied potential determines the transparency in the on-state. Literature values for the difference in on and off transparency well exceed the functional specification of 30% [G.8]. The actual layer thickness to be deposited as well as the applied potential used to switch the device will be determine as set out in Appendix E, Design Verification Plan for Paper Design.

In the group's design, these two layers are separated by a layer of electrolyte. Electrolyte and WO_3 deposition are both discussed above. The following steps outline the procedure for the electrodeposition of Prussian blue on an ITO substrate [G.8].

1. Solutions of 10mM FeCl and 10mM $K_3[Fe(CN)_6]$ were prepared.
2. Equal volumes of the two solutions were added to a 100mL beaker. A magnetic stir bar was added and they were stirred until dissolution.
3. An ITO coated lens was electrically connected to a potentiostat by clipping the alligator clip to the edge of the lens. This area was then masked to avoid deposition on and directly around the connection point, as well as around the edge of the lens as seen below in Figure G.1. This will allow electrical contact to be made to the ITO layer for the purposes of applying a switching voltage. The deposition mask will be made of an inert adhesive.

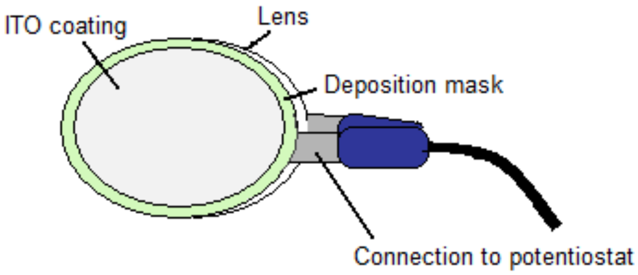
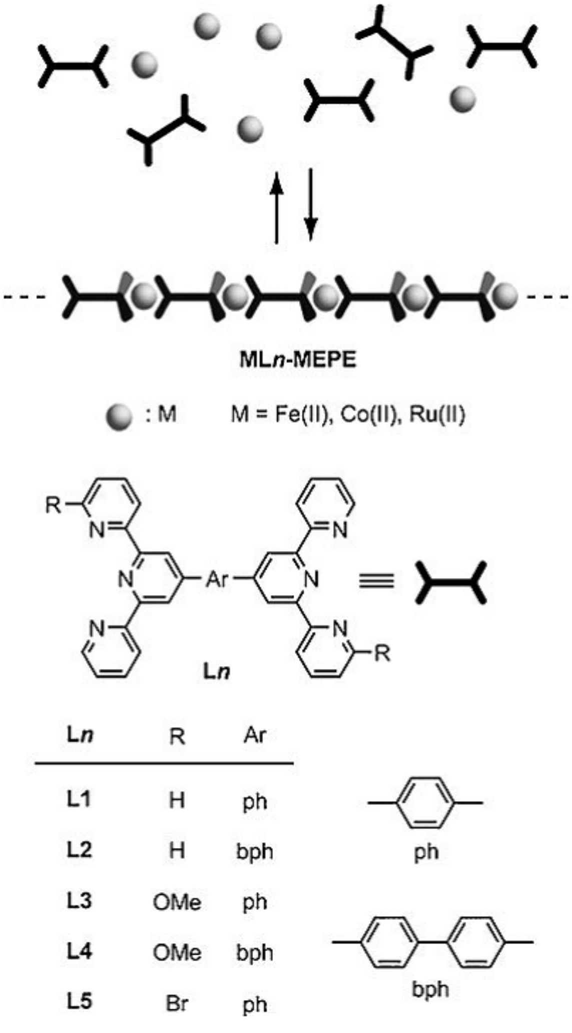


Figure G.1: Connection to potentiostat

4. The lens was suspended using a ring stand, and submerged in the solution prepared in Steps 1 and 2. The lens acts as the working electrode.
5. The counter and reference electrodes of the potentiostat were submerged in the solution.
6. The electrodeposition was performed at a fixed potential, in order to achieve a current density of 5 $\mu A/cm^2$.
7. The layer thickness was determined using ellipsometry, for the purposes of repeatability. The transparency was analyzed using UV-Vis to confirm the expected value seen in Table E.1.

