

Multi-layer Laser Mirrors for Optical Sensors Deposited by Electron Beam Deposition and Ion Assisted Electron Beam Deposition



Laxminarayana Gangalakurti, K Venugopal Reddy, I M Chhabra

Abstract: High reflectivity and low loss mirrors are very much essential for state-of-art sensors like Ring Laser Gyroscopes widely used for navigation of moving platforms. Surface scattering is the main source of loss for the Laser mirrors of high reflectivity. Surface scattering also lead to coupling of back reflected light leading to dead band of optical sensors at low input rotation. Super polished glass substrates have been prepared from low expansion glass ceramic material. Surface roughness is achieved about 3-5Å (RMS value) for the ceramic glass substrates prior to thin film coating. The substrates are deposited with 41 alternative layers of SiO₂ and Ta₂O₅ of Quarter-Wave optical thickness with Electron Beam Deposition (EBD) technique. Ion beam Assisted Electron Beam Deposition (IAEBD) is used for another batch of substrates to coat identical stack of layers on glass substrates of the same surface quality. The key process parameters of both the coating processes are presented. The optical constants of the films are evaluated with spectroscopic Ellipsometer from 400nm to 1600nm. Total Integrated Scattering is estimated from the achieved surface finishing of mirrors deposited. The multi-layer coatings are characterized with UV-Vis-IR Spectrophotometer and Cavity Ring Down(CRD) loss meter. The quantitative optical loss of the mirrors is measured at 45° angle of incidence with CRD loss meter. The merits of ion assisted EB process over simple EB coating processes are evaluated against optical constants of thin films and performances of mirrors. Fitness of the processes for realizing Laser mirrors for optical sensors at 632.8nm is evaluated.

Keywords : Ring Laser, Electron Beam deposition, Ion Assisted Electron Beam deposition, surface roughness, reflectivity, extinction coefficient, cavity ring down loss meter, Tantalum pentoxide, silicon dioxide.

I. INTRODUCTION

High reflectivity thin films have several applications in optical metrology instruments and electro-optic devices like Ring Laser Gyroscopes and Gravity Sensors. Ring Laser Gyroscopes (RLG) are rotational sensors for strap down navigation for ships, submarines and moving platforms. Rotational sensors where a rate bias stability requires better

than 0.1°/hr engages RLG for its quick start and non-moving parts in the sensor assembly. RLGs require mirrors of reflectivity more than 99.9%. High reflectivity values of the order of 99.9% or better can be achieved with dielectric multi-layer coating. Multi layer dielectric thin films are deposited with alternatively high and low refractive index materials of a quarter wave thickness of the central wavelength of interest. Generally, SiO₂ is chosen as low refractive index material while Ta₂O₅ or TiO₂ as high refractive index material¹. Hafnium oxide is also a potential candidate with high refractive index for use in the UV spectral range and with low absorption². For the applications of Laser resonators, surface scattering should be minimum possible and should be with a very low surface roughness value. Roughness of the surface is the measure of vertical deviations from a reference surface. Roughness value depends on the type of polishing techniques adopted to achieve the polished surfaces prior to thin film coating. The process of thin film coating controls the film properties like film adhesion, surface roughness, reflectivity, optical constants, etc.

In this work, thin film coating is carried out in two different techniques on the same quality of substrates, and the quantitative measurement is carried out for the roughness of the surface. Roughness values achieved in the two processes namely EBD and IAEBD technique are contemplated for the Total Integrated Scattering (TIS), which is an important parameter for the application of mirror surfaces for the application of Laser resonators^{3,4}. These techniques have been found to be very useful for fabricating Laser mirrors with TiO₂ and SiO₂ alternatively⁵. The present work brings out the merits of coating techniques correlating, optical constants, reflectivity, total loss and TIS values.

II. EXPERIMENTAL WORK

A. Electron Beam Deposition

Electron Beam Deposition is a form of physical vapour deposition in which target material is bombarded with an energetic electron beam. Electrons are typically generated by a thermionic source or by cold electron emitter, which acts as cathode. The electrons are streamed out of the cathode and directed towards target material placed in a crucible. A pressure less than 0.01 Pa is generally maintained so that one can take advantage of the long electron mean free path inside the vacuum chamber⁶.

