Deposition of Multilayer Optical Films and Rugate Filters Deposited by Reactive Magnetron Sputtering

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ABSTRACT

Reactive magnetron sputtering was used to deposit multilayer optical coatings as well as Rugate filters. For the combination of larger areas, high throughput and high precision of the films, a rotary platter coater was developed which uses reactive pulsed magnetron sputtering. Four substrates of 40 cm diameter can be processed simultaneously. For an optimized homogeneity and film quality, linear double magnetrons were implemented. Plasma process control is made by a lambda probe oxygen partial pressure measurement as well as by optical plasma emission spectroscopy. For in-situ process control of the growing films, a fast single wavelength in-situ ellipsometer and a spectral reflectometer is adapted and implemented into the process control software. All sensors are integrated via a data handler into the SPS process control. First results for the deposition of a multilayer filter as well as of Rugate filters are presented and are compared to results of RF magnetron sputtered filters.

INTRODUCTION

Magnetron sputtering is a promising tool for the future production of precision optical coatings. Advantages are the high film quality, high throughput and the possibility for automation. Possible applications are Laser mirrors or coatings for optoelectronic devices [1]. On one hand, modern plasma assisted evaporation techniques allow the deposition of high quality thin films in combination with a high throughput. In new plasma assisted evaporators areas of more than 1 m^2 can be deposited. On the other hand, dual ion beam sputtering is known to be a technique where the best film quality regarding to defects as well as density is obtained. However, it is no news that the deposition rate of ion beam sputtering too low for a production of a substantial number of pieces. In this relation, magnetron sputtering is an interesting method since it has some advantages:

- Up scaling can be made easily, cathodes lengths of 3,75 m for architectural glazing are in production since several years
- Film quality is excellent
- A flexible production with fast product changes are possible
- Also large area substrates can be coated very homogeneously
- Magnetron sputtering can be completely automized, so that optical coatings can be produced like in the semiconductor industry.

In the present paper, first results of optical coatings made in a new sputter coater "Dyscus" are presented. For comparison, Rugate filters as well as multilayer stacks were deposited on a smaller RF-magnetron sputter coater.

EXPERIMENT

Figure 1 shows schematically the setup of the "Dyscus". The machine is based on reactive pulsed magnetron sputtering with two linear double magnetrons developed in-house. The size of each cathode is 650 x 120 mm^2. As power supply, an ASTRAL Advanced Energy with 2 x 10 kW pinnacle power supplies are used. The diameter of the process chamber is 2,40 m. In addition, a linear RF plasma source (CCR) is adapted in a further compartment of the chamber. Therein, plasma assisted magnetron sputtering experiments can be performed. Results of this experiments will be presented elsewhere. After handling the substrates via the handler into the process chamber, up to four substrates can be positioned on the rotating platter. The rotating frequency of the platter can be varied between 5 and 100 rpm. Because of the larger substrate size (40 cm diameter), the substrate holders are equipped with a sub-rotation with variable speed between 25 and 500 rpm. Also, RF bias and substrate cooling are implemented for temperature sensitive substrates. For plasma process control, a 3 channel acusto-optical plasma spectrometer (AOS from IFU GmbH) as well as a λ-probe were used.

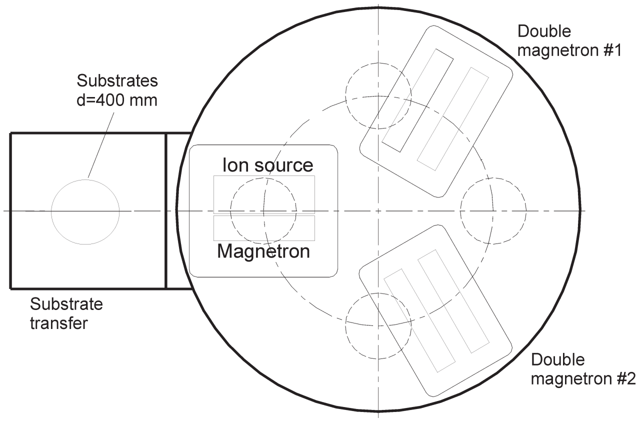

Figure 1: Setup of the sputter coater "Dyscus".