G.4 - Fabricating electrochromic organic-metallic polymers:

To accomplish the objective of two different transmittance states, organic-metallic polymers can be used as the electrochromic layer of the lens. Metallo-supramolecular polyelectrolytes (MEPEs) are complexes with metal ions bound to polymer ligands in an alternating arrangement. This gives them unique electronic properties, entirely different from strictly organic polymers. For the eye-wear, which are solid-state devices, these polymers will be cast onto ITO glass by spin-coating or electrodeposition. Then, a layer of gel electrolyte will be cast onto that layer. Finally, another layer of ITO glass will be placed onto the electrolyte. Several hybrid polymers can be fabricated which offer different colours. Examples of MEPEs are shown in Figure G.2.



Ln	R	Ar
L1	H	ph
L2	H	bph
L3	OMe	ph
L4	OMe	bph
L5	Br	ph

Figure G.2: Organic–metallic hybrid polymers (MLn-MEPE) formed by complexation of bis(terpyridine)s (Ln) with metal ions such as Fe(II), Co(II), and Ru(II) ions [G.9].

The procedure for fabrication of MEPEs with Fe as the metal ion is outlined as follows, and is summarized from [G.9]:

1. Equimolar amounts of 1,4-bis(2,2':6',2"-terpyridin-4-yl)benzene (Ligand 1, L1) (available commercially from Aldrich) and $Fe(OAc)_2$ are refluxed in argon-saturated acetic acid (10 mL of solvent per 1 mg of L1) for 24 h.
2. After the reaction is completed, the solution is cooled to room temperature and filtered to remove a small amount of insoluble residues. The filtrate is placed in a Petri dish, and the solvent is evaporated slowly until dry. The brittle film is collected and dried further in vacuo overnight to obtain Fe-L1-MEPE.
3. 20 mL of a methanol (MeOH) solution (1.0 mg MEPEs/0.5mL MeOH) is cast by electrodeposition or spin-coated (500 rpm for 60 s) on ITO electrodes (20 Ohms/sq).
4. The coated film is measured using CV, absorption spectra, and AFM. UV-vis spectra of the oxidized polymer film can be monitored by means of in situ electrospectral measurements; the UV-vis spectra of the polymer film cast on an ITO electrode are measured while applying the voltage to the electrode in a quartz cuvette (1 mm-path length) with three necks.
5. The gel electrolyte, for which a fabrication procedure is explained in another section, is applied by spin coating to this coated film and the solid-state device is encapsulated with a second layer of ITO glass.

G.5 - Device Integration Specifications

After the electrochromic layers and the electrolyte solution have been mated, infrastructure for power to be transported to the electrodes needs to be integrated. In order to maintain uniform dimming of the device, a bias must be equally distributed throughout the electrode. To do this, electrical leads going into the device must wrap around the perimeter of the lens, rather than just contacting it at a single point. The following figure illustrates this.

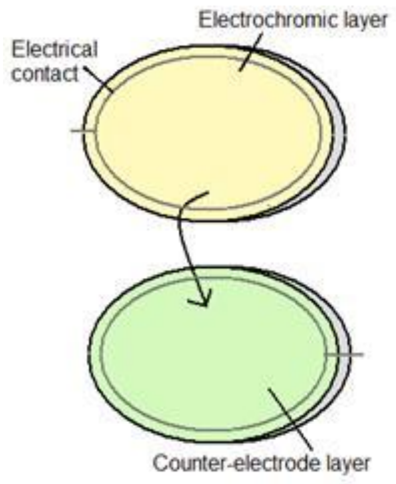


Figure G.3: Device Layer and Electrode Specifications

Once the EC device has electrical leads attached to it, it is ready to be integrated into the frame. For this to work properly, the original substrate that the ITO is deposited on must be slightly smaller than the lens openings in the frame. The process to attach the EC devices to the frame is as follows. The lenses will be places flat on a surface and the frames placed over them. To seal the gap between the lenses and frame, an epoxy will be used. This seal must be tight enough that none of the electrolyte solution can leak out.

A battery and switch will be attached once the lens is in place. To ensure that the user is not shocked by the device, the designed device must have all exposed electrical components insulated.

