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GRAZING INCIDENCE ABSORPTION MEASUREMENTS

Final Report

Submitted to
UNIVERSITY OF CALIFORNIA
LOS ALAMOS NATIONAL LABORATORY

by
SPECTRA TECHNOLOGY, INC

MASTER

DISCLAIMER

31 July 1989

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9-X18-8599Q-1

Submitted to

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31 July 1989

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Section 1

EXECUTIVE SUMMARY

This final report summarizes the results of a series of different measurements to characterize various mirrors of interest to Los Alamos National Laboratory (LANL) and the Boeing Aerospace FEL program. Originally the work was part of a different contract sponsored by U.S. Army Strategic Defense Command through the Boeing Aerospace Corporation (Contract No. GL1065). Later, it was separated into its own program. Because the nature of the work during the two contracts was part of an ongoing program, for completeness we have included in this report results from Contract No. GL1065 (see Appendix A).

Most of the measurements were of the glancing incidence characteristics of different overcoated silver mirrors. These included the absolute absorptance as a function of angle of incidence ($0 - 88^\circ$), laser wavelength (0.5145 and $1.06 \mu\text{m}$), polarization, and exposure to room air. The latter measurement examined the ability of the overcoated silver mirrors to resist tarnishing. The major accomplishments of the program are listed below:

1. The glancing incidence characteristics of single-layer ThF_4 and MgF_2 overcoated silver mirrors were measured. Besides measuring the basic characteristics listed in the previous paragraph, the mirror characteristics were also compared to thin film theory.
2. The glancing incidence characteristics of bare silver mirrors overcoated with a very thin layer (10 \AA) of alumina (Al_2O_3) were measured. In addition to measuring the basic characteristics and tarnishing resistance, the performance of mirrors coated by two different facilities were examined.

3. The glancing incidence characteristics of diamond overcoated silver mirrors were measured.

The key results of this program were presented at two scientific conferences and the papers were published in the following references.

1. W. D. Kimura, "Performance of Overcoated Mirrors Designed for Glancing Incidence Operation," *Mirrors and Windows for High Power/High Energy Laser Systems*, C. A. Klein, Editor, Proc. SPIE 1047, 10-22 (1989).
2. W. D. Kimura, Q. D. Appert, P. N. Arendt, V. E. Sanders, and M. L. Scott, "Tarnishing Measurements of Al_2O_3 Overcoated Silver Mirrors," to be published in *Laser Induced Damage in Optical Materials: 1988*, Natl. Inst. Stand. and Tech.

Since these preceding papers describe the results of this program in a complete and succinct manner, the bulk of this final report consists of copies of these two papers in Appendix A and B, respectively. The next section describes the background and summarizes the results of this program. The last section presents the conclusions and recommendations of this work. A key point in the recommendations is that the alumina overcoated mirrors show great promise, but further work is needed to address critical issues such as why the alumina works and how to achieve reliable results. Appendix C contains a reprint of a paper that describes the experimental measurement system.

Section 2

INTRODUCTION AND SUMMARY OF RESULTS

2.1 BACKGROUND

High power free electron lasers (FEL) present a difficult challenge to optics because of the very high laser flux produced by these devices. This is because the output from an FEL is in a small diameter beam comparable to the size of the electron beam inside the wiggler magnet. The high radiation environment and generation of VUV light from the wiggler system place additional strains on the optical components, especially the dielectric coatings.

One method to combat these problems is to use glancing (or grazing) incidence mirrors. This helps increase the damage threshold of the mirrors by two ways. First, the absorptance of the mirror decreases by the cosine of the incident angle (for s-polarized light); and, second, at glancing incidence the laser footprint on the mirror increases dramatically thereby distributing the laser power over a greater area on the mirror (i.e. reducing the flux on the mirror).

Another factor that must be considered is the mirror materials. Since silicon can be fabricated in large sizes, can be polished to high quality, and has good thermal conductivity, it is typically the prime choice as the mirror substrate. Selection of mirror coatings depends on several factors, the prime one being the laser wavelength. For the application of interest in this program, the wavelength is $1.06\ \mu\text{m}$ (although, as explained later, measurements are also performed at $0.5145\ \mu\text{m}$). Metals, in particular, silver have excellent reflectivity at $1.06\ \mu\text{m}$. This is fortunate because metals coatings also tend to be less susceptible than dielectrics to damage by radiation and VUV light generation, and metal coatings tend to have higher laser damage thresholds compared to dielectrics.

One potential problem with bare silver coatings is that they are very susceptible to tarnishing once exposed to room air. Silver sulfide (Ag_2S) forms on the mirror surface and significantly degrades the reflectivity of the mirror. In the FEL environment, the resonator mirrors will normally be in a vacuum. Hence, tarnishing would not be a problem. However, the mirrors must periodically be exposed to room air (e.g. for initial installation and maintenance), and although steps can be taken to minimize the exposure time to air, since tarnishing appears to start immediately upon exposure to air, it is not clear how much tarnishing can be tolerated especially at the high laser fluxes anticipated.

