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BDS thin film damage competition

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BDS thin film damage competition

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ABSTRACT

A laser damage competition was held at the 2008 Boulder Damage Symposium in order to determine the current status of thin film laser resistance within the private, academic, and government sectors. This damage competition allows a direct comparison of the current state-of-the-art of high laser resistance coatings since they are all tested using the same damage test setup and the same protocol. A normal incidence high reflector multilayer coating was selected at a wavelength of 1064 nm. The substrates were provided by the submitters. A double blind test assured sample and submitter anonymity so only a summary of the results are presented here. In addition to the laser resistance results, details of deposition processes, coating materials, and layer count will also be shared.

Keywords: damage testing, mirror, thin film, multilayer, 1064 nm, 5 ns pulse length

1. INTRODUCTION

The Boulder Damage Symposium started 40 years ago. One way of celebrating this anniversary was the introduction of a laser damage competition. The purpose of this competition was to determine the current state-of-the-art of laser resistant IR laser mirror coatings and to serve as a benchmark for future comparison. Additionally, it was anticipated that the damage and thin film community would learn from the results thus providing a catalyst for future development activities. Finally, this was an opportunity to engage the private sector into a more active role in the BDS conference since often times engineers and scientists are unable to discuss corporate research in such a public setting.

2. PARTICIPATION

Thirty-five samples were submitted to this competition from twenty different companies or institutes listed in table 1. Up to two samples could be submitted by each participant. The participants came from five different countries as illustrated in figure 1. Half of the participants came from the United States, a third from Germany, and the balance from Lithuania and Asia.

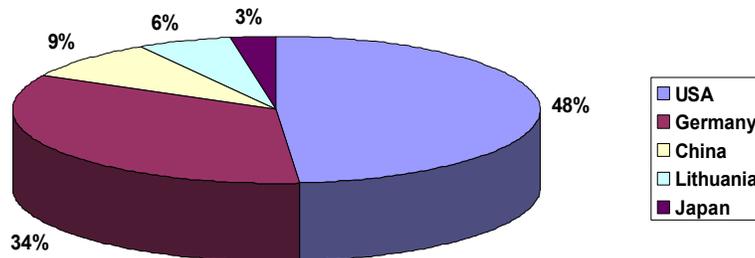


Fig. 1 Distribution of participating countries for the BDS thin film damage competition.

Table 1 List of participating companies or institutes for the BDS thin film damage competition.

Agilent Technologies	Advanced Thin Films	Berliner Glas KGaA
Institute of Optics and Electronics	Kugler	Laser Components
Laser Zentrum Hannover e.V.	Laserhof Frielingen GmbH	LaserOptik
Nikon	Optida	Photonics Products Group Inc.
Plymouth Grating	Quality Thin Films	Shanghai Institute of Optics and Fine Mechanics
TelAztec	Twin Star Optics	University of Rochester
VLOC		

3. SAMPLES

The spectral requirements were a reflectance greater than 99.5% at 1064 nm at normal incidence. Environmental requirements were ambient lab conditions (40% relative humidity and 20 degrees Celsius). There were no stress or reflected wavefront requirements. Substrates were participant supplied with dimensions of 50 mm in diameter and 10 mm thick. The substrate material was typically BK7 although a few samples (gold coated) were metallic (copper). Participants were asked to provide a spectral plot to validate spectral performance, a description of the coating process, a list of the coating materials, and finally the layer count. Some participants declined to provide all of the requested information. Based on input from conference attendees, future competitions will require that all requested information be provided or the samples will not be accepted and tested.

Samples were removed from participant supplied packaging containers and into identical PETG packaging containers in an attempt to remove any link to the supplier. Also for anonymity a unique color code was assigned to each sample. The identity of the suppliers and sample was kept by an administrative assistant to maintain a double blind experiment. The author and damage testing service did not have access to the identity of any of the samples so as to remain unbiased and to protect the identities of participants whose samples were the least laser resistant.