When choosing the frame and electrical components for the designed prototype, their weight and dimensions much comply to the functional specifications. The frames must not be larger than $20x16x5$ centimeters. The overall weight must not be more then 150 grams.

The fully integrated design must not have any sharp edges that pose any danger to the user.

G.6 - Population sample average rating

To measure the aesthetics of this device the team will gather data through a survey. The survey will use a random sample of the eye-glass wearing population and ask them if they would wear the device for functional purposes in public. Other data will be collected through this survey, but the intent of the survey will be to gain a metric of what percent of the population will wear the device due to its aesthetic appeal.

Appendix G - References

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[G.7] P.M.S. Monk et al. Electrochromism and Electrochromic Devices. New York, NY: Cambridge University Press, 2007, pp. 282-291.

[G.8] L. Su. “All solid-state electrochromic window of electrodeposited WO and 3 prussian blue film with PVC gel electrolyte.” Thin Solid Films, vol. 320, pp. 285-289, 1998.

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Appendix H - Verification Data

H.1 Model Data

The final project for the NE 336 class was for the student to model a physical system using COMSOL. One of our projects completed for this course modeled the electrochromic system described in the paper "Electrochromic Windows: An Overview" [*8*]. For simplicity, only three of the five layers where modeled; the ITO conductive layer, the electrochromic active layer, and the ion membrane. Figure H.1 illustrates the layers of the model.

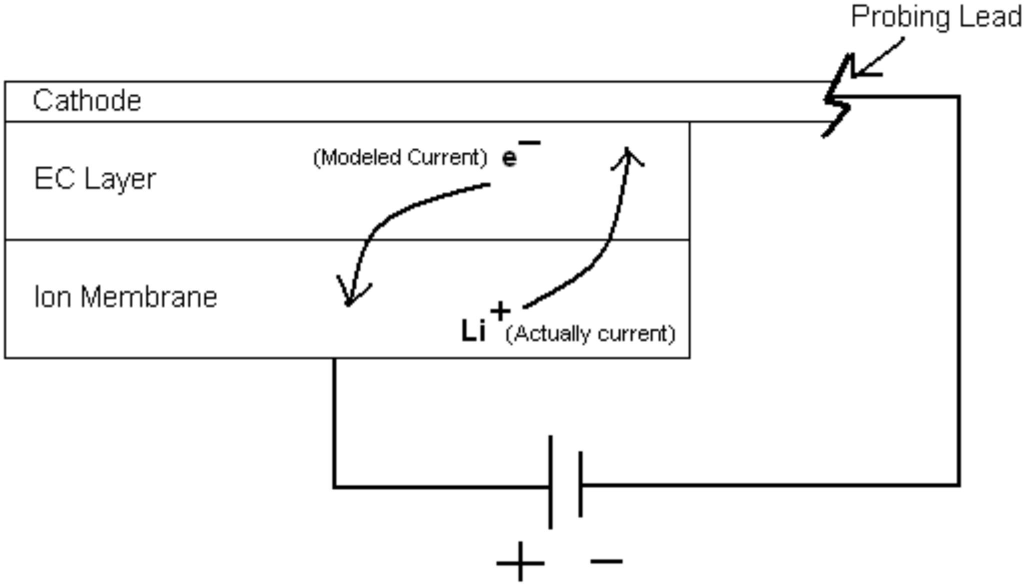

Figure H.1: Layers of COMSOL Model

Due to the limitations of the system used, the exact dimensions of the planned prototype were not used. Using said dimensions created far too many mesh elements, causing the program to stop working. Instead, the proper material parameters were used and a general, less extreme aspect ratio was used. The intent of the model was to see if when a probe was attached to the outer edge of the ITO layer, the current would be evenly distributed from the outside in. This parameter is important for the customer as the switching of the lenses should be quick and uniform. Having a lack of uniformity in the switching process may lead to one part of the lens taking much longer to change then others. Figure H.2 shows the results of the general model used for our system.

