Gold coatings for cube-corner retro-reflectors

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ABSTRACT

The Space Interferometry Mission (SIM) PlanetQuest is managed by the Jet Propulsion Laboratory for the National Aeronautics and Space Administration. SIM requires, among other things, high precision double cube-corner retro-reflectors. A test device has recently been fabricated for this project with demanding specifications on the optical surfaces and gold reflective coatings. Several gold deposition techniques were examined to meet the stringent specifications on uniformity, optical properties, micro-roughness and surface quality. We report on a comparative study of optical performance of gold films deposited by resistive and e-beam evaporation, including measurements of the scattering from the coated surfaces. The effects of oxygen bombardment and titanium under-layer on optical properties and adhesion were evaluated. The influence of surface preparation on the optical properties was examined also.

Keywords: Thin films, gold coatings, reflectors, scattering, uniformity

1. INTRODUCTION

Double cube-corner (DCC) retro-reflectors for the brass-board stage of NASA's Space Interferometry Mission were manufactured recently at CSIRO Industrial Physics, Australia, for the Jet Propulsion Laboratory, Pasadena, CA. These reflectors are very high precision optical assemblies made of Zerodur. Producing such an optical assembly is a complex task exacerbated by the very demanding specifications. For example, dihedral angle errors below 0.5 arcsec, reflective faces flat to ~ 10 nm peak-to-valley and vertex co-location errors less than 0.01 mm are required. Each reflector consists of three wedged prisms optically contacted to a cylindrical base plate. All reflective surfaces are coated with ultra-pure gold also with demanding specifications on uniformity, reproducibility of optical properties (optical constants non-uniformity < 2%), micro-roughness (< 0.5 nm rms), and surface quality.

Gold reflectors are widely used in a variety of industrial and research applications. The chemical inertness of gold makes it a very attractive choice for high quality mirrors which require stable optical properties over a long period. Apart from chemical stability another advantage for the use of gold is that it is a well researched material, which can be deposited by a number of established techniques[1-4]. Despite the fact that gold deposition is well documented in the literature a detailed investigation was necessary to meet very stringent customer requirements on uniformity, reproducibility of optical properties, micro-roughness and final surface figure on coated surfaces. In addition, the films have to be durable enough to satisfy the needs of other DCC production steps (such as photo-resist application and removal, gold etch, manual handling during assembling, testing, etc).

We concentrated our efforts on investigating the limitations at each step of the production process in order to obtain optimum performance of the multi-component optical assembly (shown in Fig.1). The main emphasis of the study was to evaluate the suitability of e-beam and resistive thermal evaporation and ion-assisted evaporation for depositing gold films with the required specifications. We report on the role of the process parameters, such as deposition rate, ion

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bombardment, choice of coating structure (1 or 2 layers), effect of surface micro-roughness, the influence of substrate preparation and cleaning techniques in achieving the specified performance of the DCC.

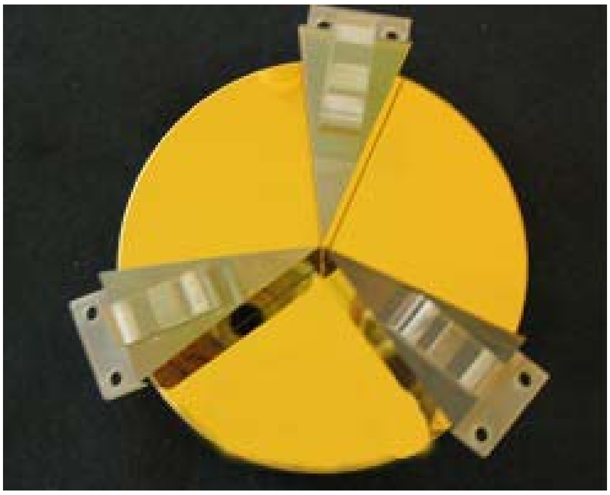


Figure1. Assembled DCC consisting of three wedged prisms bonded to a flat baseplate. Only two of the corner retro-reflectors needed to be certified to demanding requirements.

2. EXPERIMENTAL DETAILS AND PROCEDURES

Evaporated gold films were grown in a conventional, cryo-pumped vacuum deposition system of in-house construction equipped with e-beam and resistive thermal evaporation sources and an end-hall style ion gun for use in ion-assisted depositions (IAD). Alumina-coated tungsten boats were used for resistive evaporation. These boats improved the run-to-run reproducibility. To obtain the required film uniformity (less than 0.2% variation in film thickness over a 125mm diameter substrate) the system was equipped with a specially designed rotating shutter.

