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Qualification and Acceptance testplan document for Yttrium Fluoride coating on Combining Prism in ILG

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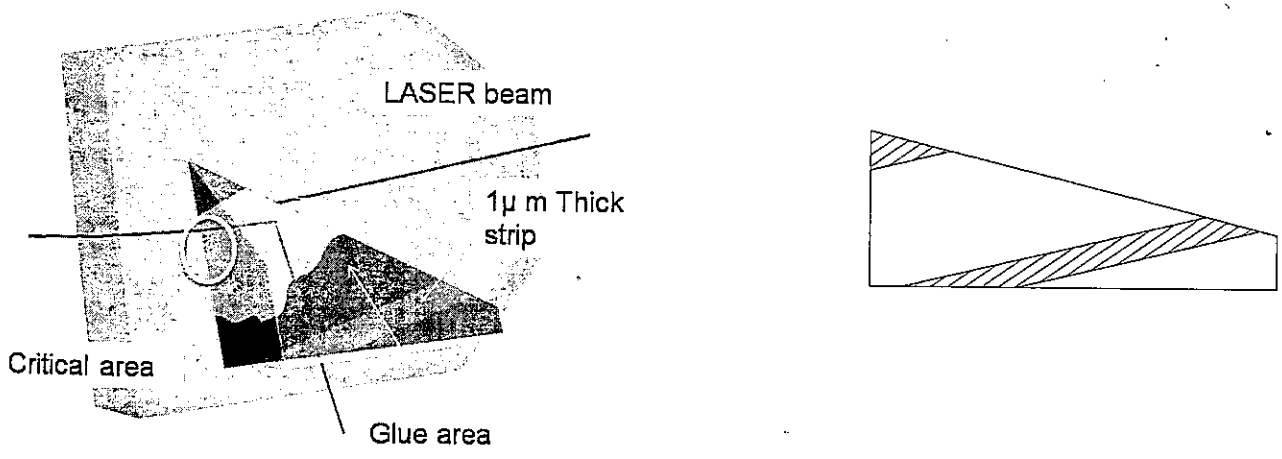
Abbreviations

ILG	ISRO Laser Gyro
CP	Combining Prism
TRP	Total Reflection prism
YF_3	Yttrium Fluoride

1. Introduction

Combining Prism (CP) is an optical component used in ILG for coupling of the two counter propagating beams inside cavity and provides an output fringe pattern which is guided to photodetector through a periscopic prism. A small portion of the two counter propagating beams is taken out through the phenomena of evanescent wave coupling, by keeping the combining prism at a gap of (1± 0.1μ) with respect to hypotenuous surface of one of the curved Total Reflecting Prism (TRP) as shown in figure 1(a) and position ILG design document[1]. This particular gap (1± 0.1μ) is very critical parameter for the performance of Sensor and is maintained by special two strip coatings of Yttrium Fluoride of thickness (1± 0.1μ) on Surface B of Combining prism[2] as shown in figure 1(b). The functional requirement of the coating is to maintain the above mentioned distance. So for proper operation of ILG, integrity of coating needs to be established and maintained during the operational life time of sensor. The scope of the document is to present a test procedure based on Military standard (MIL STD) and functional requirement of combining prism for coating qualification and acceptance.



(a) (b)

Fig.1: (a) Schematic representation of combining prism bonding of TRP (b) Schematic representation of coating strips on surface B of CP

2. Properties of Yttrium Fluoride

Yttrium fluoride, YF_3, gives low-index film layers that exhibit good transparency in the UV through infrared regions. It can be combined in multilayer's with other fluoride compounds and with higher-index ZnS and ZnSe layers for AR and filter coatings out to wavelengths >10 μm.