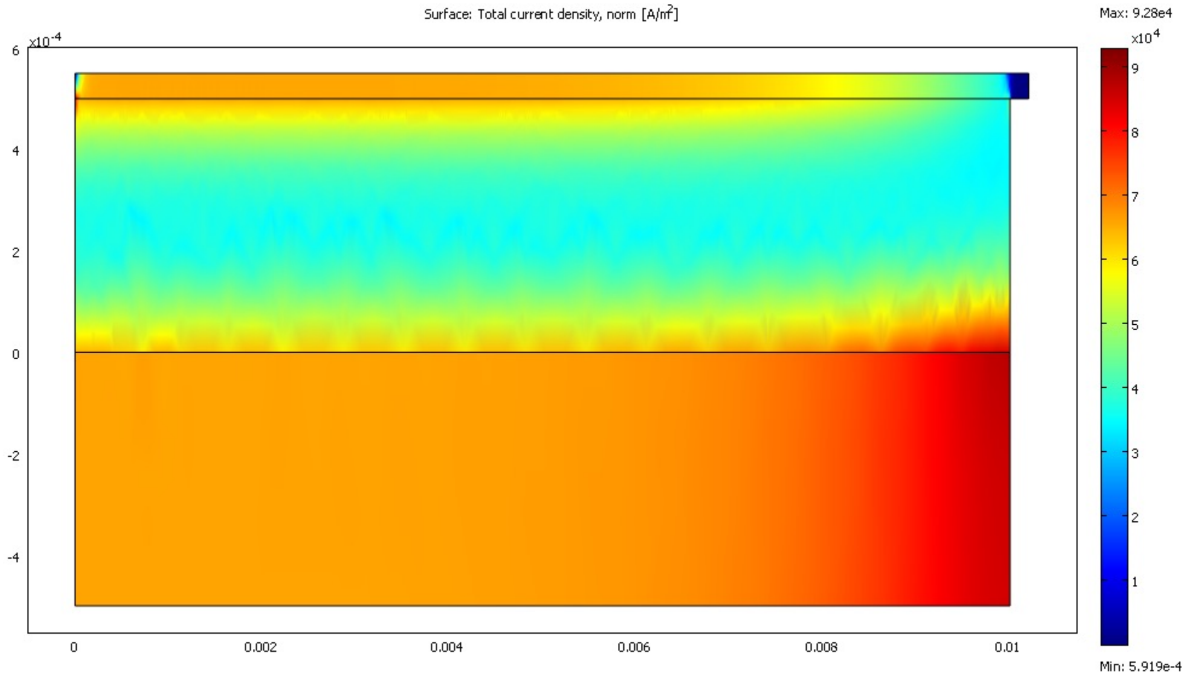

Figure H.2: COMSOL Model of Current Travelling through the EC Device.

The electrons enter the above model through the upper right area sticking out. The current then travels through the three layers and out the bottom into the counter electrode. The scale on the side of Figure H.2 indicates the current density. Evidently, the current is the most dense one the lower right side of the electrochromic layer. This information is negligible to us. the team is more interested in observing how uniform the current is over the width of the device since there is a direct correlation between the number of electrons that move and the number of EC molecules that reduce to form non-transparent complexes. Taking an average across the EC layer of Figure H.2 shows that there is a fairly uniform current density across the device. This result indicates to our group that the conductivity of the ITO chosen will uniformly spread the current across the device. To further the study, a parametric analysis was done on the system, varying the voltage applied. Figure H.3 shows the results.