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A schematic of evaporation of target material placed in a crucible with an electron beam is depicted in Fig. 1.

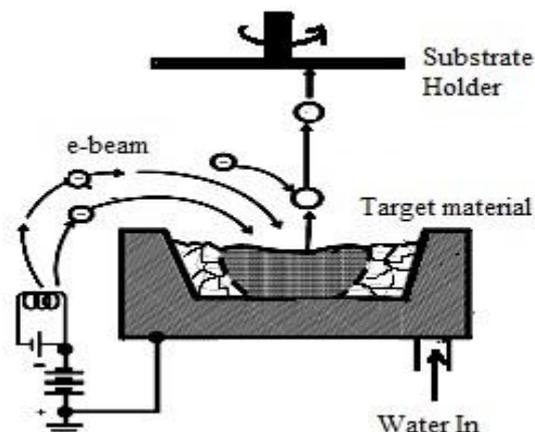


Fig. 1: Schematic of Electron Beam Deposition

In an EBD system, the deposition chamber is to be evacuated to a vacuum of at least 7.5×10^{-5} Torr to allow passage of electrons from the electron gun to the evaporation material. Vacuum in the chamber is maintained with a combination of a diffusion pump (pumping speed is around 1700 ltr/sec) and a rotary pump (pumping speed is around 40 m^3/hr). In the present work, two materials, Ta_2O_5 and SiO_2 in powder form of grain size around 1 to 3 mm in separate crucibles are used as high and low refractive index materials respectively. Target material of purity more than 99.99% is utilized in the present work. The electron beam gun is equipped with four crucibles made of Molybdenum. Electron beam emitted by Tungsten filament of the gun is further accelerated to a high kinetic energy and directed towards the material to be evaporated. The e-beam leaving the cathode is bent about 270 degree by the internal permanent magnet due to which the life of the filament increases. Upon striking the evaporation material, the electrons lose their energy very rapidly. The thermal energy generated in turn melts or sublimates the target material⁶. Accelerating voltages of electron beam can be between 4 kV – 10 kV. The filament voltages are 0-12 VAC with maximum current rating of 70 A.

Amplitude and frequency of the beam pattern in the crucible is adjusted with electromagnets. Both the crucible and the gun are cooled continuously with circulation of cooled water with isolation from the chamber. Super polished glass plates are assembled on the substrate holder and fixed on to rotating plate. The substrates are heated in the vacuum chamber up to 200°C to desorb the moisture layer from the substrates. Prior to deposition, the substrates are cleaned with discharge plasma. Bombardment of ions and electrons to clean the surface has been commonly used for many years, primarily employing glow discharge in the deposition system⁷. Ionic plasma is created inside the chamber with an Aluminum High Tension (HT) terminal at the power levels of 3.5 kW at current 500 mA. Dry Argon gas is purged continuously inside the chamber and vacuum is maintained of the order of 10^{-1} to 10^{-2} Torr. The plasma which contains Ar ions bombards with the substrates and etch the substrates. Substrate cleaning prior to deposition is extremely important for proper film adhesion⁷. The voltages to the HT terminal can be adjusted to give maximum plasma plume at a given

purging rate of Argon gas. The extent of plume can be viewed through glass windows of the chamber while adjusting the voltages. The plasma cleaning continued for about 10-15 minutes prior to thin film deposition.

Thickness of the films is monitored with a quartz crystal oscillator which has a resolution of 0.01 \AA . Quartz thickness monitor has been calibrated so as to minimize the errors associated with the location of the substrates and the quartz crystal. Evaporation rate is maintained around 1 Angstrom per second. The substrates are deposited with Ta_2O_5 and SiO_2 materials alternatively of quarter wave thickness at 632.8 nm as per the stack formula mentioned below: i.e.,

$$\text{Substrate} < [\text{HL}]^{20} \text{H} > \text{Air} \quad (1)$$

here, H indicates High refractive material i.e., Ta_2O_5 , while L indicates the Low refractive index material i.e., SiO_2 . The refractive index considered for calculation of quarter wave thickness of SiO_2 at 632.8nm is 1.457 for which the thickness of each layer is 108.57 nm^8 . For Ta_2O_5 , the refractive index is 2.1203 at 632.8 nm, for which the quarter wave thickness is 74.6 nm^9 . Chamber is vented with dry nitrogen for collecting the coated samples.