At least six different coating deposition techniques were used to manufacture the submitted samples as shown in figure 2. Unfortunately, participants declined to provide the deposition process for four of the samples. The majority of the samples were deposited by electron beam, although five samples were sputtered (either ion beam sputtering or magnetron sputtering). Some of the e-beam coatings were densified by either ion assistance or plasma assistance. To the author's knowledge, no samples were deposited by chemical vapor deposition, sol gel, laser deposition, or atomic layer deposition. Perhaps future competitions will benefit from sampling these deposition technologies. A gold coating and enhanced gold coating were submitted so it is likely that at least the gold layers were thermally evaporated. One e-beam and one ion assisted sample were etched with a grating to increase the high reflectivity. One of these samples had a lower reflectivity than the 99.5% reflectivity due to improper centering.

At least five different coating materials were used to manufacture the samples. Silica was the low index material of choice. The high index materials included hafnia, tantalum, gold, and zinc sulfide as illustrated in figure 3. Participants declined to report the high index material for ten of the samples. The gratings were imprinted on a tantalum and a zinc selenide sample. Unfortunately no scandium, zirconium, or alumina materials were confirmed although these materials (or others) may have been used on the proprietary samples.

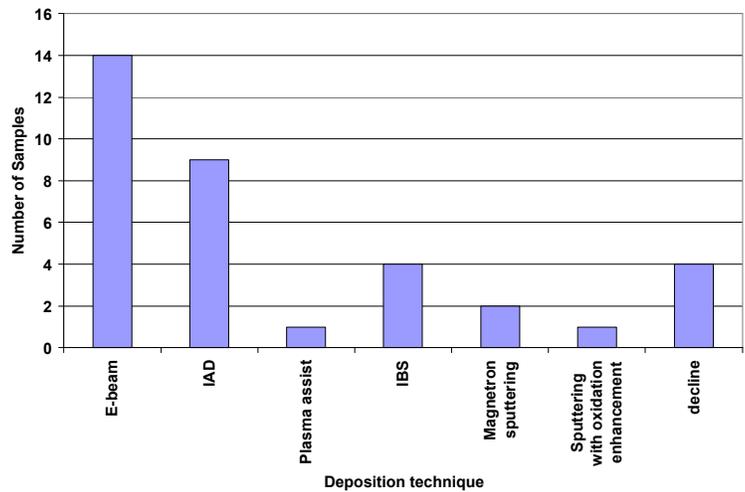


Fig. 2 Distribution of deposition technologies for the contributed samples.

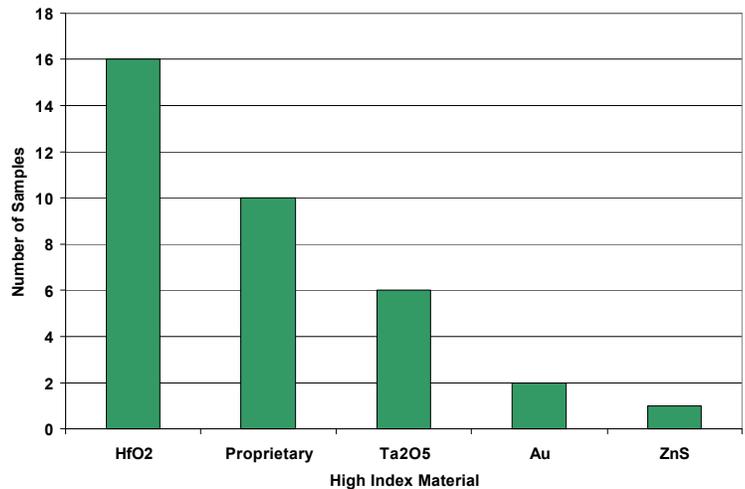


Fig. 3 Distribution of high index materials for the contributed samples.

information was not provided for it could help identify deposition technologies that could benefit from further optimization.

