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Military specifications applicable to optical coatings

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Abstract

This paper deals with physical, environmental, and some spectral requirements of thin film coatings of optical materials as found in MIL-C-675A MIL-C-00675B, MIL-O-13830A, MIL-C-13508C, MIL-C-14806A, MIL-C-48497- MIL-F-48616, and MIL-STD-810C.

Introduction

The basic tests of optical thin film coatings required by military specification deal with:

1) Physical condition of the coating which includes surface quality (scratch and dig designation) and cosmetic defects such as stains, discolorations, or blemishes that may or may not cause functional problems.
2) Environmental durability as evidenced by the ability of the coating to withstand exposure to abrasion, water vapor, salt spray, temperature change, tape, etc.

Topics to be covered by this paper will deal with the physical and environmental attributes covered in the military specifications. Methods of measuring spectral characteristics are too individualized to be covered in a general paper such as this one, but some conditions to be aware of that can affect measurement accuracy will be mentioned.

Specifications to be covered are ones most frequently used by customers ordering thin film coatings. They are:

1) MIL-C-675A, latest revision 26 March 1971.
"Coating of Glass Optical Elements (Anti-reflection)"

Specification covers magnesium fluoride single layer coating on glass, centered between 450 and 600nm.

2) MIL-C-00675B, issued for limited use 22 January 1976.
"Coating of Glass Optical Elements (Anti-reflection)"

This is not necessarily a final revision of MIL-C-675A. The requirements are much more stringent in many areas, such as sampling and environmental tests. This revision must be required by contract before it is to be used.

3) MIL-O-13830A, latest revision 23 April 1975.
"Optical Components for Fire Control Instruments; General Specification Governing the Manufacture, Assembly, and Inspection of"

Specification covers manufacturing, assembly, and inspection of glass optical components such as lenses, prisms, mirrors, reticles, and windows, but does not cover coatings.

4) MIL-C-13508C, latest revision 19 March 1973.
"Mirror, Front Surfaced Aluminized; for Optical Elements"

Specification covers overcoated aluminum film on optical elements used as front surface mirrors.

5) MIL-C-14806A, latest revision 12 June 1969.
"Coating, Reflection Reducing for Instrument Cover Glasses and Lighting Wedges"

Covers antireflection coating on glasses of index 1.47 to 1.55 to be used as instrument cover glasses or lighting wedges.

6) MIL-C-48497, 27 June 1974.
"Coating, Single or Multilayer, Interference: Durability Requirements for"

Covers coatings on elements primarily used within the protective confines of sealed optical systems.

7) MIL-F-48616, 29 July 1977.
Filter (coatings), Infrared Interference: General Specification for"

Contains spectral performance and durability requirements for coatings used in spectral region of 0.7 to 50.0μm.

8) MIL-STD-810, latest revision 10 March 1975.
"Environmental Test Methods"

Establishes uniform environmental test methods for determing resistance of equipment to natural and induced environments peculiar to military operations. Tests in this document are sometimes used for optical coatings.

Physical Attributes of Coatings

Surface Quality

One of the first thin film coatings to be categorized was the reflection reducing film of magnesium fluoride on glass substrate (JAN-F-675). A problem involving the use of this coating is that once the glass surface reflection has been reduced, any defects on the substrate are even more visible than before. In an attempt to quantify the number and types of defects allowed, MIL-O-13830 was issued containing the first reference to U.S. Army drawing C7641866, the famous Frankford Arsenal Visual Comparison Standards for scratches and digs.

These standards consist of graded scratches or digs (round defects) on transparent glass substrates enclosed in a glass and wooden frame. Scratches are graded by the visual weight of each one when compared to the master scratch standards maintained by Frankford Arsenal until that facility was closed and now by the Picatinny Arsenal. Both scratch and dig levels are denoted by numbers. Scratch levels are 10, 20, 40, 60, and 80 with the numbers having no relation to scratch width. Dig levels are 5, 10, 20, 40, and 50. However, dig numbers relate to the diameter of the defect in that a number 5 dig is .05mm in diameter. Thus digs can be either visually compared or measured under MIL-O-13830 while scratches can only be visually compared. Indeed, there are only 2 methods allowed by this specification for using the standards (para. 4.2.2.1 and 4.2.2.2): either back light the optic being tested or edge light it. In either case, the optic must be viewed by transmission while comparing it to the standards.

In the absence of any other official surface quality standard MIL-O-13830 has been and is still being used to specify surface quality for all manner of optical materials, including opaque substrates such as germanium and silicon. Development of a technique for performing visual comparison by transmission through opaque substrate is confusing at best. In frustration, industry has adopted various techniques to get around the problem. Some companies measured the width of the scratches contained in the set of standards in their possession, but this proved even more frustrating when the number 60 scratch measured less than the number 10 as in our set at OCLI. (Our No. 80 is the widest scratch, No. 60 is the narrowest.) Others arbitrarily assigned a width to each scratch number. OCLI and several other companies designated the scratch numbers to be the width in microns of the scratch when an opaque optic was being evaluated.