The ideal situation would be to somehow protect the silver surface from susceptibility to tarnishing without compromising its high damage threshold characteristics. Typically, thick dielectric coatings (of order 1000's of angstroms) are applied to the bare silver surface to prevent tarnishing. This solution to the tarnishing problem introduces two new problems. First, the damage threshold of dielectric coatings tend to be worse than bare metals. And, second, as shown in Appendix A, the reflectivity properties of mirrors at glancing incidence are very sensitive to the thickness of the dielectric coating. Indeed, in general the reflectivity of the mirror at high angles of incidence with a thick dielectric coating will not be as high as it is when just a bare metal.

2.2 GOALS OF PROGRAM

Given the preceding background, the goals of this program were to investigate the glancing incidence properties of various overcoated silver mirrors in order to understand how the overcoat affects the mirror performance and how effectively it stops tarnishing. In the process of this investigation, a novel coating developed at LANL was tested. It shows great promise in solving the tarnishing problem without creating the new ones mentioned earlier (see Appendix B).

The primary diagnostic for this program is a system for measuring the absolute absorptance of a mirror as a function of angle of incidence and polarization of the laser light. The system is based upon photoacoustic calorimetry and it is described in detail in a paper given in Appendix C. It is capable of detecting the very small amount of energy deposited on the mirror surface at glancing angles of incidence.

Three different general techniques for overcoating the silver mirrors were examined. The first was conventional overcoating of bare silver mirrors using thick dielectric coatings. The second was the novel, ultrathin alumina overcoat developed by LANL. Lastly, some basic measurements (but not tarnishing) were performed on diamond overcoated silver mirrors. These measurements are discussed in the next sections.

2.3 THICK DIELECTRIC OVERCOATED SILVER MIRRORS

This is the work sponsored by the U.S. Army Strategic Defense Command through the Boeing Aerospace Corporation (Contract No. GL1065). The description of the measurements and the results are given in Appendix A.

Summarizing the experiment and results, bare silver mirrors were overcoated by either ThF_4 and MgF_2 at various thicknesses varying from 550 to 2460 Å. These thicknesses were selected using thin film theory to optimize the reflectivity properties of the mirror at glancing incidence. For example, the thicknesses were chosen to minimize the absorptance at 85° for either s-polarization, p-polarization, or both polarizations simultaneously.

The glancing incidence properties of these overcoated mirrors were measured using the photoacoustic calorimetry system and compared with the predictions from thin film theory. For the ThF_4 overcoated mirrors, the agreement between theory and measurements is quite good. For the MgF_2 overcoated mirrors, the agreement is not as good implying that the values of parameters for the dielectric (such as the index of refraction) may not

be as expected or that the overcoated surface of the mirror is not pure MgF_2 .

These overcoated mirrors were also exposed to room air for extended periods of time (up to 2 months). All the mirrors, with the exception of one, did not show any signs of tarnishing during this period. The one exception appeared to show signs of tarnishing, but its absorptance characteristics were not consistent with normal tarnishing behavior.

2.4 THIN ALUMINA OVERCOATED SILVER MIRRORS

P. N. Arendt and M. L. Scott of LANL developed a novel alumina overcoat that appeared to protect bare silver mirrors from tarnishing. What is novel about the overcoat is that the alumina is only 10 Å thick. Although the precise mechanism that prevents the formation of silver sulfide on the mirror surface is still not known, it is certain that the 10 Å thick alumina overcoat is much too thin to cover the entire silver surface as is the case for thick dielectric overcoats. This implies that the alumina must be somehow protecting the silver surface via some process other than simply covering the surface. One possibility is that the alumina molecules are selectively attaching to the same sites on the surface as the sulfur molecules would be.

The reasons why the thin alumina overcoat appears to be protecting the silver are intriguing; however, the primary purpose for testing these mirrors was to: 1) Determine the effect of the alumina overcoat on the glancing incidence properties of the mirrors; and 2) to verify that the alumina overcoat did indeed prevent tarnishing. (Up until this study the tarnishing resistance of the alumina overcoated mirrors had only been qualitatively observed by exposing the mirrors to fuming ammonium sulfide.) The details of the measurements and results are given in the paper in Appendix B.

Summarizing the results, the alumina overcoat does not appreciably affect the glancing incidence characteristics of the mirror. In other words, the mirror behaves as if it were a bare silver mirror probably because the alumina overcoat is too thin to affect the $0.5\ \mu\text{m}$ laser light. (Tests by LANL also indicate that the alumina overcoated mirrors have comparable laser damage thresholds as bare silver mirrors.)

With regard to tarnishing, two different sets of mirrors coated the same way by either LANL or Spectra Physics Optics Division were exposed to room air for an extended period of time (maximum 170 days). The results indicate that the protective nature of the thin alumina overcoat is not consistent between coaters. The overcoat also does not appear to completely stop the onset of tarnishing; although, it does greatly retard its formation, which makes it still much better than bare silver.

