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SMALL BUSINESS INNOVATION RESEARCH PROGRAM PHASE 1 — FY 1987 PROJECT SUMMARY

Topic No. <u>A88-175</u>

Military Department/Agency <u>Army</u>

Name and Address of Proposing Small Business Firm

Evaporated Coatings, Inc. 2365 Maryland Road Willow Grove, PA 19090

Name and Title of Principal Investigator

John J. Walls, Jr.

Proposal Title

Ocular Protection from Laser Hazards

Technical Abstract (Limit your abstract to 200 words with no classified or proprietary information/data.)

Studies will be directed toward developing/improving current absorbers used in polycarbonate materials as well as providing additional coverage in other spectral regions. In addition to the polycarbonate studies, plasma sputtering deposition techniques will be evaluated to determine their potential use for depositing multilayer coatings on absorbing polycarbonate ophthalmic material.

Anticipated Benefits/Potential Commercial Applications of the Research or Development

Enhanced laser protection properties with abrasion resistance and ballistic protection are anticipated benefits. There are a host of applications in both the military and commercial sector where laser protective devices can be utilized.

List a maximum of 8 Key Words that describe the Project.

Eye protection Laser spectacles Laser hazards Nothing on this page is classified or proprietary information/data • Proposal page No. 2

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INTRODUCTION

Today's modern integrated battlefield exposes ground troops to laser emissions either from hostile forces or inadvertently from friendly forces from both the ground and aircraft. These emissions can originate from laser designators, rangefinders. or other laser systems that have visible or infrared emissions of sufficient energy to result in temporary or permanent visual loss. Laser Eye Protection (LEP) as well as ballistic eye protection is therefore a requisite for today's ground troops. In order to achieve this dual protection many Government programs are being directed toward the development of a combat spectacle that provides multiline laser emission protection and protection from physical impact of fragmentations exposure during battlefield action.

The spectacles being developed by various Government programs utilizes polycarbonate material because of its excellent impact resistant properties and the ability to form ophthalmic shapes. Also, the material can act as a host material for various types of absorbing dyes that can be used to absorb laser emissions. The problem associated with using an all absorption type of approach, especially for rejection of visible emission lines, is the wide spectral bandwidths associated with most of the dyes. Although, excellent rejection properties can be obtained, poor photopic and scotopic luminous transmittances are always present. Because of the low luminous transmittance, it is extremely difficult for personnel to see at dusk, dawn or night. In addition, because only certain colors are transmitted, it is very difficult to sight targets because of poor color contrast. The absorption approach can be used in spectral regions outside the visible spectrum if the dye has minimal effects in the visible spectral region.

The two approaches currently being evaluated, for providing narrow band rejection, for the visible spectrum are multilayer interference coatings and holographic gels. The holographic gels do provide excellent spectral properties, but are susceptible to environmental conditions and durability properties. As with the multilayer coatings, emissions that impinge on the filters at large angles of incidence will cause rejection bands to shift to lower wavelengths and even outside the required rejection emission wavelength.

The multilayer interference coatings do provide enhanced durability and environmental properties compared to the holographic gels. There are however, some limitations that restrict spectral performance. Limitations in the number of layers and their adhesion to polycarbonate material as well as the overall quality of the films can limit optical density performance.

The objectives of this research effort are specifically directed toward enhancing multilayer performance and developing new absorptive dyes for the near IR spectral region. Evaporated Coatings, Inc.*will evaluate a plasma deposition technique to determine the feasibility of applying durable and adherent coatings to the polycarbonate materials. Glendale Protective. Technologies, Inc. will provide an effort primarily directed toward developing a dyed polycarbonate material that absorbs near IR spectral emissions and has maximum visible transmittance.

The studies performed for each of the two specific areas include thin film computer design, determination of optical and physical properties, environmental and spectral evaluations of the coated components. Discussion of results and recommendations are included. Test samples will be provided to Letterman Army Institute of Research (LAIR) personnel for evaluation.

*Robert J. Tucker, Technical Director Glendale Protective Technologies, Inc.