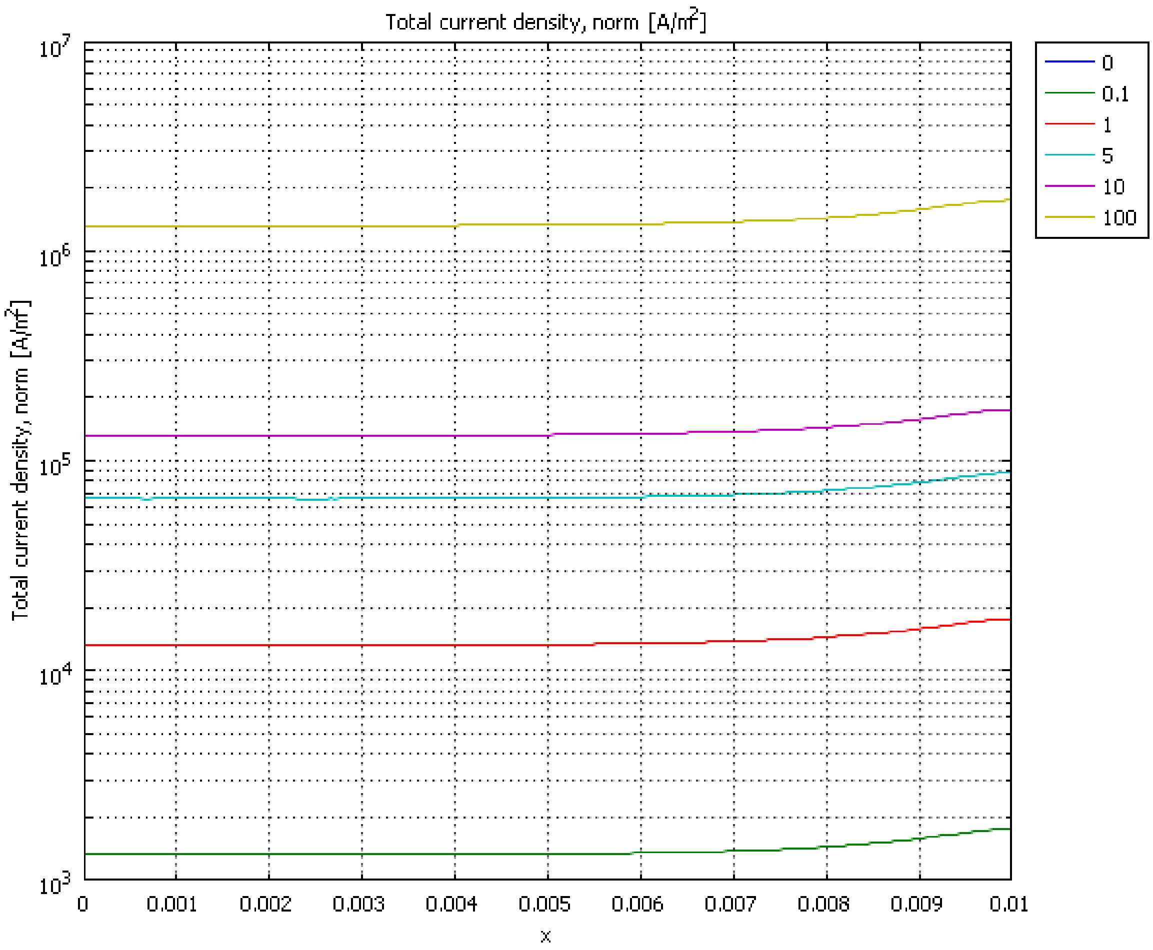

Figure H.3: Parametric analysis of the system.

As the voltage is increased, there seems to be an overall linear increase in the current density, which maintaining a relatively uniform density across the device. This characteristic indicates that by increasing the voltage into the device, the current can be increased, thus increasing the rate in which the device switches.

Upon the successful competition of this model, it was indicated that the parameters the team chose for our prototype should yield use the results the team is looking for in your customer requirements; a uniform and fast switch in opacity.

H.2 Indium Tin Oxide Characterization

ITO coated glass slides for use as electrodes in the electrochromic device were obtained from the Nanotechnology Department and were purchased from Delta Technologies. These are for the purposes of testing and characterizing the films characteristics. The group has purchased ITO on glass with similar specification from Delta Technologies. Two different sized slides were tested and they have the following specifications:

1. Large: 25mmx50mm with resistivity of 7-12 Ω/sq
2. Small: 15mmx15mm with resistivity of 70-90 Ω/sq

It was found through testing that the higher resistivity film actually had a similar resistivity to the larger film, so they were both almost identical. In transparent conductive oxides the general rule is that the thicker the film the lower the resistance and lower transmittance. In general the team want a sheet resistance of <20Ω/sq so the current specified devices work well for electrochromic applications.

Film thickness was determined by ellipsometry, this allows the total resistance of the device to be determined as well as provides a useful benchmark to link the transparency and sheet resistance.