B. Ion Assisted Electron Beam Deposition

In ion beam assisted electron beam deposition, coating material is evaporated with electron beam and ion beam is projected onto the substrates. The concurrent ion bombardment process differentiates it from other thin film deposition techniques, with an increased kinetic energy of the *adatoms* resulting increased surface mobility⁹. It is well established that ion beam cleaning significantly improves adhesion and permits control over film properties such as morphology, density, stress level, crystallinity and chemical composition¹⁰. Arrangement of IAEBD is depicted in Fig. 2.

The work carried out by Commandre and Pelletier for the study of optical properties of TiO_2 films by using different processes, indicated that TiO_2 films deposited by the energetic processes are more stable than electron-beam (EB) films¹¹. Vemuri S Praveen Kumar *et al* demonstrated angle dependent graded index films of SiO_2 from for deposition¹² angles from 10° to 80° . In the present work, vapour plume is allowed to incidence at 45 degree of incidence covering thickness monitor at the centre of the substrate rotor.

A set of super polished ceramic glass substrates are placed in the vacuum chamber to have multi-layer coating with the Ion Assisted coating process. Before proceeding to thin film deposition, the substrates are heated to a temperature of 150°C . Marco Alvisi *et al.* carried out 5 minutes pre cleaning prior to deposition with ion beam of Ar- O_2 mixture, which resulted higher damage threshold and improved film adhesion to substrate¹³. Jian Leng *et al* reported Ge film deposition on ZnS & Bk7 substrates where the substrate surfaces were pre-cleaned using Ar ion bombardment for 5 minutes in order to further reduce contamination on the substrate surfaces¹⁴.

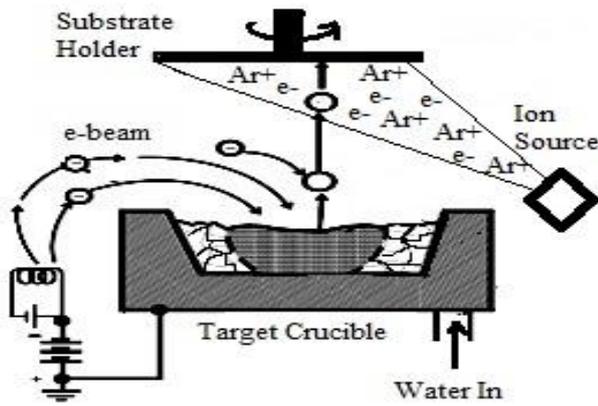


Fig. 2: Schematic of Ion Assisted Electron Beam Deposition.

It is also reported that transmittance peak of the sample prepared without ion-beam bombardment is less than the sample prepared with 150 eV ion energy. The substrates in the present work also were cleaned with ion beam for 5 minutes at a stretch. A grid-less end hall ion source is engaged for generating divergent ion beam on to the substrates. Commandre *et al.* varied ion beam energy up to 1500 eV for deposition of TiO₂ in ion assisted EB coating method and it was observed that the refractive index is maximum when ion beam energy is operated at 300- 400 eV¹¹. In ion-assisted deposition of SiO₂ and TiO₂, when the ion beam energy is more than 500 eV, assist beam damages the film and produces other effects also¹⁵. For SiO₂, Ta₂O₅ films deposition in the present work, the ion source is operated with ion beam energy in the range 300-400 eV. The amount and type of ion bombardment greatly influence film structure and composition. This, in turn, also affects optical, mechanical and electrical properties of the film¹⁶. A partial pressure of around 2x10⁻⁴ mbar to 4x10⁻⁴ mbar is maintained inside the chamber with oxygen gas flow to facilitate better stoichiometry of the films during deposition. The substrates are deposited with 41 layers of Ta₂O₅ and SiO₂ materials alternatively by ion assisted electron beam evaporation process. Throughout the deposition process, the substrates are continuously shone with the assist ion source. In both the deposition processes, the substrates are deposited with the same optical thin film design and stack formula.