2.5 DIAMOND OVERCOATED SILVER MIRRORS

The last set of measurements were a small scale effort to perform a limited amount of tests on diamond overcoated silver mirror samples. The absolute absorptance was measured using the photoacoustic calorimetry system and a similar procedure as the previous overcoated mirrors.

At $1.06\ \mu\text{m}$, the absorptance of the diamond overcoated mirror is: $2.2 \pm 0.3\%$ at normal incidence and $0.12 \pm 0.03\%$ at 88° angle of incidence (s-polarization). At $0.5145\ \mu\text{m}$, the absorptance is: $6.1 \pm 0.2\%$ normal incidence and $0.27 \pm 0.01\%$ at 88° angle of incidence (s-polarization). Assuming normal Fresnel reflection scaling applies, the absorptance at 88° should have decreased by a factor of 0.035. Hence, the absorptance at 88° for the $1.06\ \mu\text{m}$ case is approximately 56% higher than what would be theoretically predicted and for the $0.5145\ \mu\text{m}$ case the absorptance is roughly 27% higher than the ideal case.

There are many possible reasons why the absorptance is not scaling with angle as it should for a bare metal surface. The diamond layer

thickness is nominally 200 Å, which may affect the glancing incidence properties of the mirror. There may be imperfections in the coating that could adversely affect the mirror reflectivity when operated at high angles of incidence. There was not time during the program to investigate these and other possibilities; hence, the cause of the increased absorptance at high angles of incidence is not known.

There was also not enough funds available to perform any tarnishing measurements of the diamond overcoated silver mirrors.

Section 3

CONCLUSIONS

As a result of the work performed during this program, significant progress has been made to find a better alternative to bare silver mirrors for high power laser applications. Thick dielectric overcoats do protect the silver mirrors from tarnishing, but problems associated with accurately controlling the optical constants of the dielectric during the coating process appear to make it difficult to achieve the desired optical properties of the mirrors at high angles of incidence. Although the absorptance at high angles of incidence can in principle be made comparable to its value for a bare metal surface by appropriately selecting the coating thickness, the issue of the lower damage threshold of dielectrics relative to bare metals must still be addressed. In certain applications, the decrease of damage threshold may be acceptable, thereby making a thick dielectric overcoat a satisfactory alternative to bare silver.

The alumina overcoated silver mirrors show the potential of overcoming all the undesirable shortcomings of dielectric overcoated silver mirrors while at the same time eliminating or at least significantly reducing the susceptibility to tarnishing. (As an addendum to the paper given in Appendix B, at the time of this writing, the alumina overcoated mirrors have been exposed to air for over a year. The LANL overcoated mirror now shows visible signs of tarnishing. A white "fog" covers the central portion of the mirror surface similar to the fog that is observed when the bare silver mirrors tarnish. The Spectra Physics coated mirror still looks very good; however, with very close visual examination of the mirror surface it is possible to see the faint signs of tarnishing beginning to appear. Hence, neither overcoated mirrors has entirely stopped the onset of tarnishing. Compared to bare silver mirrors, however, the alumina overcoated mirrors are vastly superior in their resistance to tarnishing.)

Despite the very good performance of the alumina overcoated mirrors, it is clear that several important issues still need to be addressed. The first is understanding why the thin alumina layer seems to retard the formation of tarnish. The precise mechanism is not well understood and optimization of this technique is made much more difficult by this lack of understanding.

This understanding may be also very important for the second issue which is the problem of reliably reproducing the protective properties of the overcoat. The LANL and Spectra Physics mirrors were supposedly coated using the same procedure, but the tarnishing resistance of the mirrors is dramatically different between the two sets of mirrors. It is not known why the mirrors behave differently.

Additional issues originate from discussions with others in the optics community. The thin alumina overcoat may be a step in the right direction, but it may not be the complete answer to the tarnishing problem. For example, tarnishing may be also occurring underneath the silver coating (perhaps between the silver and chromium underlayer). If this were true, then perhaps an alumina underlayer may help.

Another possibility is that the surface morphology changes over time. In the beginning, the alumina may attach to silver sites that are more susceptible to attack by sulfur. After a while, though, those silver sites, which did not have alumina attached to them because their morphology was such that sulfur would not readily attach to them, change so that they are now susceptible to sulfur attack. The solution to this problem might be to recoat the surface with thin alumina after a certain period of time.

Finally, there is the question of whether alumina is the best material. There may be other dielectrics that are better than alumina. However, without understanding why alumina protects the silver surface, it will be a trial and error process to find any better materials.

If this technique is to be developed into a useful procedure, these preceding issues must be addressed. Therefore, it is the recommendation of this study that additional effort be made to investigate these issues. The potential benefits of this technique reach beyond high power laser applications. Areas such as telescopes for astronomy and remote sensing, and applications where the optical properties of silver are desired could be helped. In addition, the basic mechanism behind the protective properties of the alumina (once it is understood) may be applicable for protecting other types of metal surfaces.

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