The spectral characteristics were determined by UV-Vis spectrometry. It is imperitive to have a baseline of our maximum absorbance with the transparent conductive oxides on glass. Now as each layer is deposited, a UV-spec will be taken to see the changes on the optical transmittance. It is important to do this such that the group can predict and optimize certain steps of the fabrication.

H.3 Sheet resistance

The sheet resistance of the ITO glass slides was tested on a four point probe. Sheet resistance is similar to the resistivity, except in a thin film setting where:

$R = \rho/t * L/W = R_s*L/W$

With a current of 0.4532mA passing between the outer probes Table H.1 [T1] summarizes the voltages and hence sheet resistances were obtained for the Large and Small samples.

Table H.1 - Voltage measured and calculated sheet resistance of ITO films

Sample	Voltage Measured	Sheet Resistance[3]
Large	7.825 mV	7.825 Ω/sq
Small	10.279 mV	10.279 Ω/sq

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[3] $R_s = 4.532 * V/I$

The resistivity is within range of several papers in order to be suitable for electrochromic applications.

H.4 Film Thickness

The thin film thickness was measured by ellipsometry, a technique using polarized light incident on a sample surface wherein the different layers contribute to distortions to the light polarization. By fitting the data to a model of the substrate and subsequent layers the film thicknesses can be determined.

The model used was that of a SiO_2 substrate (representing glass) and a ITO layer deposited on top (no voids or spaces, assuming a uniform thickness).

Using an initial guess of 1000Å (a decent estimate for an ITO deposition in the resistance range measured previously), the results were as follows for the two samples.

Table H.2: Thickness results.

Sample	Thickness
Large	1791 Å = 179.1 nm
Small	1410 Å = 141 nm

Using different initial guesses from 10-200nm converged to the same result when the data was fit to the model, so there is quite high confidence that these are the actual film thicknesses. Additional methods of verification are profilometry and atomic force microscopy (after scratching away a portion of the ITO to measure step height). The latter method, AFM, can also be used to get an image of the surface topography and this characterization will be completed at a later date.

There is agreement between the thickness and the sheet resistance because the smaller sample showed a higher sheet resistance, which corresponds to a thinner film thickness.

H.5 Optical Characteristics

The optical characteristics were measured by a Perkin Elmer UV-Vis spectrometer. The range was specified to be 200-800nm including the UV range and visible spectrum. Three samples were testing for this: Large ITO, Small ITO, and a blank glass slide. The blank slide was used as a comparison to show the decrease in optical transparency as layers are built. The blank will be used throughout the project to ensure the 'background' is constant.