III. CHARACTERIZATION OF THIN FILMS

A.Surface Roughness of Thin Films

Surface quality of the bare substrates and the coated surfaces are measured with non-contact Veeco optical surface profiler which has an accuracy of 1A^o. This equipment works on the principle of Phase Shifting Interferometry (PSI). In Phase-Shifting Interferometry, a white light beam is filtered and passed on to a test optical surface. The interferometer beam splitter reflects half of the incident beam to the reference surface within the interferometer¹⁰. Interference fringes are formed on combining waves from test surface and reference optical surface. The reference wave front is moved along the direction of propagation with respect to the wave front being analyzed, thus changing the phase differences¹⁷. Surface roughness of the bare substrates of each batch prior to deposition is recorded with surface profilometer and 2D

profile of one of the bare substrates is shown in Fig.3.

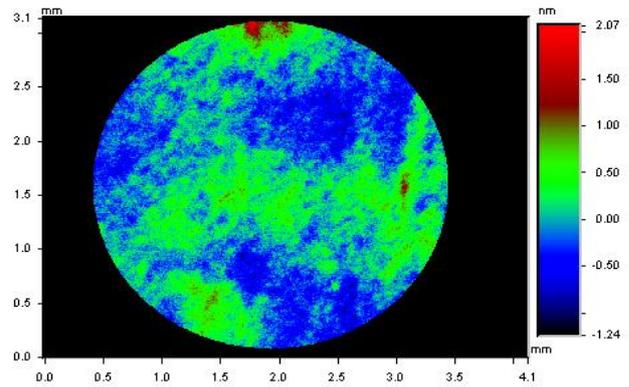


Fig. 3: 2D Surface Profile of Bare Substrate

The multi-layer coatings are evaluated for surface roughness with optical profilometer. The average roughness and rms roughness of the films are 0.85 nm and 1.76 nm respectively with EBD technique. 2D profile of films with EBD and IAEBD mirrors are shown in Fig. 4 and Fig.5 respectively. IAEBD mirrors exhibited an average and rms roughness of 0.36 nm and 0.46 nm respectively. Roughness parameters such as average roughness value R_a, RMS roughness value R_q, 10 point average of peak-valley roughness R_z, highest peak-lowest valley point roughness R_t are measured and are tabulated in Table-I.

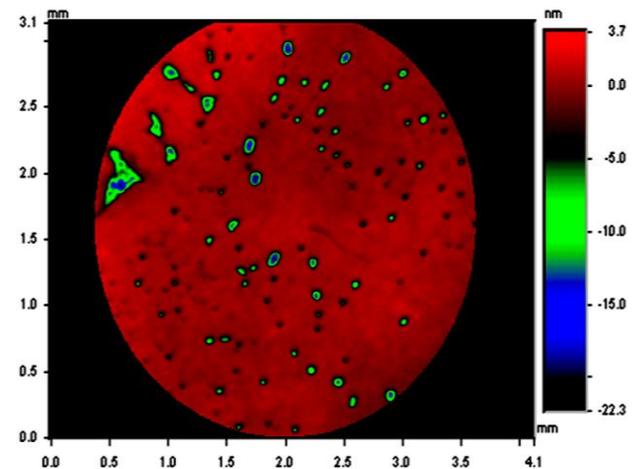


Fig. 4: Surface Profile of EBD Laser Mirror

Table-I: Surface Roughness of Bare Substrate, EBD and IAEBD Films

Roughness parameter	Bare substrate	EBD Film	IAEBD Film
R _a (nm)	0.27	0.85	0.36
R _q (nm)	0.34	1.76	0.46
R _z (nm)	2.82	21.80	4.61
R _t (nm)	3.31	26.06	11.17