From a no damage perspective, the coating with the higher laser resistance was deposited by e-beam. For this particular sample a plasma etch was used to clean the sample before deposition. The details of this process were not provided, although given how well this sample performed, it will likely create significant interest within the thin film community and hopefully lead to future publications. It is possible that defects are ejecting from this sample, but may be undetected because the pinpoints do not scatter more light than before ejection. Although unproven, it is likely that the plasma etch increases the adhesion of the multilayer to the substrate which could lead to smaller ejection sites. Comparison of the irradiated and non-irradiated section of the sample could provide insight into why the sample did so well, however to protect company proprietary information no microscopy analysis occurred.

Coatings deposited by e-beam have the highest laser resistance in both figures. However, the top 10% of the coatings in figure 5 were deposited by e-beam, IAD and IBS indicating that a number of deposition techniques offer promise for producing high quality laser resistant coatings for the tested parameters. The grating technology which reported extremely high thresholds for an antireflection surface at the 2007 BDS conference⁵ performed poorly in this competition. Very little development occurred before these samples were manufactured. Process optimization such as selection of different coating materials as illustrated in figure 7 or imprinting in a thick overcoat previously discussed for

compression gratings⁶⁻⁸ might help produce higher laser resistance. A significant advantage of this technique is the low layer count which can't support large inclusions and only very small nodular defects. Hopefully as this technology matures, advances are reported.

Hafnia is clearly the most laser resistant high index material for the coatings that were submitted as illustrated in figure 7. Unfortunately a large number of participants declined to share information about their coating materials thus denying readers an opportunity to learn both materials that perform well or poorly. As expected, oxide materials clearly performed better than metallic films for the test pulse length and wavelength of this competition. The second most popular high index material in this study is tantala which clearly had an average lower laser resistance. Although tantala films generally have less scatter and fewer defects than hafnia coatings, it is more challenging to produce fully stoichiometric films. It is the author's experience that high laser resistant tantala coatings can be manufactured, although the process is significantly more difficult to develop than for hafnia films.

The final parameter that was explored in this study was evaluation of the impact of overcoats on laser resistance. Typically the first layer in a high reflector coating would be the high index to take advantage of the reflectivity achieved by large contrast in refractive indices. For the same reason high reflectors based on quarter-wave designs end with the high index material leading to an odd number of layers. It has been shown that the laser resistance of high reflector

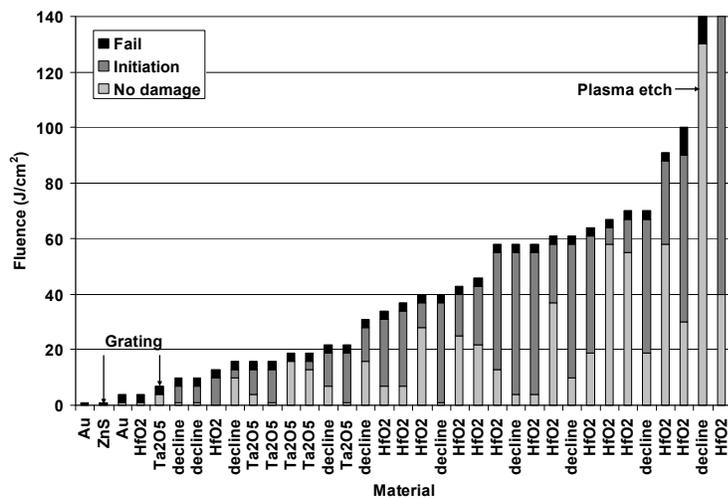


Fig. 7 Impact of high index material on laser resistance of submitted samples sorted by damage initiation.