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For the optical monitoring, an in-situ ellipsometer (Sentech Instruments, Germany, model SE 401) and a spectral reflectometer (Eta Optik, Germany) are implemented. Since the ellipsometric measurements have to be performed under dynamic deposition, it has to be very fast. Thus, it was decided to use a single wavelength ellipsometer, where data acquisition time of 100 msec can be obtained. For reflectivity measurements, data acquisition time is well below 50 msec. In order to get access to the monitor data, the different optical sensors are triggered individually and the data are collected by a separate computer, so called "data transfer handler". This computer is directly connected to the SPS process control of the sputter machine. Different control types can be performed: Time control, monitor mode and fully automized control. In the present paper, only time control was used.

MATERIAL PROPERTIES

Figure 2 shows the refractive index of different films deposited on glass in the dynamic mode, i.e. with moving substrates and with sub-rotation. The films were deposited in the oxide mode at 2 x 6 kW (7.7 W/cm^2). For deposition of TiO_2, Si_3N_4 and SiO_2, metallic Ti and Si targets were used while for Nb_2O_5, a ceramic NbO_x target of high density was used. The refractive index for Nb_2O_5 (2.29 at 550 nm) is very similar to the value obtained from sputtering from a metallic target [2] and also in agreement to values reported by Strümpfel [6]. The refractive index of TiO_2 (2.70 at 550 nm) is very high, indicating a high content of Rutile. It is known that the refractive index of sputtered TiO_2 films is sensitively dependent on the plasma conditions and the temperature. In older experiments [3], similar refractive indices were found at mid-frequency magnetron sputtered TiO_2 films in the oxide mode, but also in the transition mode [4]. TiO_2 films deposited by plasma assisted evaporation showed values of around 2.35 [5].

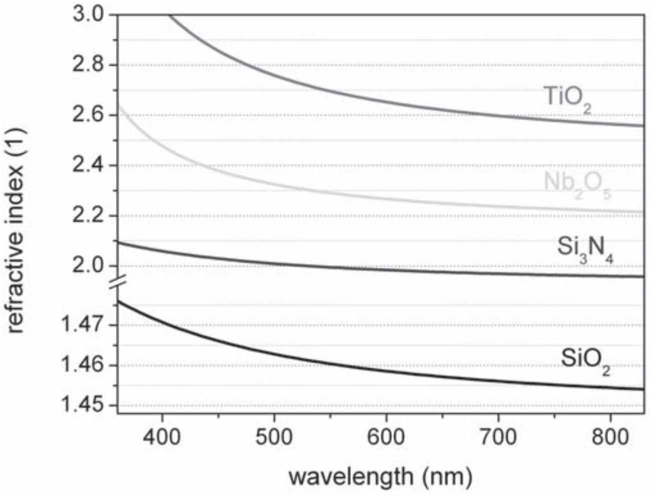

Figure 2: Refractive index dispersion of different materials deposited by dynamic deposition.

To measure the absorption of the films, Laser calorimetric measurements according to ISO 11551 (performed at the Laser Center Hannover, Germany) were performed at 1064 nm. The absorption index calculated from the total loss is around 3.5 x 10^{-6} at 1064 nm for SiO_x and Nb_2O_5 films. The thickness of these films was between 250 nm and 1500 nm.

DEPOSITION OF MULTILAYER FILTERS

Small Area Filter Deposition

In Figure 3, a multilayer long pass filter consisting of 37 alternating layers of SiO_2 and Ta_2O_5 are deposited by RF magnetron sputtering. The specifications of the filter are denoted by the dotted lines. The targets (4'' diameter) used are Silicon oxide and Tantalum oxide. The power was below 1 kW and only time control was applied. The difference of the transmission edge of the designed filter to the experiment is below 1% and it can be seen that the specifications are met (note the measured transmission included back side reflections and has to be shifted vertically by 5% to compare it with the calculated spectrum). FT-IR measurements as well as measurements of the shift of the band edge after a treatment of the sample in vapor for 20 hours (20h, 40°C, 90% r.h.) show that no water is absorbed in the filter, indicating a dense and compact structure.

