

University of Nebraska - Lincoln

DigitalCommons@University of Nebraska - Lincoln

---

U.S. Air Force Research

U.S. Department of Defense

---

2005

## Sample preparation and characterization of artificially aged aircraft coatings for microstructural analysis

Lisa M. Farrier

*Wright-Patterson Air Force Base*

Stephen A. Szaruga

*Wright-Patterson Air Force Base*

Follow this and additional works at: <https://digitalcommons.unl.edu/usafresearch>



Part of the [Aerospace Engineering Commons](#)

---

Farrier, Lisa M. and Szaruga, Stephen A., "Sample preparation and characterization of artificially aged aircraft coatings for microstructural analysis" (2005). *U.S. Air Force Research*. 20.

<https://digitalcommons.unl.edu/usafresearch/20>

This Article is brought to you for free and open access by the U.S. Department of Defense at DigitalCommons@University of Nebraska - Lincoln. It has been accepted for inclusion in U.S. Air Force Research by an authorized administrator of DigitalCommons@University of Nebraska - Lincoln.

# Sample preparation and characterization of artificially aged aircraft coatings for microstructural analysis

Lisa M. Farrier \*, Stephen L. Szaruga

*Air Force Research Laboratory, Materials Directorate, Wright-Patterson Air Force Base, Ohio 45433-7750, United States*

Received 24 May 2004; received in revised form 13 April 2005; accepted 13 April 2005

---

## Abstract

The paper describes a method for metallographic preparation of artificially aged aircraft coatings. In order to better understand the in-service performance and identify degradation mechanisms of an aircraft coating, complete characterization of the microstructure is essential. This paper discusses metallographic sample preparation and subsequent microscopy techniques (light optical microscopy, scanning electron microscopy) for characterization of a standard polyurethane aircraft coating system. The preparation method has proven to produce good, consistent results on a wide range of laboratory-produced simulated environmental exposures.

The military specification coating system under study (MIL-PRF-85285C and MIL-PRF-23377G) degrades severely after accelerated weathering. Typical degradation includes deterioration of the polyurethane-based resin system in the topcoat and is observed as a visible change in the color. Increased porosity and some physical deterioration were also observed. In addition, some inorganic pigments in the primer appear to migrate into the topcoat during simulated exposure.

Published by Elsevier Inc.

*Keywords:* Military aircraft; Microstructural analysis; Coating degradation; Polyurethane

---

## 1. Introduction

Military aircraft require and employ exterior coating systems that are very different from automotive and architectural coatings. The coating system for a fighter, bomber, or transport aircraft serves three purposes. Most importantly, the coating system must protect the aluminum structure and associated fasten-

ing and joining mechanisms from corrosion. In addition, the exterior coating system must provide survivability features that reduce the aircraft's chances of detection from other aircraft and MANPADS (Man-Portable Air Defense Systems). Furthermore, the exterior coating system must maintain its barrier, weathering, and fluid resistant properties for a reasonable length of time to prevent excessive refinishing between depot maintenance cycles. Ideally, the coating would last for 6–8 years, but in practice refinishing is required after 18–24 months.

---

\* Corresponding author.

*E-mail address:* Lisa.Farrier@wpafb.af.mil (L.M. Farrier).

In general, military aircraft utilize a three-layered coating system comprised of a conversion coating, primer, and topcoat as shown in Fig. 1. The conversion coating layer is an aluminum surface treatment that primarily promotes adhesion of the primer to the substrate. The thin (10–20  $\mu\text{m}$ ) aluminum oxide coating is deposited through an electrolytic process involving sulfuric and chromic acid immersion [1]. It is then sealed and densified by boiling in water. Chromium salts are dissolved within the oxide film during the sealing process to help protect aircraft structures from corrosion on the underlying aluminum by inhibiting oxidation and by modifying the local acidic/alkaline (pH) chemistry of the surface.