The Army drawing was quietly changed in 1974 (Rev. H) to allow scratches to be measured, and stated that the scratch number denoted the width in microns. Quickly, and even more quietly, in 1976 (Rev. J) it was changed back to visual comparison.

Today, we have two specifications, MIL-F-48616 and MIL-C-48497 which specify surface quality for non transparent substrates. Instead of designating scratches and digs by number, letters of the alphabet are used to avoid confusion with visual standards of MIL-O-13830A. Table I gives a comparison of scratches; Table II of digs by letter designation and allowable size.

Out experience at OCLI has been that many times our customers dealing with government contracts can more easily substitute F-F per MIL-C-48497 on a germaninum optic for 80-50 per MIL-O-13830A than they can accept our internal document interpreting a number 80 scratch as being 80 microns wide.

Other Physical Defects

These are usually physical blemishes such as stains on the surface of a part that cannot be quantified other than by a discontinuity in the appearance of the coating (by color change, haziness, etc.). Such defects are normally referred to as "cosmetic defects," since they usually do not affect functional performance.

However, the coating should be checked both spectrally and environmentally before a decision can be made whether a defect of this type is cosmetic or functional.

Environmental Tests

Types of Testing Required

Environmental tests of thin film coatings are accelerated life tests and are therefore destructive. Usually, witness samples coated along side actual optical elements are submitted to the environmental tests. In this manner a valuable finished optic is not sacrificed.

There are 3 basic tests used to evaluate thin film coatings: 24 hours of steady state humidity exposure, abrasion (either eraser or cheesecloth), and adherence. Other specialized tests include: 240 hour exposure to cycling temperature/humidity, salt fog, solubility (water or salt solution), temperature influence, and cleanability.

Depending upon the specification involved, evaluation of a coating passing a given test may be a) visual inspection for no visual degradation; b) subjection of the coating to another test (abrasion or tape); and 3) a functional check of spectral performance and surface quality characteristics.

Listing of Tests by Specification

Tables III-VIII list environmental tests, where they are found in the military specifications, and methods to be used in evaluating test results.

The following guidelines may be useful in performing the tests:

1) Humidity - Table III. Since this is not a solubility test, always warm the sample slightly above the temperature of the chamber before inserting it into the moist atmosphere. This prevents water droplets from condensing on the optic.

2) Be sure the holding fixtures are inert material such as Delrin, so no interaction takes place between the fixture and coating.

3) Coating should not touch the fixture during a humidity or salt spray test as this may trap moisture against the coating.

4) Eraser Abrasion - Table IV. The manufacturing process and composition of the eraser is very important. MIL-E-12397 covers the grade of erasers to be used, but does not specify whether they be molded or extruded. OCLI prefers extruded erasers, as these seem to be more uniform in quality. The abrasive usedin the manufacturing process is critical. Be sure and check hardness of the erasers before using them. Keep all contacting surfaces clean. Coating or glass particles can become trapped in the rubber during one test and give false results on subsequent test.

Spectral Attributes

Optical coatings are designed to be used in various wavelength regions from the far ultraviolet (below 0.180μm) to the far infrared (above 100μm). Thus, instruments used to measure spectral qualities of the coatings are quite diverse and require specialized techniques.

It is not the purpose of this paper to detail how scans of spectral performance are obtained, but there are some general problems or pitfalls to be aware of. Some of these are listed below, and a more complete treatment of them may be found in paragraph 6.4 (informational notes) of MIL-F-48616.

1) Wavelength Calibration

Spectrophotometer manufacturers usually suggest wavelength calibration points and materials to be used as a part of the instruction manual. These are very good for keeping track of the general condition of the instrument. However, if parts are to be manufactured to tight wavelength tolerances, additional calibration points should be found and checked frequently to insure parts meet wavelength requirements. For example, if an infrared radiometer filter is being manufactured, daily calibration checks may be required with gas bands known with a precision of $\pm$ .01cm $^{-1}$.

2) Temperature Effects

Coatings tend to shift to shorter wavelengths with a decrease in temperature. Therefore, it is important that parts be scanned at the same temperature as the operating temperature of the system, or that some method of extrapolation be used to predict coating performance.

3) Angle of Incidence

Coating performance changes significantly as the angle of incidence increases.
a) Spectral features shift to shorter wavelengths.
b) Transmission and reflection levels change.
c) Coating becomes more sensitive to the polarization of the incident beam.
Spectrophotometers used to scan parts use beams that are partially polarized and include rays that are not at normal incidence. This must be factored into any calculation to predict final filter performance based on a spectrophotometer measurement.