Yttrium Fluoride films can be deposited from the melt by resistance-heated or electron beam evaporation. Film density and refractive index increase with substrate temperature. Thicknesses greater than 2 μm will adhere to zinc sulfide, glass, and Germanium substrates heated to 250°C. Yttrium fluoride is insoluble, therefore making it suitable for humid conditions when applied hot. Amorphous films exhibiting low scatter can be deposited below 150°C substrate temperature, but adhesion and refractive index properties are compromised. Above ~250°C, the films become crystalline and harder, but they exhibit noticeable scatter and are more stressed. In our application, light is not interacting with coating, hence scattering and stress effects are not applicable but adhesion and coating thickness uniformity is of prime importance.

3. Coating Process Parameters

The coating is done by **Electron Beam Evaporation** method as per optimized coating process explained in separate document[3] [IISU/TR/1773/2016]. The main parameters of the coating are given below:

a) **Substrate :** Combining prism [Material: Fused silica]
b) **Substrate Temperature:** Temperature should be maintained at 200^0 C.
c) **Chamber Pressure: $5^* 10^{-6}$ mbar**
d) **Source Current:** Set at a value of 10 mA/A and to be monitored continuously.
e) **Coating Rate**: between (5-10 A˚ /sec)
f) **Target thickness: 1 μ** to be monitored at quartz crystal

4. Qualification Process

4.1 Tests for Coating process Qualification

The qualification of such coating when used as anti reflection coating comes under the scope of **MIL-C- 675C**[4] standard though our application is different. This standard establishes the durability requirements for such coating after completing the coating process. As discussed above, functional application of YF_3 coating is not as antireflection but to provide a step thickness of (1± 0.1μ). So all the qualification tests mentioned in **MIL-C- 675C** are not necessary. It is important to note that coating process is optimized after many trials by varying different parameters and once it is optimized, the parameters are frozen for further batches of coating. So it is necessary to first qualify the coating process. Thus, it is proposed to do following tests on **witness samples (Coupons) to qualify the coating process,** from the above standard along with some additional special tests which are necessary to access the optical quality and durability characteristics for Yttrium fluoride coating for qualification.

1) **Thickness Measurement:** It should be measured by non contact method (Talysurf CCI) and should conform the target thickness of (1± 0.1μ).

2) **Physical Inspection of Coating:** Coating should be inspected through reflected light under microscope with 20X magnification and it should conform to following points:

a) The coating shall be uniform in quality and no evidence of deterioration such as flaking, peeling, cracking or blistering. There shall be no visible discontinuities or blemishes in coated area. The edges of longer strip should be sharp.

b) Coating should not have any deep scratches and dig or pits.

3) **Acetone Cleanability:** Coated prism shall be cleaned about 25 times by cotton wad dipped in acetone. After that it should be inspected under microscope with 20X magnification and it should conform to points **2 (a) & 2 (b).**

4) **Adherence:** The coated component shall show no evidence of coating removal when cellophane tape is pressed firmly against the coated surface and quickly removed at an angle normal to the coated surface.

5) **Temperature Survivability:** Coated prism should be exposed to temperatures of 85°C in vacuum of 10^{-6} mbar for 5 hours and 8°C for 5 hours in thermal chamber. It

should be inspected under microscope with 20X magnification and it should conform to points **2 (a) & 2 (b).**

6) **Acetone Dipping:** Coated prism is dipped in acetone in a beaker and kept for full night (approx 16 hrs) and checked for acetone Cleanability as done in point 3. It should be inspected under microscope with 20X magnification and it should conform to points **2 (a) & 2 (b).** This test is specially done to check the survivability of coating as similar process is followed to remove the combining prisms during its rework in optical assembly of ILG. This test will ensure it reusability in the assembly.

7) **Durability in Humidity:** The coated witness piece should be placed in environmentally control chamber and exposed to a temperature of 45^0C and 95 to 100% relative humidity for a minimum of 24 hours subsequent to this, the exposed witness sample should be removed from the chamber and cleaned with acetone. After that, it should be tested for Acetone Cleanability. The coated surface should be inspected under microscope with 20X magnification and it should conform to points **2 (a) & 2 (b)** above. This test is similar to accelerated life test of coating due to storage.