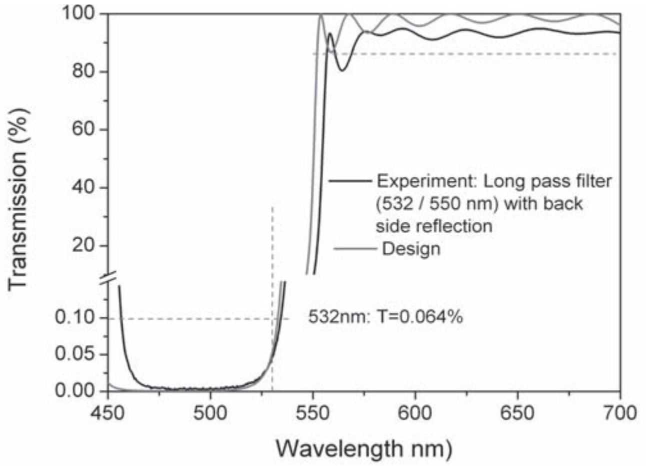

Figure 3: Transmission spectrum of a long pass filter. The specifications are indicated by dotted lines. The filter consists of 37 layers of SiO_2 and Ta_2O_5.

In the second experiment, a Rugate filter consisting of a multi ramp was deposited. The design is shown in Figure 4. The a total thickness is 5 μm. Again, only time control was applied here. Figure 5 shows the resulting transmission spectrum. Also, the calculated design spectrum is shown, with and without absorption in the layers, respectively. First, it shows that the spectra correspond very well together, indicating a good process stability over a long time. Second, it can be seen that the agreement is much better if the absorption in the films is neglected. This corresponds to the results that also the RF-sputtered films have a very low absorption index as shown above.

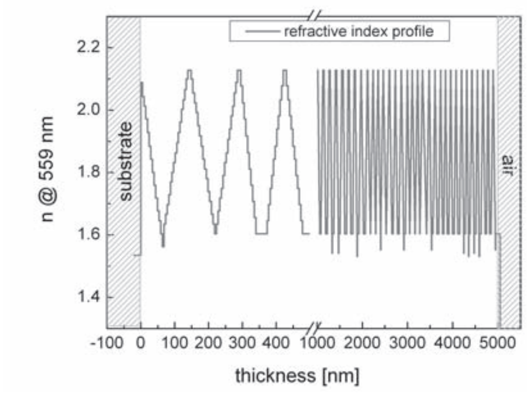

Figure 4: Design of a long pass filter based on linear ramps.

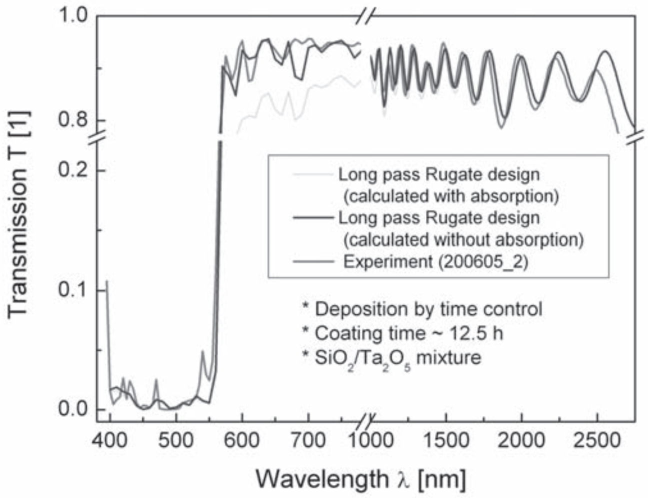

Figure 5: Transmission spectrum of a long pass Rugate filter. The design is shown in Figure 4.

The two examples show that with using RF magnetron sputter technique, a very stable process can be obtained using only time control. Even Rugate filters with good performance can be deposited in the time control mode.

Large Area Filter Deposition

To transfer the results on larger areas, a multilayer filter consisting of SiO_2 and Nb_2O_5 was deposited. To start with, a 32 layer optical filter was deposited on a 40 cm diameter glass substrate in the oxide mode. Only time control was used here. The resulting transmission spectra at different positions at the substrate are shown in Figure 6.