4) Internal Defects of Substrates

Internal defects of substrates can seriously deteriorate filter and related system performance. Visually opaque substrates require specialized tests to characterize these defects.

Improperly oriented birefringent substrates, such as sapphire, deteriorate high resolution performance. Thus, if birefringence isa problem, proper orientation should be specified at the procurement stage.

5) Physical Thickness of Coatings

Coating thicknesses can vary in thickness up to several thousands of an inch. This parameter should not be ignored when designing filter holders.

6) Test Conditions

When test conditions must vary from operating conditions, the filter manufacturer and customer should work together to insure that the proper adjustments have been made in theoretical calculations or in the type of tests performed to account for effects of temperature, angle of incidence, and polarization.

7) Wrapping Tissue

Papers containing certain chemicals, or which are fabricated from bleached wood pulp, may cause deterioration of coatings. Be sure to use chemically inert paper to wrap coated optics.

TABLE I

SCRATCH DESIGNATION

Letter (MIL-F-48616) (MIL-C-48497)	Number (Unofficial)	Width in Millimeters	Width in Inches
A	5	.005	.00020
B	10	.010	.00039
C	20	.020	.00079
D	40	.040	.00157
E	60	.060	.00236
F	80	.080	.00315
G	120	.120	.00472

TABLE II

DIG DESIGNATION

Letter		Average Dig Diameter	
(MIL-F-48616) (MIL-C-48497)	Number (MIL-O-13830A)	In Millimeters	In Inches
A	5	.05	.0020
B	10	.10	.0039
C	20	.20	.0079
D	30	.30	.0118
E	40	.40	.0157
F	50	.50	.0197
G	70	.70	.0276
H	100	1.00	.0394

TABLE III

24 HOUR HUMIDITY TEST

(120°F + 4°F, 95-100% Relative Humidity)

No.	Specification	Test Paragraph	Method of Evaluation
1.	MIL-C-675A	4.6.9	Visual inspection.
2.	MIL-C-00675B	4.5.8	Visual inspection followed by 40 rubs with eraser then visual inspection again.
3.	MIL-C-13508C	4.4.7	Visual inspection.
4.	MIL-C-14806A	4.4.6	Visual inspection.
5.	MIL-C-48497	4.5.3.2	Visual Inspection.
6.	MIL-C-48616	4.6.8.2	Visual inspection and surface quality evaluation.

TABLE IV

SEVERE ABRASION (ERASER) TEST

No.	Specification	Test Paragraph	No. of Strokes	Method of Evaluation
1.	MIL-C-675A	4.6.11	20	Visual inspection.
2.	MIL-C-00675B	4.5.10	40	Visual inspection.
3.	MIL-C-14806A	4.4.7	20	Visual inspection.
4.	MIL-C-48497	4.5.5.1	20	Visual inspection.
5.	MIL-F-48616	4.6.10.1	20	Visual inspection.

TABLE V

MODERATE ABRASION (CHEESECLOTH) TEST

(1-1½ Pounds Pressure with Cheesecloth Conforming to CCC-C-440. Pad to be ¼ inch Thick x 3/8 inch Wide)

No.	Specification	Test Paragraph	No. of Strokes	Method of Evaluation
1.	MIL-C-00675B	4.5.11	50	Visual
2.	MIL-C-13508C	4.4.5	50	Visual
3.	MIL-C-48497	4.5.3.3	50	Visual followed by spectral test.
4.	MIL-F-48616	4.6.8.3	50	Visual

TABLE VI

ADHESION TEST (Type 1 Tape per L-T-90)

No.	Specification	Test Paragraph	Speed of Removal	Method of Evaluation
1.	MIL-C-00675B	4.5.12	Quick	Visual
2.	MIL-C-13508C	4.4.6	Slow	Visual
3.	MIL-C-48497	4.5.3.1	Quick	Visual
4.	MIL-F-48616	4.6.8.1	Quick	Visual

TABLE VII

SALT SPRAY (FOG) Test Conditions as Specified in ASTM-B117

No.	Specification	Test Paragraph	Exposure Time	Method of Evaluation
1.	MIL-C-675A	4.6.10	24 hours	Pass eraser test, 20 strokes.
2.	MIL-C-00675B	4.5.9	24 hours	Pass eraser test, 40 strokes.
3.	MIL-C-13508C	4.4.8	24 hours	Visual
4.	MIL-C-14806A	4.4.8	24 hours	Visual
5.	MIL-F-48616	4.6.10.4	24 hours	Visual, then moderate or severe abrasion depending on contract.