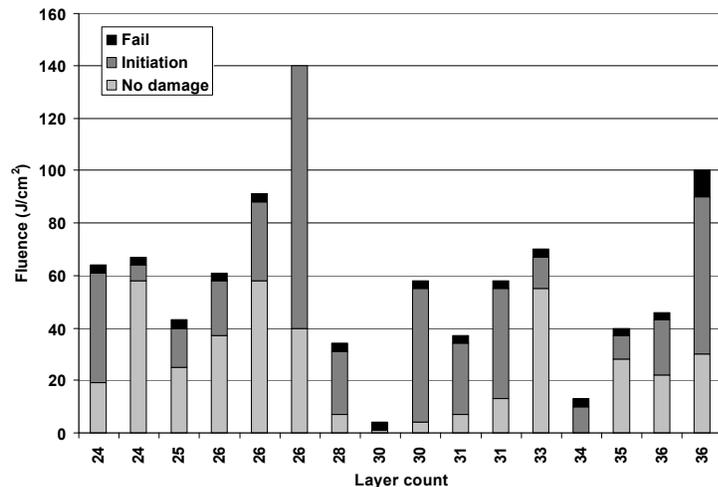


Fig. 8 Impact of layer count on the laser resistance of submitted samples containing hafnia as the high index material.

coatings can be increased with an overcoat of a low index material.⁹⁻¹⁰ Typically overcoats are half-wave in optical thickness so are optically absentee meaning they don't reduce the reflectivity and would result in an even number of layers in the multilayer stack. With these assumptions, the data was analyzed by layer count to see if a pattern would

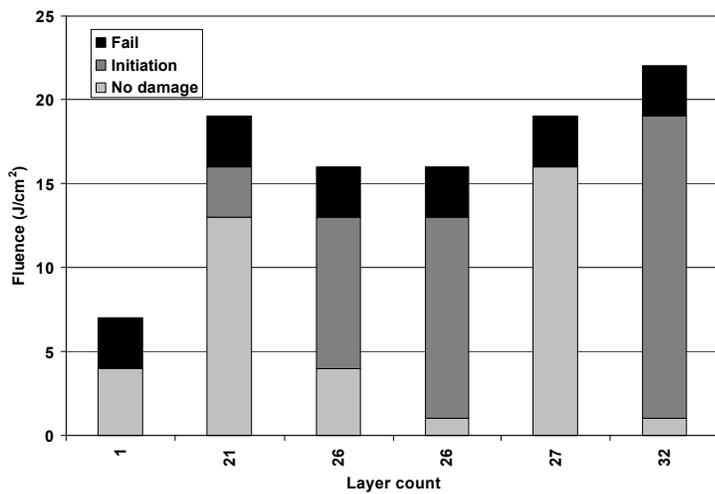


Fig. 9 Impact of layer count on the laser resistance of submitted samples containing tantala as the high index material.

emerge with respect to an odd versus even number of layers. No cross sections were made of the coatings to quantitatively determine the actual presence of overcoats and their respective physical thicknesses to protect the proprietary designs of each participant.

Figures 8 and 9 illustrate the impact of the layer count on the mirror laser resistance for hafnia or tantala based coatings respectively. Coatings with hafnia tend to perform better on average with an even number of layers indicating that overcoats may be helpful. Multilayer coatings with tantala tend to perform the same for both even and odd layer counts indicating for this material overcoats make little difference. For both high index material multilayer coatings, it does not appear that there are any strong trends of laser resistance with respect to fewer or a greater number of layers.

5. CONCLUSIONS

There is a significant range in laser resistance of 1064 nm (5 ns pulse length) high reflector coatings across the thin film community. Electron-beam deposited coatings tend to have the highest laser resistance, however Ion Assisted Deposition and Ion Beam Sputtering also performed extremely well. Hafnia based reflectors tended to be the most laser resistant. A plasma etch before deposition appears to have a very positive impact on laser resistance. Overcoats tend to increase the laser resistance for hafnia based multilayers, but a similar trend was not observed for tantala based coatings. Metallic coatings had poor laser resistance. Gratings also had poor laser resistance however, the results of other published work suggests development would lead to improvement.