EXPERIMENTATION

PLASMA COATING STUDIES

A major problem associated with depositing multilayer coatings on plastic materials is the inability of the coating to adhere to the plastic surface. Defects such as coating crazing and delamination are common type defects that are experienced when coatings are applied to plastics. The defects occur because the stress in the multilayer coating becomes greater than the adhesive force of the coating to the plastic surface. Therefore, to minimize this type of defect, the stress in the multilayer coating should be reduced and/or the adhesion of the coating to the plastic substrate increased. To date, the number of layers that can be deposited on polycarbonate material, without crazing, is approximately thirty layers. This yields and optical density in the 2.0 to 3.0 range in the visible spectral region. It would be very advantageous if the optical density range could be increased to 3.0 to 4.0. This would require approximately an additional fifteen layers.

The basic approach for this study is to determine if the plasma deposition technique will yield better and more adherent coatings than has been obtained through conventional deposition techniques. All of the studies performed are related to reducing coating stress and increasing the coating adhesion properties. The items to be addressed in this investigation include:

> *Experimental Set-up *Material Process Studies *Results

EXPERIMENTAL SET-UP

The deposition technique chosen for this study is a plasma rf sputtering technique. Sputtering provides approximately 100 times the energy of material depositing on the substrate surface compared to a conventional evaporation technique. It is expected that these higher deposition energies will enhance adhesion properties as well as provide a more dense and void free film because of the higher packing energies of the depositing atoms, molecules and ions.

Illustrated in Figure 1 is the sputtering system used for the studies. The vacuum system is an eighteen inch diameter glass chamber pumped with an oil diffusion pump and a mechanical pump. The sputtering system used is a two inch planar magnetron RF sputter source (U.S.Gun). The sputter source

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FIGURE 1 SPUTTERING SYSTEM

provides a high magnetic field that allows low pressure operation (1 to 5 microns) with higher deposition rates than conventional sputtering systems. The magnetic field concentrates the plasma that in turn increases bombardment density and increases deposition rates. The sputtering source is cooled with water to maintain target temperatures at acceptable levels. An "eni" rf power supply provides up to 1100 watts of rf power to the sputter gun. A capacitor/inductor rf matching network is used to match the impedance between the sputter source and the rf power supply. This enables maximum power to be transmitted to the sputter source for any plasma condition. A DAIWA SWR and power meter is used to evaluate the rf matching and setting of the rf power to the sputter source.

A dual gas inlet micrometer system is used to control the ratio of Argon to Oxygen gas for the sputtering plasma. A thermocouple pressure gauge is used to monitor the plasma gases.

The procedure used for a typical sputtering run is as follows:

The chamber pressure is pumped down to the low 10^{-5} torr value. Argon gas is bled into the chamber, via the dual gas control, to a given value. Depending on the material to be sputtered, oxygen can then be added to a new pressure valve, thereby providing a set Argon to Oxygen ratio. The rf power supply is then turned on to a power level to initiate the plasma. At this point the rf matching network is adjusted to minimize reflected power from the target (sputter gun). The power level to the target is then increased to a desired valve (material dependent). The part being coated by the sputtering process is approximately three inches above the sputtering target. The process is then timed for a given value for a specific film thickness. This completes a typical coating process. The system used appears to be very consistent and dependable.

MATERIAL PROCESS STUDIES

The process chosen for depositing optical coatings on plastic substrates must allow minimal heating of the substrate. Temperatures in excess of 75° C will cause substrates to deform as well as cause a stress condition to be created because of the difference in expansion coefficients between the plastic substrate and the coating material. The rf sputtering technique is a process that can be used at controlled temperatures. By controlling the input power levels and gas pressures, temperature effects can be minimized.

The approach that is studied for several optical coating materials is to determine process parameters that will minimize temperature and maximize deposition rates and quality of depositing materials. The process parameters studied for each of the coating materials are: Argon/Oxygen process pressures, sputtering power levels and deposition times. Each of these parameters are varied to yield practical and optimal physical end optical properties for the deposited films.