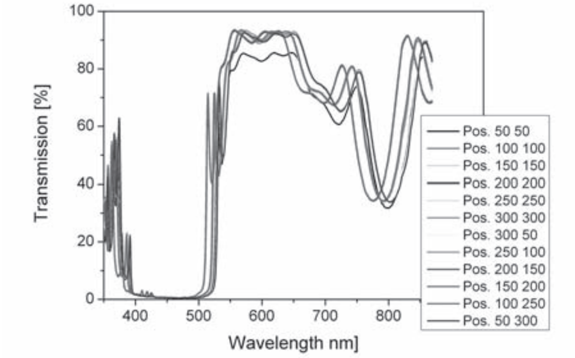

Figure 6: Transmission spectrum of a long pass filter, deposited on a 40 cm diameter glass substrate by pulsed reactive sputtering. Note that no sharper aperture was used here.

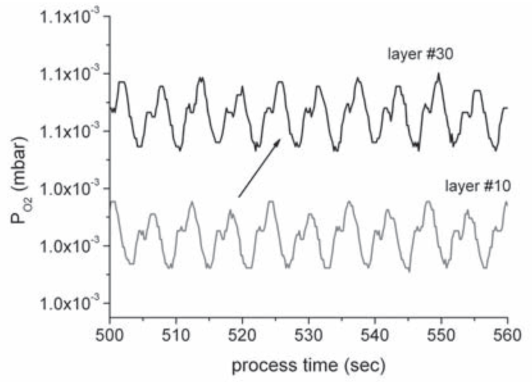

Figure 7: Oxygen partial pressure of the reactive Si process monitored with a λ-probe during the deposition of the filter of Figure 5. Note the long term drift of the pressure from layer 10 to layer 30.

The 31-layer filter was investigated with different techniques: X-ray reflectivity (XRR), spectroscopy multi-angle ellipsometry and secondary ion mass spectroscopy (SIMS). The SIMS data were used as start values for fitting the XRR and ellipsometry data. On one hand, the ellipsometric and the XRR spectra could be fitted very well. The MSE for the ellipsometric fit was 1.6, which is a very good value for such a thick filter. The XRR fit is shown in Figure 9 and the ellipsometric fit in Figure 8. The results show that the XRR thicknesses are within ±2 nm from the SIMS data, while the ellipsometric data vary at maximum ±10 nm, with a standard deviation of 1.8 nm (see Figure 10).

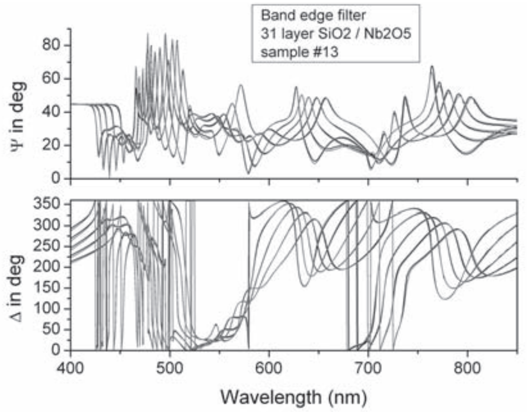

Figure 8: Measured and fitted ellipsometric spectra of the multilayer filter. The mean square error is 1.6 which indicated a very good fit.

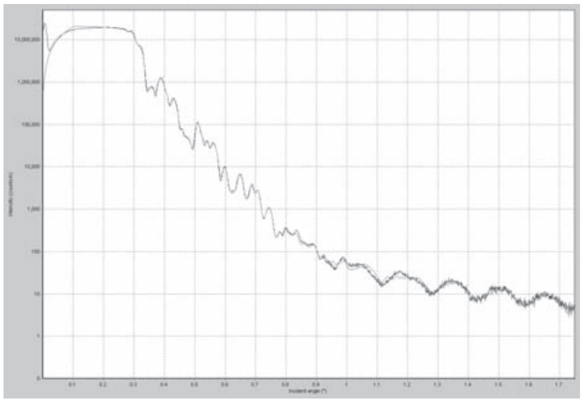

Figure 9: Measured and fitted X-ray reflectivity spectrum of the 31 layer filter of Figure 5.

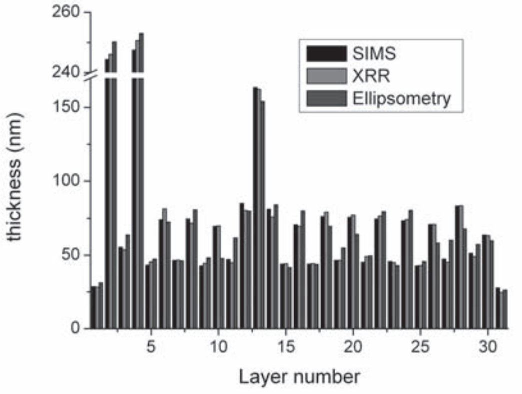

Figure 10: Comparison of the thicknesses of the filter measured by SIMS, XRR and ellipsometry.