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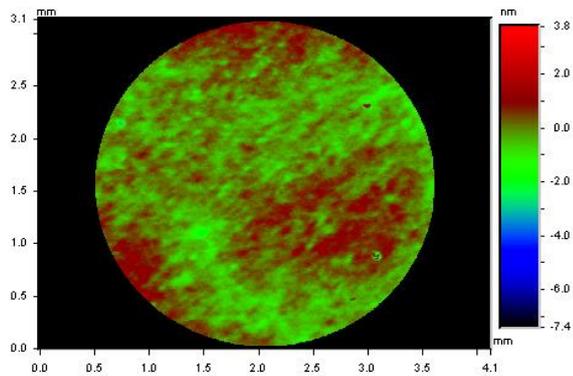


Fig. 5: Surface Profile IAEBD Laser Mirror

The total integrated scattering is related to the amplitudes of scattered irradiance and the total irradiance as follows:

$$TIS = 1 - \frac{R_s}{R_t} \quad (2)$$

According to Beckmann and Spizzichino's rough-surface light scattering model, the relation between the scattered light and surface roughness is as follows⁴ in the equation (3).

$$TIS = 1 - e \left[- \left(\frac{4\pi \cdot \cos \theta_i \cdot R_q}{\lambda} \right)^2 \right] \quad (3)$$

here R_q is rms roughness of an optical surface, θ_i is angle of incidence, and λ denotes wavelength of light.

Table-II: Total Integrated Scattering Values of Bare Substrate, EBD and IAEBD Films

Parameter	Bare Substrate	EBD Mirror	IAEBD Mirror
TIS	4.6×10^{-5}	1.2×10^{-3}	8.3×10^{-5}

IAEBD mirrors have lower scattering values compared to EBD mirrors.

B. Optical Constants of Thin films

An automated M-2000 VI Spectroscopic Ellipsometer of J A Woollam make, which can measure thin film properties in the range of 370 - 1690 nm wavelength, is engaged for analysis. Spectroscopic ellipsometry (SE) is a non-destructive method used to determine physical thickness and optical constants of coatings. In ellipsometry, the variation of the amplitude and the phase difference between the perpendicular (p) and the parallel (s) components of the reflected light polarized with respect to the plane of incidence are measured¹⁸. Mukesh *et al* fabricated Ta_2O_5 films of refractive index of 2.0419 at 550 nm without assisted ion beam on a heated substrate at 250°C¹⁹. The single layer SiO_2 film and the single layer Ta_2O_5 film deposited on two separate substrates, by EBD process are evaluated for optical constants with the ellipsometer. Similarly, single layer SiO_2 film and the single layer Ta_2O_5 films deposited on two separate substrates by IAEBD process are also evaluated. Najwa sidqi *et al* demonstrated SiO_2 films deposited by IAD process from SiO_2 source with no oxygen backfill at a deposition rate of 2 Å/s and achieved refractive index between 1.47 and 1.45 in the wavelength range 500-1000 nm²⁰.

Refractive indices and extinction coefficients of SiO_2 are plotted in Fig.6 & Fig.8. For Ta_2O_5 film the optical constants are plotted in Fig.7 & Fig.9.

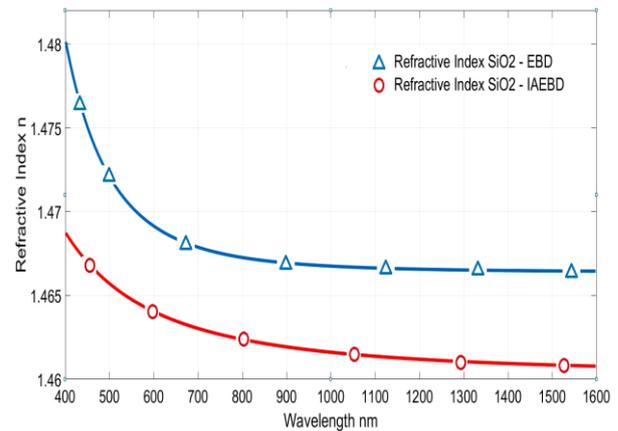


Fig. 6: Refractive indices of SiO_2 films with EB and IAEBD process.