The materials studied for processing are silicon dioxide, tantalum oxide and titanium oxide. These materials were selected based on their bulk optical and physical properties. For example, using the bulk optical properties of these materials a thirtyfive layer theoretical design could yield the spectral transmittance properties illustrated in Figure 2. This design was generated to maximize the blue transmittance (scotopic transmittance) and reject a line in the green spectral region. The optical density is in the 3.0 to 4.0 range with a half band This filter has good scotopic and width of approximately 40nm. photopic transmittance. This is but one design example of the type of designs that can be generated with these three materials. Other more complex designs can be generated to yield multiline rejection bands with these same materials.

Each of the materials were initially studied using glass substrates to determine their optical and physical properties. After optimal process parameters were determined, polycarbonate substrates were used to determine the compatibility of the process and coating material with the polycarbonate material.

RESULTS

TiO, Films - Several sputtering runs of titanium oxide were performed using the sputtering system illustrated in Figure 1. The initial power was set at 80 watts and the Argon to Oxygen pressure ratio to about 3.5. Several coating runs were performed with these parameters with the exception of varying the deposition time to obtained various thicknesses of the titanium oxide films. The films formed on glass substrates were evaluated for adhesion, abrasion resistant properties and refractive index. The films formed on glass were very adherent and durable. The reflectivity of the films formed was lower than those formed by conventional evaporation techniques. This translates into lower refractive index values. The sputtered films had refractive index values of about 2.0, whereas conventional coatings have index values of approximately 2.28. This lower index value is attributed to argon entrapment in the titanium oxide films, thus forming voids that reduce the overall index values. The same experiment was repeated using a lower Argon to Oxygen ratio and all other process parameters the same. Higher refractive indicies were obtained because of the higher oxygen levels.

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OFTICAL COATINGS DESIGN

SOFTWARE PACKAGE By David F.Taylor, Jr

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REF. WAVELENGTH:	0.71000 micron.	EXIT MED.	MAT'L:	BK7ALL
INCIDENCE ANGLE:	0.000000 DESREE	INCIDENT	INDEX:	1.000000
SOURCE SPECTRUM:	UNIFORM	DETECTOR	TYPE:	UNIFORM

WAVELENGTH	S-TRANSMITTANCE	P-TRANSMITTANCE	S & P AVERAGE
(micron)	(PER CENT)	(PER CENT)	(FER CENT)
0.40000	93.13847 4%	93.138474%	93.138474%
0.41000	95.259003%	95.259003%	95.2590037
0.42000	95.630951%	95.630951%	95.630951%
0.43000	95.623627%	95.623627%	95.623627%
0.44000	95.659492%	95.659492%	95.659492%
0.45000	95.598526%	95.598526%	95.598526%
0.46000	95.554550%	95.554550%	95.554550%
0.47000	96.416328%	96.416328%	96.416328%
0.48000	98.745491%	98.745491%	98.745491%
0.49000	99.845436%	99.8454367	99.845436%
0.50000	92.152153%	92.152153%	92.152153%
0.51000	11.135572%	11.135572%	11.135572%
0.52000	0.146898%	0.146898%	0.146898%
0.53000	0.030539%	0.030539%	0.0305397
0.54000	0.031677%	0.031677%	0.031677%
0.55000	0.165017%	0.165017%	0.165017%
0.56000	44.047585%	44.047585%	44.047585%
0.57000	39.985855%	39.985855%	39.9858557
0.58000	35.267048%	35.267048%	35.267048%
0.59000	94.013283%	94.0132837	94.013283%
0.60000	46.9023137	46.902313%	46.9023137
0.61000	81.358322%	81.3583227	81.3583227
0.62000	54.923153%	54.9231537	34.9231334
0.63000	83.355812%	83.3558127	83.3008127
0.64000	55.398567%	33.3783674	33.37836/%
0.65000	93.113342%	93.113342%	73.113342%
0.66000	54.421288%	54.4212887	54.4212887
0.67000	92.1236/27	92.1236/27	72.1200/24
0.68000	61.6363/3%	61-6363/3/	
0.67000	68.81/6767	08.81/676/	00.01/070/
	50.575/437. (5.5707/07/	40.070/40%	45 570740%
WVL.HVE	03.3/7207% 40 047074%	03.3/7207% 40 3430347	0J.J/7207/ 20 2030304
WVH.HVE	67.2400346	07.243034%	07.240004%

Figure 2 - Multilayer Rejection Filter

(Spectral Transmittance)

Films were then deposited on a polycarbonate lens using the new process parameters. The coated lens appeared to be cloudy and the film formed was no longer durable. One explanation for the effect is that polycarbonate absorbs water and during the high energy coating process, this water vapor is released from the plastic. At the same time water vapor is being emitted, titanium oxide molecules are being formed on the plastic surface. This water vapor creates poor adhesion for the depositing film, thus resulting in a crazed film.