4.2 Tests for Coating batch Qualification

Once coating process is qualified, it is not required to repeat all the tests done for qualification of coating process. So it is proposed to perform following tests on **witness samples to qualify a particular batch of coating,** performed with same optimized parameters as done in earlier batches.

I. **Thickness Measurement:** It should be measured by non contact method (Talysurf CCI) and should conform to the target thickness of ($1\pm 0.1\mu$).

II. **Physical Inspection of Coating:** Coating should be inspected through reflected light under microscope with 20X magnification and it should conform to following points:

a) The coating shall be uniform in quality and no evidence of deterioration such as flaking, peeling, cracking or blistering. There shall be no visible discontinuities or blemishes in coated area. The edges of longer strip should be sharp.

b) Coating should not have any deep scratches and dig or pits.

III. **Acetone Cleanability:** Coated prism shall be cleaned about 25 times by cotton wad dipped in acetone. After that it should be inspected under microscope with 20X magnification and it should conform to points **II (a) & II (b)** above.

IV. **Adherence:** The coated component shall show no evidence of coating removal when cellophane tape is pressed firmly against the coated surface and quickly removed at an angle normal to the coated surface.

4.3. Witness Sample: The above qualification tests should be done on witness pieces. **Minimum 2 witness samples should be taken for a coating batch of 10 jobs and 3 witness samples upto 15 jobs.** The coating fixture has a maximum capability of holding 12 jobs. The witness pieces shall be positioned in the coating chamber such that they represent the optical and durability characteristics of the whole lot. The witness sample should have following properties:

i. The witness piece shall be such that it presents no difficulty in measuring and testing the optical and durability requirements of the coating.

ii. The witness sample shall have a surface finish similar to the component to be coated.

It is proposed to take rejected combining prisms but with same optical quality of coating surface as witness sample. As the coating is dissolvable in nitric acid, so the same can be used further.

5. Acceptance Process

Once the witness pieces clear the qualification tests for a particular batch, all the jobs are clear with regard to coating durability. But all the coated jobs should be subjected to following acceptance tests before use in actually optical assembly:

1. **Thickness Measurement:** It should be measured by non contact method (Talysurf CCI) and should be conformed to the target thickness of ($1 \pm 0.1\mu$).
2. **Physical Inspection of Coating:** Coating should be inspected through reflected light under microscope with 20X magnification and it should conform to following points:

a) The coating shall be uniform in quality and no evidence of deterioration such as flaking, peeling, cracking or blistering. There shall be no visible discontinuities or blemishes in coated area. The edges of longer strip should be sharp.

b) Coating should not have any deep scratches and dig.

3. **Acetone Cleanability:** Coated prism shall be cleaned about 25 times by cotton wad dipped in acetone. After that it should be inspected under microscope with 20X magnification and it should conform to points **2 (a) & 2 (b)** above.

6. Precautions

1. All the samples should be handled after wearing clean room compatible and lint/dust free finger coats or hand gloves.
2. Before and after subjecting a coated sample (actual component or witness sample) to any inspection or test, the sample should be thoroughly and carefully cleaned with acetone/ Ethyl alcohol to remove any dust, finger marks, smears etc.
3. The measurement of coating thickness should be done after mounting the job in a suitable Teflon fixture and carefully to avoid any damage to the job.
4. All the accepted prisms should be stored in dehumidifier boxes.
5. All prisms of a coated batch should be labeled properly with Identity no, Batch no. and date of coating.
6. Acetone and Ethyl alcohol should be of high purity as used in ILG assembly.

References:

1. ISRO LASER GYRO- ILG 200 Design document.
2. "CCD on ISRO LASER GYRO- ILG 200 (Optics)" IISU/TR/350/2011.
3. "Process optimization of Yttrium Fluoride coating on combining prism in ILG" IISU/TR/1773/2016.
4. MIL standard **MIL-C- 675C.**