To achieve the desired performance, the gold films must adhere well to a Zerodur surface. Unfortunately, ordinary evaporated gold does not generally adhere well to oxide surfaces. Sputtering, instead of evaporation, on a rough surface improves adhesion, but also results in a rougher film surface. A widespread industrial practice to improve the adhesion of the gold is to deposit an intermediate thin metal undercoating, Ti and Cr being the two most frequently used metals. They form a covalently bonded metal-oxide mixture at the substrate interface and form an alloy with gold at the other interface which improves the adherence[5]. All double layer films presented in this paper contained ~10nm of Ti as an under-layer. Substrates were pre-cleaned prior to deposition by argon ion-bombardment for 10 seconds. The Ti under-layer was deposited by e-beam evaporation at a rate ~1-2 Å/s without ion bombardment. The gold was then either e-beam or resistively evaporated at a rate ~6-8Å/s, also without ion bombardment. A quartz crystal thickness monitor was used to measure the deposition rate and film thickness. The thickness was verified by ex-situ transmittance measurements performed in a Varian Cary-5 spectrophotometer.

Another known method to improve the adhesion of gold films is the use of IAD. Ion bombardment of a growing film is known to enhance nucleation, reduce the coalescence thickness and thus improve the Au film adhesion[7-8]. Both resistive and e-beam evaporated single layer films discussed in this article were prepared with the use of oxygen ion bombardment.

In ion-assisted evaporation runs, substrates were pre-cleaned by oxygen ion bombardment for 10 seconds. The first 10 nm of gold film was deposited slowly at a rate below ~1Å/s. The growing film was bombarded with ~200eV oxygen ions. The remainder of the gold was deposited at a much higher rate ~6-8Å/s, without ion bombardment, to ensure high reflectivity. Real-time, in-situ monitoring of the optical properties was carried out using a single wavelength (633nm) rotating polariser type ellipsometer constructed in-house[6]. Real-time ellipsometry was used to determine the point of coalescence of the gold film, where the measured ellipsometric data intersects with the calculated theoretical curve for bulk gold (see Fig. 2). Measurements are represented in terms of ellipsometric angles Del and Psi.

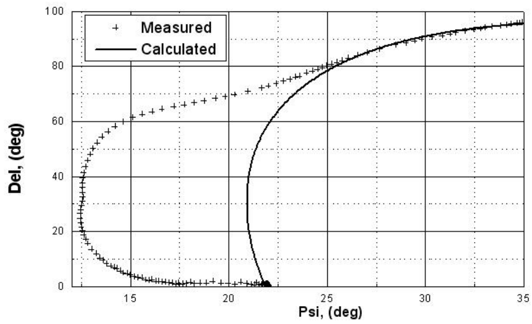


Figure 2. Typical in-situ ellipsometric monitoring curve for gold evaporation during film coalescence.

All tests and real coatings were carried out on optically polished Zerodur substrates. High purity (99.99%) gold was used in all deposition runs. Typical thicknesses of the films were in the 100–150nm range. Film adhesion was verified by tape peel-off and scratch testing.

A non-contact optical surface profiler (WYKO TOPO-3D) was employed to measure the surface roughness of the coated and uncoated surfaces. All measurements presented in this paper were performed using the high (x40) magnification objective to enable the roughness to be determined at the finest spatial resolution.

A simple scatterometer was also constructed and used in scattering and surface roughness measurements on the gold coated surfaces. A schematic diagram of the device is shown in Fig. 3. An obliquely incident 4mW He-Ne laser illuminates a small spot on the surface which is then imaged with a microscope through an x5 objective lens. This dark field (scattered) image is recorded on a camera or the integrated scattered intensity measured with a photo-multiplier tube. An estimate of the scattering can be obtained by comparison of the sample signal with that from a calibrated white diffuse reference sample. The roughness can then be estimated from the scattering value. Measurements from the surface profiler and scatterometer did not agree exactly due to spatial frequency mismatch, but the general trend was similar in the majority of comparative experiments.

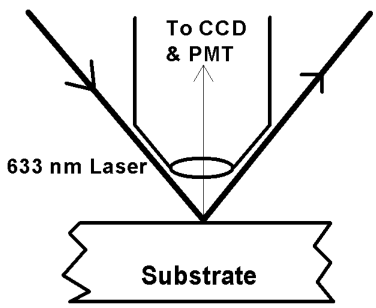


Figure 3. Schematic diagram of the scatterometer

Photolithographic processing was used to pattern the gold on the DCC baseplate (Fig. 4). This required application and removal of photoresist and chemical etching of the masked gold surfaces and removal of remnant photoresist and suitable cleaning. Photoresist was also used to protect particular surfaces of the wedge prisms from overspray during gold deposition and this also required removal and cleaning. The roughness of the bare Zerodur and gold coated surfaces was also examined following these treatments to ensure no degradation in roughness or scattering occurred and to determine the most suitable cleaning process.