In order to circumvent this problem a polycarbonate lens was baked to remove the water vapor and coated with a thin protective organic film. This element was then placed in the vacuum chamber and a titanium oxide sputtered onto the surface using the same process parameters. The results of this test indicated a clear, durable and adherent film of titanium dioxide was formed. It appears that the organic film sealed the polycarbonate surface from absorbing considerably lower water vapor.

Process times of the order of forty five minutes to one hour were required to form an optical quarter wave film thickness at 550nm. There was approximately a 15 to 20°C temperature rise for the polycarbonate substrate during this process time. This is well within the constraints required for this plastic material. Further process studies, for the titanium oxide films, will no doubt provide better optical properties that should approach bulk properties. It appears that this material is a good, durable, high refractive index film material that could be used in a multilayer structure.

<u>Ta O₅ Films</u> - Initial Ta O₅ sputtering was done on glass substrates to isolate optimum sputtering parameters. Sputtering parameters which were varied were power and oxygen/argon sputtering gas mixtures. Base pressure and deposition time were held constant. A one minute low power pre-sputter was done just prior to each deposition, to condition the target. Deposited films were measured for their optical spectral properties using a Perkin-Elmer 330 spectrophotometer. Figure 3 shows a typical spectral transmittance curve of the resultant sputtered film on glass. A perceptible amount of absorption may be seen from the reduction in the peak and valley transmittance at smaller wavelengths. This slight absorption was removed following a low temperature post deposit heat treatment and may be due to the incorporation of argon atoms into the growing film or the lack of enough oxygen during the sputtering process.

The properties of the films may be deduced from the transmission values taken at the peaks and valleys of the scan. The



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index Ni of the film may be calculated from the formula

$$Ni^2 = NoNs \left(\frac{1+\sqrt{R}}{1-\sqrt{R}}\right)$$

where R is the reflectance from the film, No is the refractive index of air and Ns is the refractive index of the glass sv - strate. The order m of the peaks may be deduced from the expression

$$m = \lambda_2 / (\lambda_1 - \lambda_2)$$

and finally the physical thickness d may be obtained using the equation

$$n_i d = m \lambda 4$$

The results of these calculations for several coating runs are illustrated in Figure 4. In addition, a plot of film physical thickness vs sputtering power is illustrated in Figure 5. The deposition rate can be seen to be approximately linear with deposition power. Application engineers have indicated that this may be true over narrow power ranges, but for oxide sputtering in general this cannot be expected to hold. Deposition rates for metal oxides are ~ 1 A/sec. which is much lower than the rates of similar metals at the same power levels. Calculated film indicies varied in the range of 1.9 to 2.1. This is approximately 10 to 15% lower than evaporated films of tantalum oxide.

The variation in film index seen in Figure 4 is due to the variation in the Oxygen/Argon gas ratio that makes up the plasma. The presputter oxygen level drifted during the deposit and did not coincide with the post sputter oxygen level. To eliminate absorption and increase the index of the sputtered films more attention must be given to the oxygen level during the deposit. Two additional deposits were made using polycarbonate as well as glass substrates. At 200 watts of sputtering power the films formed were durable and adherent on glass as well as polycarbonate. At 250 watts of sputtering power the plastic softened during the deposit, thus crazing the film. An additional run was performed by pre-baking the polycarbonate at 100°C in air for one hour. The 250 watts of power produced the same results - softening of