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REFERENCES

1. The Essential Macleod, Thin Film Center Inc. www.thinfilmcenter.com
2. M. R. Borden, J. A. Folta, C. J. Stolz, J. R. Taylor, J. E. Wolfe, A. J. Griffin, and M. D. Thomas, "Improved method for laser damage testing coated optics," in *Laser-Induced Damage in Optical Materials: 2005*, G. J. Exarhos, A. H. Guenther, K. L. Lewis, D. Ristau, M. J. Soileau, and C. J. Stolz, eds., Proc. SPIE 5991, 59912A-1-8 (2006).
3. H. Bercegol, "What is laser conditioning? A review focused on dielectric multilayers," in *Laser-Induced Damage in Optical Materials: 1998*, G. J. Exarhos, A. H. Guenther, M. R. Kozlowski, K. L. Lewis, and M. J. Soileau, eds., Proc. SPIE 3578, 421-425 (1999).
4. A. B. Papandrew, C. J. Stolz, Z. L. Wu, G. E. Loomis, S. Falabella, "Laser conditioning characterization and damage threshold prediction of hafnia/silica multilayer mirrors by photothermal microscopy," in *Laser-Induced Damage in Optical Materials: 2000*, G. J. Exarhos, A. H. Guenther, M. R. Kozlowski, K. L. Lewis, and M. J. Soileau, eds., Proc. SPIE 4347, 53-61 (2001).

5. D. S. Hobbs and B. D. MacLeod, "High laser damage threshold surface relief micro-structures for anti-reflection applications," in *Laser-Induced Damage in Optical Materials: 2007*, G. J. Exarhos, A. H. Guenther, K. L. Lewis, D. Ristau, M. J. Soileau, and C. J. Stolz, eds., Proc. SPIE 6720, 67200L-1-10 (2008).
6. H. T. Nguyen, J. A. Britten, T. C. Carlson, J. D. Nissen, L. J. Summers, C. R. Hoaglan, M. D. Aasen, J. E. Peterson, and I. Jovanovic, "Gratings for high-energy petawatt lasers," in *Laser-Induced Damage in Optical Materials: 2005*, G. J. Exarhos, A. H. Guenther, K. L. Lewis, D. Ristau, M. J. Soileau, and C. J. Stolz, eds., Proc. SPIE 5991, 5991M-1 – 1M-7 (2006).
7. G. Razé, J. Néauport, G. Dupuy, M. Balas, G. Mennerat, E. Lavastre, "Short pulse laser damage measurements of pulse compression gratings for petawatt laser," in *Laser-Induced Damage in Optical Materials: 2007*, G. J. Exarhos, A. H. Guenther, K. L. Lewis, D. Ristau, M. J. Soileau, and C. J. Stolz, eds., Proc. SPIE 6720, 67200Z-1-9 (2008).
8. J. Keck, J. B. Oliver, T. J. Kessler, H. Huang, J. Barone, J. Hettrick, A. L. Rigatti, T. Hoover, K. L. Marshall, A. W. Schmid, A. Kozlov, and T. Z. Kosc, "Manufacture and development of multilayer diffraction gratings," in *Laser-Induced Damage in Optical Materials: 2005*, G. J. Exarhos, A. H. Guenther, K. L. Lewis, D. Ristau, M. J. Soileau, and C. J. Stolz, eds., Proc. SPIE 5991, 59911G-1-6 (2006).
9. C. C. Walton, F. Y. Génin, R. Chow, M. R. Kozlowski, G. E. Loomis, and E. Pierce, "Effect of silica overlayers on laser damage of HfO₂ – SiO₂ 56 degree incident high reflectors," in *Laser-Induced Damage in Optical Materials: 1995*, G. J. Exarhos, A. H. Guenther, M. R. Kozlowski, K. L. Lewis, and M. J. Soileau, eds., Proc. SPIE 2714, 550-558 (1996).
10. C. K. Carniglia, J. H. Apfel, T. H. Allen, T. A. Tuttle, W. H. Lowdermilk, D. Milam, and F. Rainer, "Recent damage results on silica/tatania reflectors at 1 micron," in *Laser-Induced Damage in Optical Materials: 1979*, H. E. Bennett, A. J. Glass, A. H. Guenther, and B. E. Newnam, eds., NBS SP 568, 377-390.