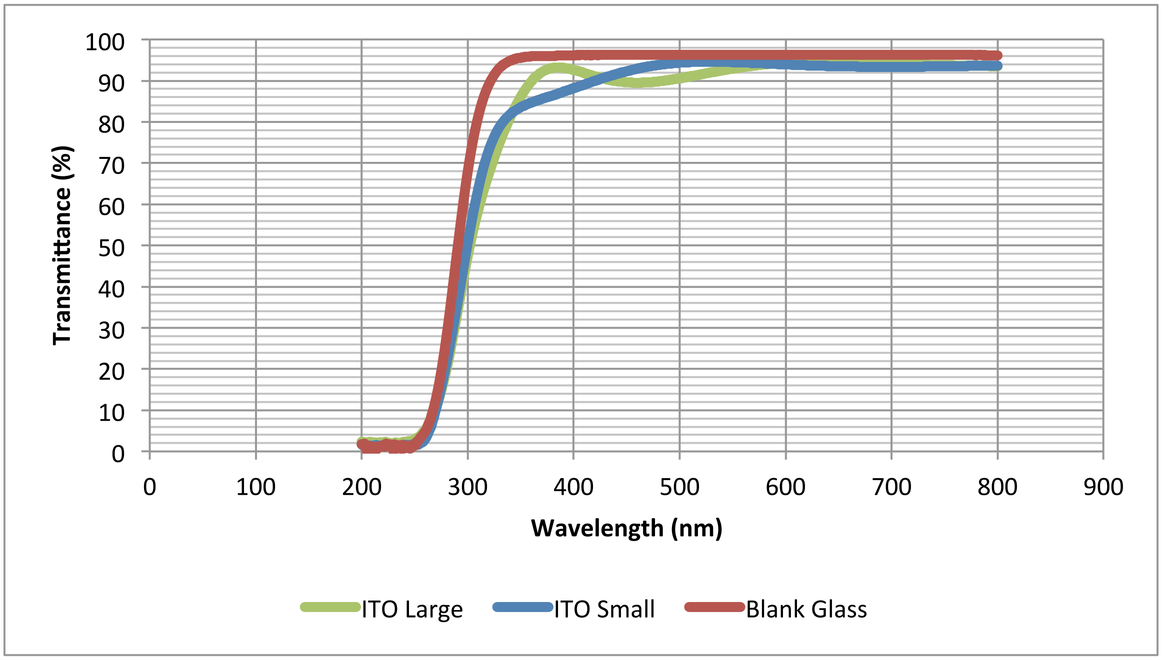

Figure H.4 - UV-Vis spectrum of various ITO slides against a blank slide

There is approximately a 15% decrease optical transparency in the range of 300-400nm for the Small ITO sample. The Small sample increases in transmittance throughout the visible range until it reaches the transmittance of pure glass (upon which the team cannot improve) at 480nm. The Large sample shows a dip in transparency between 400 and 500nm. Hence blue light is being absorbed and the film gives off a green color when tilted to the light.

The averaging the values for UV transmittance in the 200-400nm range is 45%. In the visible range (400-750nm) the transmittance is 93%. Placing two slides will result in a transparency of 86% visible and 20% UV.

H.6 Consequences for Project Design

If the group would like to deposit their own ITO on polycarbonate lenses or other substrates, the team now have solid evidence to determine what layer thicknesses to put down to achieve desired resistivity and optical transparency.

As the specifications for resistivity are within range, the group most likely does not need gold patterned grid on top of the ITO. This should save significant design time. Although the actual test of this should come from modeling, and from the actual device once it is made.
The optical transparency is quite high, however the group must keep in mind that there will be two layers of ITO to make a complete circuit. Having two ITO layers will have an average transparency of 86% which is rather high. Adding our gel electrolyte will reduce this by a few percent and our layers by another. The team is expecting an off state of 75-80% transparency. If the team can change the on state to <40% the team will have a successful prototype.

H.7 Literature Research

A significant body of research supports our design. Similar designs using identical materials for the layers have been implemented for other applications such as windows. In particular, the article, a group in China has had success in building a solid-state electrochromic device using the exact layer composition the team intend to use for the polymer method [H.1].They discuss windows as the main application for this device. The PMMA gel electrolyte of $LiClO_4$/PC was found to have a conductivity of 10^{-3} S/cm. They report the main drawback as leakage of electrolyte and weight gain from the electrolyte solution within the windows. Leakage could be a similarly important setback for our team. The team intends to use an adhesive film to seal the lenses. Weight gain should not be a significant problem in the case of our design owing to the much smaller surface area for our application in comparison to a window [H.1].

The annealing temperatures discussed in the article should not pose a problem so long as the device is built on glass. In order to move to PE, lower temperatures would have to be experimented with. For this reason, it is likely our final design will be on glass.

The deposition techniques discussed in the article are also available to us. They include manual dipping for WO_3, electrochemical deposition through the use of a potentiostat for Prussian blue, and injection by syringe for the electrolyte layer. As with our design, glass with ITO already deposited was purchased pre-manufactured. The gelation time for the electrolyte was approximately three days. 0.5 to 1 V were used to switch the device. The rates of colouring and bleaching were 60s and 30s, respectively. However, these values correspond to a device with dimensions of 40cm by 40cm, whereas are device will be close to $1cm^2$ [H.1]. Hence it is reasonable to expect that our device will require less voltage and less time to switch.

Similarly, a good body of literature supports the hybrid organometallic method. In particular a team in Japan had success with a process very similar to the one the team intends to use [H.2]. Again their intended application was windows. The organic-metallic polyelectrolytes were formed by the complexation of metal ions with bis(terpyridine) organic ligands.

Appendix H – References

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