Figure 4. DCC baseplate prepared for bonding of wedged prisms to form cube corners.

The efficiency of three types of surface cleaning processes was also investigated in this study:
1. Ultrasonic cleaning was tested on bare surfaces and gold coatings at room temperature. Substrates were held in a teflon jig in the ultrasonic bath for 15 to 30 minutes. The cleaning solution contained hi-purity de-ionized (DI) water and ~3% of a commercial neutral pH detergent. After ultrasonic cleaning the substrates were thoroughly rinsed in DI water and blow-dried with filtered ultra-high purity nitrogen.
2. Hand cleaning using a similar detergent solution was used to rub-clean the substrates. The components were soaked in warm (~50^{o}C) cleaning solution for 20 minutes then followed by vigorous hand rubbing using ultraclean polyurethane gloves soaked in detergent solution. Rinsing and drying was the same as for the ultrasonic clean.
3. Some experiments were performed using CO_2 snow cleaning of coated and uncoated surfaces. The surfaces were sprayed with high purity carbon dioxide from a nozzle[9] in a nitrogen atmosphere to reduce water condensation.

3. RESULTS AND DISCUSSION

3.1 Cleaning Methods

Scattering results show a significant effect of the cleaning method used. The ultrasonic method was tested on bare substrates and gold coatings. Uncoated substrates were cleaned in the ultrasonic bath for up to 30 minutes. The number of particles in the scattering image was significantly reduced, but not completely eliminated. Scattering measurements following a liquid hand clean, however, showed that most particles were removed. Ultrasonic cleaning, while not adding to the particulate contamination, isn’t able to remove all particles from the substrate effectively.

The surface roughness of a gold coating after 15 minutes ultrasonic cleaning increased from 2.5Å to 9.5Å (from scatterometer measurements). The result presented in Fig. 5 proved that ultrasonic does add imperfections and particles to a gold coating. It was determined that ultrasonic cleaning was not suitable for substrate or gold film cleaning in our case. Ultrasonic cleaning appears to add scattering centers and increase roughness (according to scatterometry). The procedure does not appear to alter the ellipsometric values, and so does not change gold’s optical properties.

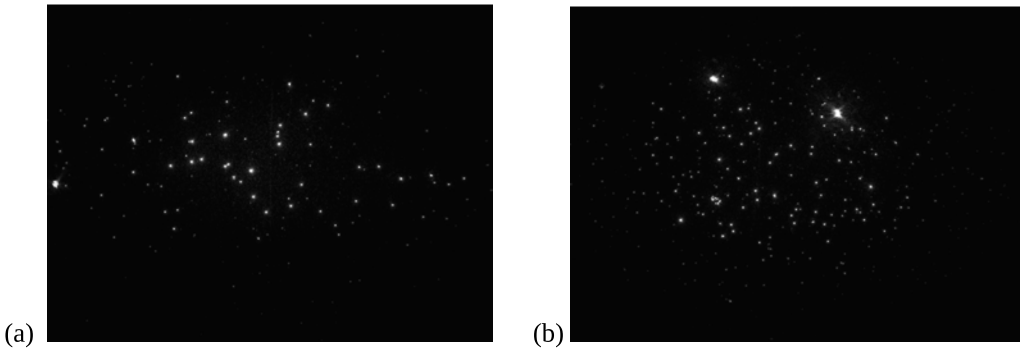


Figure 5. Dark field image of e-beam evaporated gold before (a) and after (b) 15 minute ultrasonic clean. The rms surface roughness of the film increased from 2.5Å (as deposited) to 9.5Å. The field of view is about 2 mm wide.

CO_2 snow cleaning of coated and uncoated surfaces did not produce satisfactory results, with considerable particulates being added to the surfaces (Fig. 6). This probably indicates the need for much greater cleanliness in the CO_2 delivery system; however, this made the method uneconomical for our purposes.

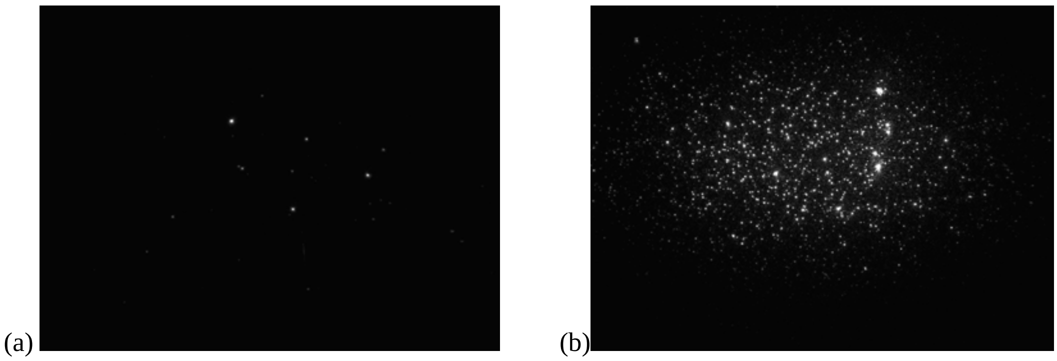


Figure 6. Dark field image of thermally evaporated gold before (a) and after (b) snow cleaning.