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TRIAL	(ORDER)	PEAK	RFILM	BK INDEX	FILM	PHYS.THK.	POWER	TIME	POST BAKE
8Г	2/4	671	18	1.5139	1.94	865A	300WATTS	4 5 M I N	8
9Г	3A/4	622	17.2	1.5155	1.91	2443	150	45	
101	52/4	580	21.4	1.51715	2.03	3571	250	45	1
WII	5A/4	550	20.3	1.51854	2.00	3438	200	45	1 нк.
WIT	5A/4	533	20.3	1.51854	2.00	3331	200	45	1 НВ. 160 ⁰ С
WII	52/4	534	19.8	1.51854	1.99	3354	200 -	45	1 HR. 225 ⁰ C
1 2 M	5A/4	598,	22.4	1.5164	2.06	3629	300	45	1
1 3M	5A/4	500	23.5	1.52153	2.09	2990	200	45	

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Figure 4 Ta₂0₅ Films

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the plastic and crazing of the film.

It appears that by controlling the sputtering power levels to less than 200 watts, good films of tantalum oxide can be formed. Again the deposition rates are slow, but acceptable.

SiO, Films - Initial deposits of silicon dioxide films, with sputtering power levels at 300 watts produced no appreciable deposit of SiO₂. Figure 6 shows some indication of SiO₂ deposits on a chromé coated slide. The chrome coated slide is utilized to help make the SiO, films optically visible. Computations from the spectral reflectance curve of the chrome/SiO slide indicate that the film is approximately 500Å thick. This film was deposited in 45 minutes. Estimates of the deposition rate is approximately 10Å/min. The power levels were increased to approximately 400 watts. A high index glass (n=1.608) was placed in the chamber for processing. This was done to help make a more accurate spectral reflectance measurement. Data from this curve can be used to determine film thickness, refractive index and deposition rate. An eighty-five minute deposit was performed on the high index glass. Illustrated in Figure 7 is the spectral reflectance curve obtained for the SiO, film. Computations from this curve indicate a refractive index value of 1.48 at 675nm. The thickness of the deposited film is 1140Å. This yields a deposition rate of approximately 13.6Å min.

The SiO₂ films are clear and durable. It is expected that sputtering powers less than 300 watts are required to deposit these films on polycarbonate substrates.

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ABSORPTIVE STUDIES

Glendale Protective Technologies Inc., were tasked to formulate single as well as multiline absorbers in the near IR spectral region. The primary goal is to maximize the optical density for the near IR emission lines and maximize the visible transmittance.

The approach used was to evaluate various formulations in polycarbonate thin film form to determine first if the dye is compatible with the host material--polycarbonate and second to determine the optical properties of the polycarbonate absorber prior to forming it into polycarbonate blanks. It is easier in the initial stages of study to use thin film formulations for evaluation. Those dyes that are compatible with polycarbonate and have desireable optical properties (optical density, scotopic and photopic transmittance) were selected and molded into polycarbonate blanks. Single, double and triple emission line absorbers, for the near IR spectral region, were molded and evaluated for their spectral properties.

Another absorption approach that was evaluated was to determine if the surface of a plain polycarbonate ophthalmic blank could be dyed. This approach was studied, but was not successful. It was decided to concentrate our efforts on the homogeneous polycarbonate absorbers.

There were six different absorbing polycarbonate systems selected for evaluation. Each was molded into polycarbonate blanks. The spectral transmittance, optical density, scotopic and photopic luminous transmittance were evaluated and are presented below.

SINGLE LINE ABSORBERS

ABSORBER #R10113-80.10. - The spectral transmittance for three different concentrations was measured and is illustrated in Figures 8, 9, 10. The highest concentration yields a near IR emission line optical density of greater than 5.0. The scotopic transmittance is 17.1% and the photopic transmittance 34.4%. The middle concentration yields an optical density of 4.0 with a scotopic and photopic transmittance of 41.3% and 56.5%. The lowest concentration yeilds an optical density of 1.4 with a scotopic and photopic transmittance of 77.6% and 84.1% respectively. The visible transmittance is fairly neutral for the lower and middle concentrations.

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ABSORBER #R10113-80A. - The spectral transmittance curve for this absorber is illustrated in Figure 11. The optical density is greater than 4.0 at the wavelength of maximum density and has a scotopic and photopic transmittance of 41.6% and 50.1% respectively. There is a possibility that the location of the maximum absorption wavelength band could be moved by further synthetic and analytic techniques.