Furthermore, the XRR analysis can give some more details about the interface roughness, as shown in Figure 11. In spite of the fact that the analysis is difficult because of the large number of layers, it can be seen that the interface roughness increases with increasing thickness. At higher thicknesses it seems to be that there is a steady state of the interface roughness. Similar results were obtained at simple oxide films [1].

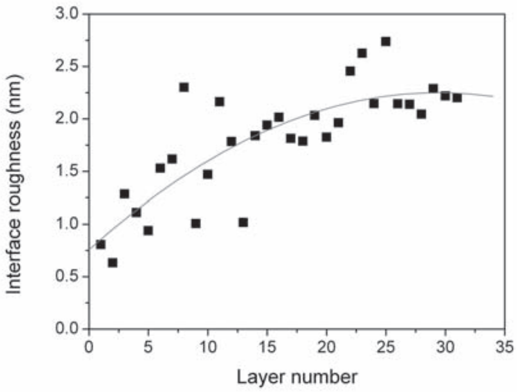

Figure 11: Interface roughness of the filter, determined by the XRR analysis. The solid line is a polynomial fit.

In Figure 12, the homogeneity of the filter of Figure 6 was analyzed. This was done by measuring the relative shift of the wavelength next to the band edge. On the 40 cm diameter substrates, a thickness difference of ±1.25% is obtained. The solid line are calculated curves including statically measured thickness profiles. It is important to notice that no sharper aperture was used here. Therefore, the main contributions of inhomogeneities are arising from the relative difference of the angular speed of the different substrate positions and the stray deposition of the substrate.

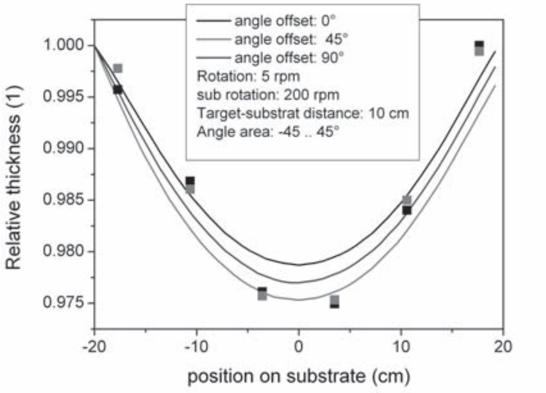

Figure 12: Analysis of the homogeneity of the filter of Figure 6 by measurement of the edge wavelength of the filter. Note that no sharper aperture was used here.

Due to the fast sub-rotation of the substrates, the film thickness distribution is rotation symmetric. Sharper apertures are currently being implemented and further results will be presented soon. It should be noted that the sub rotation speed has to be fast enough to prevent inhomogeneities. In the present case of the long pass filter and a main rotation of 5 rpm, 200 rpm is sufficient.

DEPOSITION OF MULTI GRADIENTS

As a first step to transfer the results for the deposition of Rugate filters on larger areas, gradients and multi gradient filters were deposited with pulsed magnetron sputtering.

Due to the strong coupling of two reactive processes, it was decided to use a ceramic NbO_x target in combination with a Si target. Figure 13 shows the oxygen partial pressure as a function of the power of the NbO_x targets. The power of the Si targets was 2 kW in this case. It shows that the oxygen partial pressure is mainly constant. Vice versa, the oxygen partial pressure during a power ramp at the Si targets with constant power at the NbOx target shows also only a slight variation from 60 to 50 mPa. This profess that the NbO_x target behaves quasi neutral with respect to the oxygen partial pressure.