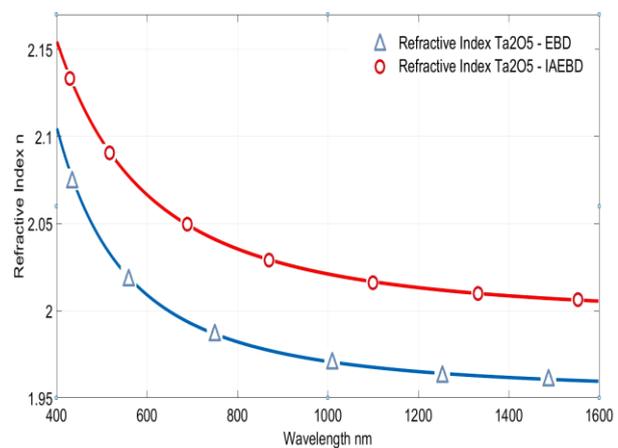


Fig. 7: Refractive indices of Ta_2O_5 films with EB and IAEBD process.

Optical constants of SiO_2 and Ta_2O_5 are tabulated in Table- III and Table- IV at the wavelength of interests where metrological and High power Laser applications. SiO_2 and Ta_2O_5 films deposited with assisted Ion beam coating shown zero value of extinction coefficient, purely dielectric in nature and suitable for Laser mirrors.

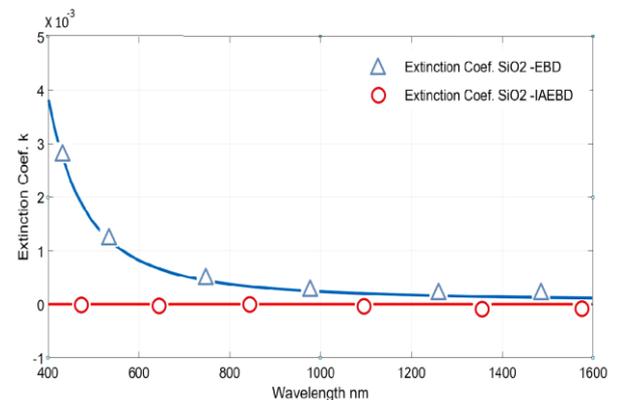


Fig. 8: Extinction Coefficients of SiO_2 films with EB and IAEBD process.

Table-III: Optical Constants of Silicon Dioxide and Tantalum Pentoxide Films with EBD Process

Optical Constant	SiO ₂ Film		Ta ₂ O ₅ Film	
	@ 532 nm	@ 632.8 nm	@ 532 nm	@ 632.8 nm
n	1.4708	1.4686	2.0276	2.0026
k	0.00122	0.0007	0.0049	0.00282

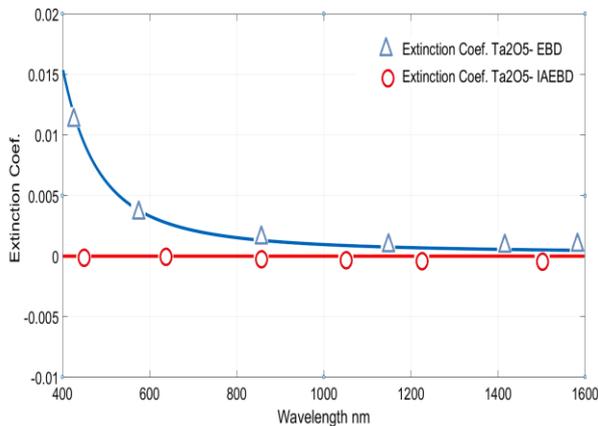


Fig. 9: Extinction Coefficients of Ta₂O₅ films with EB and IAEBD process.

Table-IV: Optical Constants of Silicon Dioxide and Tantalum Pentoxide Films with IAEBD Process

Optical Constant	SiO ₂ Film		Ta ₂ O ₅ Film	
	@ 532 nm	@ 632.8 nm	@ 532 nm	@ 632.8 nm
n	1.4650	1.4636	2.0858	2.0594
k	0	0	0	0

C. Reflectivity of EBD and IAEBD Multi Layer Mirrors

Deposited films are measured for reflectivity with Agilent make UV-VIS-NIR Spectrophotometer. The spectrophotometer has capability of measuring reflectivity and transmission of mirrors at variable angle of incidence. The mirrors deposited, in the present work are measured at 45° angle of incidence and data is plotted in Fig. 10 & Fig. 11. The EBD films showed a flat band of S- polarization from 530 nm to 710 nm with centre wavelength at 620 nm. The flat band width is 180 nm. The IAEBD films, on other hand showed, flat band of S-polarization from 520 to 670 nm with central wavelength of 595 nm. IAEBD mirrors shown narrow flat band and central wavelength shift towards lower wavelengths. IAEBD films exhibited good control of thickness witnessing of smooth fall in amplitudes of harmonics on either side of the flat band of the reflection curve.