The best results were achieved in trials using hand cleaning in neutral pH detergents, no touching of the coated surfaces with solid material and blow-drying for all coating methods. Coatings do not appear to be adding roughness to samples.

We also investigated the effect on surface roughness and scatter when gold films were removed with acid or iodine etchants. The gold etching process does not add micro-roughness to the exposed Zerodur surface according to TOPO measurements, but particle addition is a problem. Hand cleaning, however, was found to be a satisfactory method to remove such particles.

The Ti coating in double layer films proved not to be affected by hand cleaning. The film shown in Fig. 7 was coated with a Ti/Au double layer, and then Au was removed. The Ti layer was liquid cleaned and over-coated with Au again. This resulted in roughness of 1.5Å by TOPO and 2.3Å by scatterometric measurements.

Figure 7. Darkfield image of a Ti/Au double layer sample with gold etched, the Ti layer then liquid cleaned and re-coated with Au.

3.2 Deposition Methods

Figure 8 shows darkfield images of two substrates coated in the same deposition run by thermal evaporation. Substrate A has 1.5Å rms roughness and Substrate B has 5Å rms roughness. However, the resulting coatings have similar appearance and roughness values (3.1Å and 3.2Å, respectively). Thus at a level below ~5Å rms, the substrate roughness does not determine the final roughness of the coated surface and hence a substrate roughness no better than 5Å is required in order to achieve the specified coating roughness.

The lowest scattering results (1.3Å) were obtained for resistively evaporated Au on super-polished substrates produced by Wave Precision, Inc. Scattering from polishing defects and particles is almost undetectable on these substrates (Fig. 9). Slightly more scatter was detected on in-house manufactured super-polished substrates. Thermal resistive evaporation gives the best roughness measurements for both single Au and double Ti/Au layer coatings. E-beam coatings produced approximately equivalent rms roughness figures but always had significantly higher particulates and defects than thermally evaporated films (compare Fig 5(a) with Fig. 8(a)). Substrates were cleaned using the liquid hand cleaning method only.

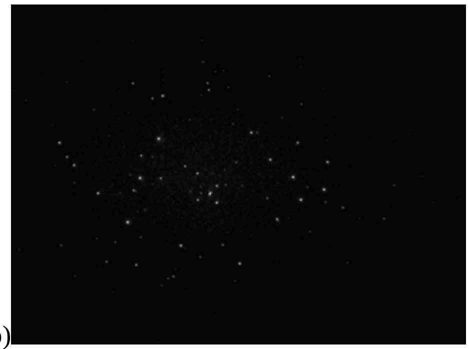
(a)  (b) 

Figure 8. Scattering measurements on thermal evaporated gold coated in the same deposition run. (Substrate A was polished to 1.5Å rms, substrate B to 5Å rms), Resulting coating roughness is 3.1Å for sample A and 3.2Å for sample B

Figure 9. Darkfield image of resistively evaporated Au on super-polished substrates produced by Wave Precision, Inc. The roughness is very low at 1.3Å rms and defects and particles are almost undetectable on these substrates.

Good adhesion results were achieved with all deposition methods investigated in this article. Both double layer e-beam evaporated gold (with an under-layer of Ti) and a gold Au film with O_2^+ IAD under-layer passed the tape adhesion and scratch test. Very good adhesion was achieved over a wide range of deposition conditions.

4. SUMMARY AND CONCLUSIONS

It is possible to produce Au films with good adhesion, optical properties, reproducibility and uniformity by all methods investigated in this work. We elected to use a single layer coating to simplify the photoresist patterning process. This is because removal of a titanium under–layer requires complex processing: it is not readily removed by chemical etchants without damaging the precision glass surface. Resistive evaporation resulted in slightly better results for both scattering and roughness measurements compared with e-beam evaporation. All components of the DCC retro-reflector were single layer gold coated by ion-assisted resistive evaporation.

Efficient cleaning of the substrates proved to be important and hand cleaning using neutral-pH detergents was found to be the safest and most effective cleaning method.

AKNOWLEDGEMENTS

The research described here was carried out by the Commonwealth Scientific and Industrial Research Organisation under contract with the Jet Propulsion Laboratory, California Institute of Technology, and was sponsored by the National Aeronautics and Space Administration.
The authors also wish to acknowledge helpful discussions with Dr J. Burke and Dr B. Oreb.

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