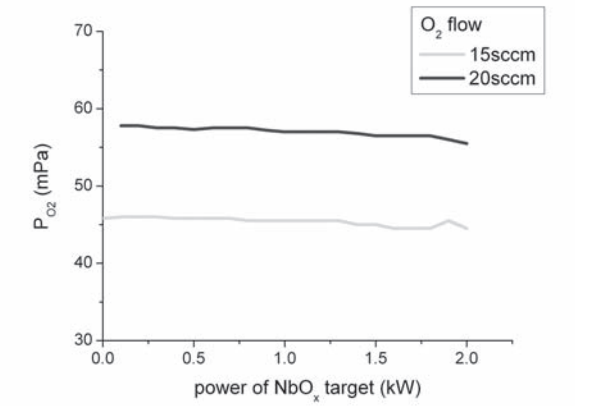

Figure 13: Oxygen partial pressure with two targets running parallel. The power of the Si targets was constant while the power of the NbO_x target was varied. In the other case, the Po2 varies from 60 to 50 mPa while the Si power is increased from 0 to 2 kW.

In Figure 14, a multilayer filter consisting of linear power ramps was deposited. The deposition also was performed without an active process control. Only the power and the oxygen flow was varied during the deposition. It shows the SIMS profile of the filter. It came out that the rate is not linear as the power varies linearly. Therefore, the Silicon peaks are smaller than the Nb peaks. Figure 15 shows the resulting transmission and reflectivity spectrum of the filter, together with a simulation. Note that the simulation is based on linear ramps in stochiometry, so the disagreement is not surprising.

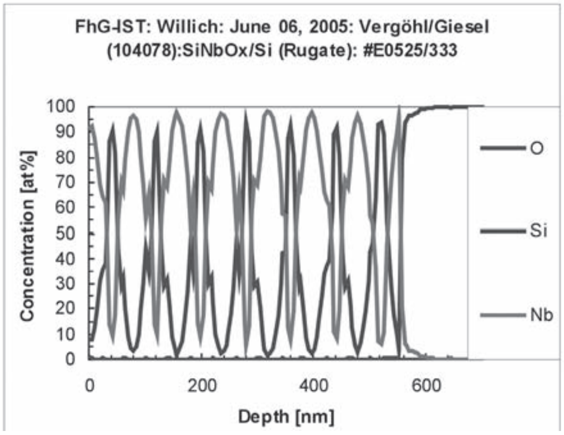

Figure 14: Secondary ion mass spectroscopic data of a linear multi ramp filter of SiO_2/NbO_x. The power of the two targets was linearly varied.

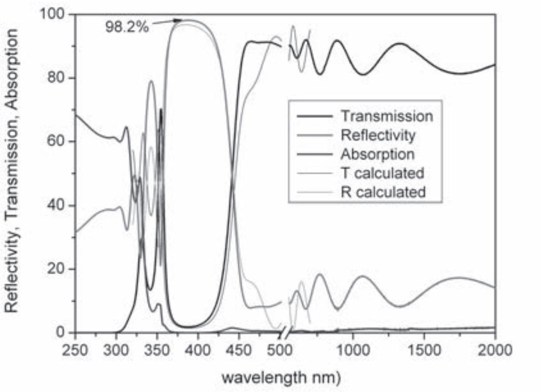

Figure 15: Transmission spectrum of the filter of Figure 7. The solid line was calculated with a sinus profile.

CONCLUSION

In the present paper, multilayer filters as well as gradient films and Rugate filters were deposited with both reactive and non reactive magnetron sputter techniques. With RF magnetron sputtering on smaller substrates (4''), a good process stability and thickness reproducibility could be obtained by using only time control. For an upscaling of the substrate size from 4'' to 40 cm diameter, a reactive magnetron sputter coater was developed. Due to the rotating platter with sub rotation geometry used here, it is possible to obtain homogeneities of ±1.25% without shaper shields. Optical and non-optical investigations by x-ray reflectivity, secondary ion mass spectroscopy and spectroscopic ellipsometry show that the multilayer filters can be deposited with high quality. The density is high (i.e. index of refraction Nb_2O_5: n550 = 2.35; SiO_2: n550=1.458) and the absorption is very low (k=3.5 x 10^{-6} at 1064 nm). It shows that without any process control, the thickness deviations are somewhat larger compared with the RF-process (with smaller cathodes). This is mainly due to the long term drift of the oxygen gas partial pressure, which was not controlled but only monitored in the experiment. Long term partial pressure control will improve the thickness stability. In the case of Rugate filters, encouraging results where obtained on large areas of 40 cm diameter with ceramic NbO_x and metallic Silicon targets.

ACKNOWLEDGEMENT

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