ABSORBER #R10113-96B. - The spectral curve for this absorber is illustrated in Figure 12. The optical density is 1.0 at the wavelength of maximum absorption. The scotopic and photopic luminous transmittance are 69.5% and 75.8%. There is also a possibility of shifting this absorption band. One positive property for this absorber is that it has higher blue transmittance values than are currently being used.

DUAL LINE ABSORBERS

ABSORBER#10113-89B - This absorber provides protection for the red spectral line and a near IR line. Illustrated in Figure 13 is the spectral transmittance curve for this absorber. The optical density for the red line is 2.3 and for a near IR line is 3.2. The scotopic and photopic luminous transmittance are 42% and 47% respectively.

ABSORBER #R10093-136A - This dual absorber is designed for two near IR lines. A typical spectral transmittance curve is illustrated in Figure 14. The optical densities at each of the IR lines are 2.8 and approximately 7. The scotopic and photopic luminous transmittance are 49% and 63% respectively. If the concentration for the higher OD line is reduced to an OD of 3.5 the scotopic and photopic transmittance values are increased to 68% and 73% respectively. This absorber is very promising.

TRIPLE LINE ABSORBER

ABSORBER #R10123-102B - This absorber provides protection for a red and two near IR lines. The spectral transmittance curve is illustrated in Figure 15. The optical densities for the red and two near IR spectral lines are 2.9, 2.4, and 3.7 respectively. The scotopic and photopic transmittance values are very balanced and are 37.0% and 36.2% respectively. This filter provides excellent coverage throughout the near IR spectral range and partially into the visible red spectral region. All molded samples will be sent to LAIR personnel for evaluation.

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CONCLUSIONS/RECOMMENDATIONS

The plasma coating studies yeilded results that indicate that this process can be used to deposit high quality interference films on polycarbonate substrates. The positive results from the study indicate that titanium oxide, tantalum oxide and silicon oxide materials could all be used as base materials for a multilayer stack design. The films formed are durable, clear, adherent and environmentally stable. Some negative aspects of the study are: The deposition rates are rather slow. They are of the order of 2-3Å/min. compared to standard evaporation techniques of approximately 60Å/min. The limitation on the depositon rate is directly related to the amount of sputtering power applied to the target material. For each of the plastic materials studied, power levels above a certain value caused the plastic material to start to deform because of the heat load on the plastic substrate. Another effect that was observed was the non-uniformity of coating on a substrate beyond 1.5 inches in diameter.

The recommendations resulting from this plasma deposition study are as follows:

l. Refine and optimize process parameters to yield higher deposition rates and minimize substrate heating.

2. Utilize a substrate rotation fixture to improve coating uniformity.

3. Evaluate a high pressure plasma process that provides more uniformity on curved surfaces by a diffusion process.

4. Evaluate forming metal oxides by sputtering the base metal with oxygen as the sputtering gas. Higher deposition rates may be possible with this approach.

5. Deposit a multilayer stack by sputtering the materials studied to determine their properties compared to a conventional deposited multilayer stack.

6. The final recommendation is to utilize the souttering process to form the initial layers of a multilayer stack in order to form a good base on the polycarbonate substrate. This could then be followed by using fast conventional evaporation techniques to complete the hybrid multilayer coating. This could provide a more practical approach because of the long process time required for an all sputtered multilayer coating. Mostly all of these recommendations have been included in the SBIR Phase II proposal. The absorptive studies have provided several absorbers that reject several emission lines in the red and near IR spectral regions. The study has demonstrated that our initial goals are feasible as well as practical.

It is recommended that studies be pursued to refine current absorbers and develop new absorbers to enhance visible transmittance. Also, selected polycarbonate absorbers should be formed into spectacle shapes with base curvatures of 6 or 9 to demonstrate both ballistic as well as laser eye protection.

A final recommendation is to combine both the plasma sputtering coating process and the absorptive polycarbonate approach to yield a spectacle with ballistic eye protection as well as laser eye protection for both the visible and near IR spectral range.