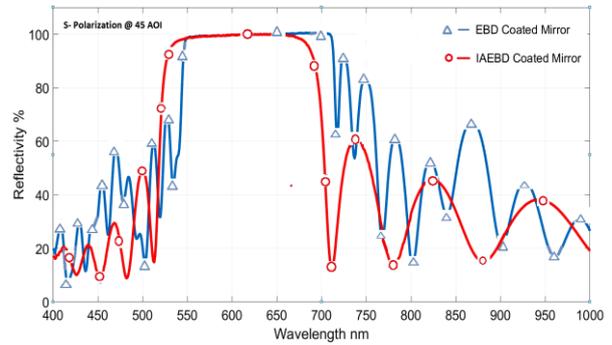


Fig.10. S-Polarization Reflectivity of EB and IAEBD mirrors

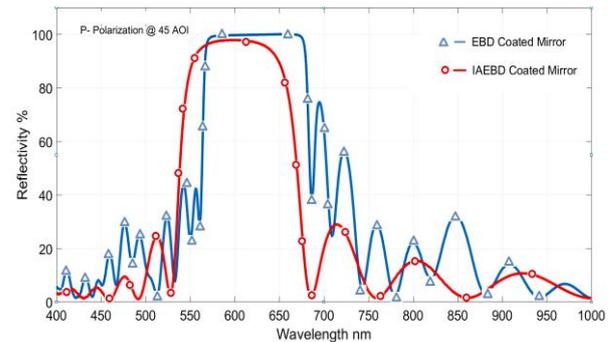


Fig.11. P-Polarization Reflectivity of EB and IAEBD mirrors

D. Cavity Ring Down Loss of EBD and IAEBD Multi Layer Mirrors

Accuracy of the reflectivity of high reflectivity Laser mirrors measured by spectrophotometer is limited to 1%. Cavity ring down loss meter works on the principle of measurement of decay time of the light of the resonator. This technique helps in measuring reflectivity or total loss of very high reflectivity mirrors, where conventional measurement techniques fail. Another advantage of this technique is having immunity towards intensity fluctuations of the Laser source, as the measurement completely relies on the relative change in intensity of the pulse²¹. When the Laser is launched inside the optical cavity, Laser intensity decreases after every round trip. The decrease in intensity is measured with a photodiode by monitoring the light leaking through one of the cavity mirrors. The intensity after a launching time t, inside the cavity enclosed by two mirrors of reflectivity R₁ and R₂ separated by a distance d, is given by²¹

$$I(t) = I_o \cdot e^{-\frac{t}{\tau}} \tag{4}$$

$$\text{where } \tau = -\frac{2d}{c \cdot \ln(R_1 \cdot R_2)} \tag{5}$$

The multi-layer mirror coatings are tested with the loss meter for total loss of the mirror at 635 nm wavelength. Initially, the loss meter measurements are carried out with reference linear cavity of mirrors R₁ and R₂. Laser pulse is

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launched in to the cavity from R_1 and the leaking beam is monitored from mirror R_2 . The test mirror R is introduced in the cavity such that Laser beam from mirror R_1 is incident at 45° angle to the test mirror. The cavity is aligned so that the photons will be making rounds with in the triangular resonator. Schematic of the loss meter experimental set up is shown in Fig. 12.