TABLE VIII

SUMMARY OF ENVIRONMENTAL TESTS REQUIRED LISTED BY MILITARY SPECIFICATION

MIL-C-675A	4.6.8	Solubility - 24 hour immersion in salt solution. (visual inspection)
	4.6.9	Humidity (Table III)
	4.6.10	Salt Spray (Fog) (Table VIII)
	4.6.11	Abrasion (Table IV)
MIL-C-00675B	4.5.7	Salt Solubility-24 hour immersion in salt solution followed by severe abrasion (Table IV).
	4.5.8	Humidity (Table III)
	4.5.9	Salt Spray (Fog) (Table VII)
	4.5.10	Severe Abrasion (Table IV) (Knoop hardness $\geq$ 450 kg/mm^2)
	4.5.11	Moderate Abrasion (Table V) (Knoop hardness $<$ 450 kg/mm^2)
	4.5.12	Adhesion (Table VI)
MIL-C-13508C	4.4.4	Temperature Influence -80°F and 160°F for 5 hours each (visual inspection)
	4.4.5	Hardness (Table V)
	4.4.6	Adherence (Table VI)
	* 4.4.7	Humidity (Table III)
	* 4.4.8	Salt Spray (Table VII)
MIL-C-14806A	4.4.6	Humidity B (Table III)
	4.4.7	Abrasion (Table IV)
	4.4.8	Salt Fog (Table VII)
	4.4.9	Humidity C MIL-STD-810B Method 507-1, 48 hrs. (visual)
	** 3.11.1	High Temperature, 160°F, 48 hrs. (visual)
	** 3.11.2	Low Temperature, -65°F, 48 hrs. (visual)
	** 3.11.3	Thermal Shock, 160°F, 4 hrs. then within 5 minutes -65°F, 4 hrs, then within 5 minutes 160°F (visual).
	** 3.11.4	Humidity A, MIL-STD-810B Method 507-1, 240 hrs. (visual)
	** 3.11.5	Dust, MIL-STD-810B, Method 510-1, 28 hrs. (visual)
	** 3.11.6	Fungus, MIL-STD-810B, Method 508-1, 28 days (visual)
MIL-C-48497	4.5.3.1	Adhesion (Table VI)
	4.5.3.2	Humidity (Table III)
	4.5.3.3	Moderate Abrasion (Table V)
	4.5.4.1	Temperature, -80°F for 2 hrs. and 160°F for 2 hrs. followed by adhesion (Table VI).
	4.5.4.2	Solubility and Cleanability - Immersion in solvents (visual and spectral tests for evaluation).
	* 4.5.5.1	Severe Abrasion (Table IV)
	* 4.5.5.2	Salt Solubility - 24 hour immersion in salt solution (visual).
	* 4.5.5.3	Water Solubility - 24 hours immersion in distilled water, (visual).
MIL-F-48616	4.6.8.1	Adhesion (Table VI)
	4.6.8.2	Humidity (Table III)
	4.6.8.3	Moderate Abrasion (Table V)
	4.6.9.1	Temperature - 80°F for 2 hrs. and +160°F for 2 hrs. followed by adhesion (Table VI).
	4.6.9.2	Solubility and Cleanability - Immersion in solvents (visual).
	* 4.6.10.1	Severe Abrasion (Table IV)
	* 4.6.10.2	Salt Solubility - 24 hr. immersion in salt solution (visual).
	* 4.6.10.3	Water Solubility - 24 hr. immersion in distilled water (visual).
	* 4.6.10.4	Salt Spray (Fog) (Table VII)

*   Must be required by contract to be applicable.
**   Certification tests only.

Questions from the Floor

Question 1: Do you have non-military standards for commerical hardware? Who is responsible for the military standards now?

Answer 1: I don't know of any non-military "standards." In the past, Davidson Optronics has sold comparison standards with numbers comparable to the Frankford Arsenal standards. (Davidson also has scratch "standards" up to #200.) However, until these are visually matched to the master standards maintained by the army, they cannot be used as standards. We purchased some of these standards several years ago and found the Davidson standards did not always match the Army standards.

Pickatinny Arsenal now is responsible for the military standards.

Question 2: How do you handle scratches which are jagged, rough, etc., in comparison to Arsenal standards which are smooth and straight?

Answer 2: Approach each area of a jagged scratch separately as if it were a separate scratch. For example, one scratch may be composed of areas that are comparable to #80, #60, and #20 standards. If the required surface quality were 60-40, the part fails. If it were specified as 80-50, and the portion comparable to #80 is less than 1/4 the diameter in length, evaluate the part per paragraph 3.5.2.1.1 of MIL-O-13830A.

Question 3: Do you think the number of rubs in the cheesecloth test is significant? Do you think a single number is adequate?

Answer 3: No, I don't think the number is significant by itself, but it can be a measure of comparative cleanability or durability of a coating. This test like all other environmental tests is an artificial life test. Our company, for example, uses more eraser or cheesecloth rubs for process control on certain coatings, so we can tell if the coating parameters need to be adjusted before we are faced with an environmental failure.

The number is adequate for a "Go-No-Go" decision only.