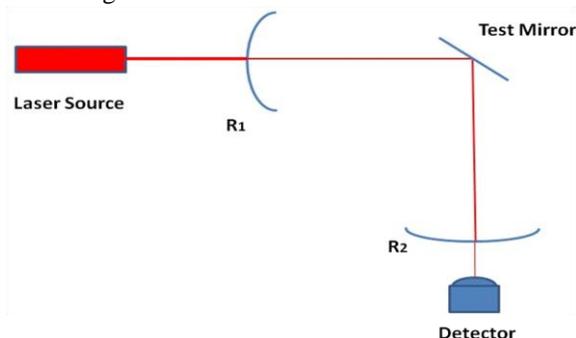


Fig.12. Schematic of Cavity Ring down Loss Measurement Setup

The transmitted light collected from the mirror R_2 is plotted with time. The decay time is measured and reflectivity of test mirror is derived. The total loss obtained for IAEBD mirror is 1753 ppm, where as for the EBD mirror the loss is 3189 ppm. The EBD mirrors shown relatively higher scattering loss due to higher surface roughness and film defects, compared to IAEBD mirrors. The measurements are repeated for three samples in each batch, and a close match of readings is obtained within 20 ppm.

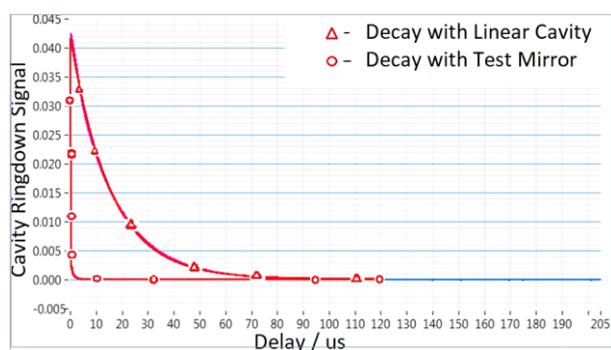


Fig. 13. Cavity Ring Down Decay Time Measurements of EBD Mirror

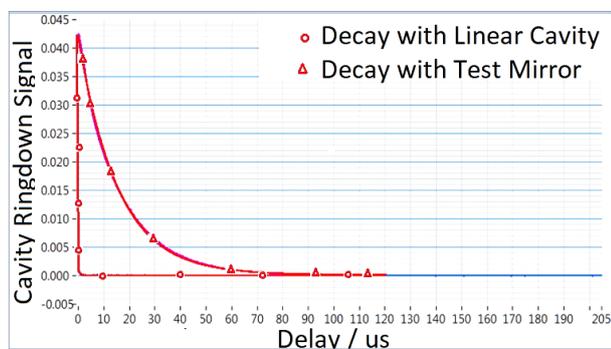


Fig. 14. Cavity Ring Down Decay Time Measurements of IAEBD Mirror

IV. RESULTS AND DISCUSSION

A. Laser Mirrors with EBD Process

The multi-layer dielectric Laser mirrors deposited with EB deposition techniques shown a degraded surface roughness compared to the uncoated substrates due to columnar and bubbles structures of the film grown. The single layer SiO_2 and single layer Ta_2O_5 films exhibited non-zero coefficients of extinction which may lead to absorption of light. The absorption loss is evidenced with more cavity decay loss when measured with cavity ring down loss meter for mirrors realized with EBD process.

B. Laser Mirrors with IAEBD Process

The films grown with additional ion beam gun during electron beam deposition exhibited surface roughness nearly the same as the roughness prior to coating due to enhanced distribution of the film with assisted ion beam. Near zero extinction coefficients for single layer SiO_2 and Ta_2O_5 films are achieved by the coating process of maintaining of partial pressure of oxygen gas inside the chamber during deposition. The estimated Total Integrated Scattering values are two orders lower for the mirrors done with assisted ion beam EB coating than simple EB coated mirrors. The cavity loss of mirrors coated with assisted ion beam process is also relatively lower than that of mirrors deposited with Electron beam alone.

V. SUMMARY AND CONCLUSION

Ta_2O_5 and SiO_2 films realized with IAEBD process are dielectric in nature with zero coefficients of extinction, and also with better surface smoothness, which proves that IAEBD process is superior to simple EBD process. The total integrated scattering should be low for a Laser interacting optical surface to avoid lock-in errors of Laser based rotational sensors. The measured reflectivity and optical losses of mirrors realized with IAEBD process are encouraging and suitable to use mirrors for He-Ne Laser Resonator assemblies at 632.8 nm wavelength.

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