The primer layer overcoats the treated aluminum surface to provide additional corrosion protection and promote adhesion of the topcoat. The primer is typically epoxy- or polyurethane-based with added corrosion inhibitors such as strontium chromate. The primer has a high pigment volume concentration (PVC) resulting in a porous, brittle coating with no durability [2]. The topcoat layer serves as the outermost barrier to the environment. The most important feature of the topcoat is to impart camouflage features such as proper color (typically gray shades) and low surface gloss (sheen). The low visual gloss feature requires that the topcoat contain high PVC of inorganic pigments and flattening agents to produce a rough uneven surface. This scatters reflected light and reduces glint from the sun, which helps to avoid visual detection. Details of the organic coating chemistry are provided elsewhere [3].

Due to the high PVC of inorganic pigments, the camouflage topcoat is extremely sensitive to ultraviolet degradation from the sun. Camouflage coatings contain a smaller amount of polymeric resin to wet out the pigments and fillers compared to high gloss coat-

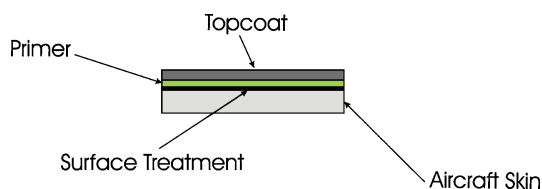


Fig. 1. Components of U.S. military aircraft coating system.

ings. The resultant deterioration of the resin exposes more inorganic pigment, causing chalking or discoloration which commonly leads to the need for refinishing. Currently, color and gloss measurements are used for monitoring and determining the extent of coating degradation; however, these measurements do not quantify specific degradation mechanisms or remaining service life. Therefore, a method for determining coating microstructure of aircraft coating systems is explored in this paper in order to observe degradation of the coating.

## 2. Experiment procedure

### 2.1. Coating application

The typical procedure for coating application is described. Panels of Aluminum 2024 T-3 are first cut into  $76 \times 152$  mm pieces and then cleaned using standard surface preparation procedures. Each panel is hand scrubbed with a 10% by volume of cleaner solution<sup>1</sup> using a scouring pad<sup>2</sup> followed by a rinse with warm tap water. The panels are then immersed in a heated tank of the same cleaner for 5 min at 60 °C. The panels are rinsed and a water break test is performed to determine if contaminants are present on the surface. Next, the panels are immersed in a deoxidizer solution for 2 min at room temperature. The deoxidizer solution is generally formulated using the following volume–volume percentages: 35% *n*-butyl alcohol, 25% isopropyl alcohol, 15% *ortho*-phosphoric acid (85%), and 25% de-ionized (DI) water. Again, the panels are rinsed with warm tap water and a water break test is performed.

Following the cleaning procedures, the panels are chromate conversion coated in accordance with MIL-C-5541E [4]. The panels are immersed in a chromate conversion coating solution<sup>3</sup> at room temperature. Depending on the substrate, the time of immersion varies from 90 to 120 s and the solution

<sup>1</sup> Brulin 815GD (*Brulin Corporation, Indianapolis, Indiana*).

<sup>2</sup> Red Scotch-Brite (*3M, St. Paul, Minnesota*).

<sup>3</sup> Alondine 1200S (*Henkel Technologies, Madison Heights, Michigan*).

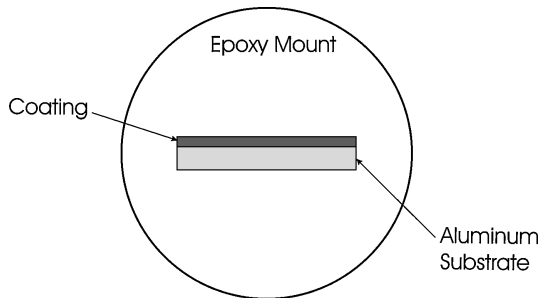


Fig. 2. Schematic of mounted sample.

is maintained at a pH between 1.3 and 1.8. Next, the panels are thoroughly rinsed with dionized water. The panels are then allowed to dry overnight in a climate controlled room operating at the temperature and humidity that will be present when the panels are coated. Before applying the coatings, each panel is wiped with Methyl Ethyl Ketone (MEK) to remove dust and contaminants. The coatings are applied using a High Volume Low Pressure (HVLP) spray gun. The applied coatings meet the U.S. Department of Defense specification number for standard gray MIL-PRF-85285C [5] polyurethane topcoat and MIL-PRF-23377G [6] high solids epoxy primer. The desired dry film thickness (DFT) of the epoxy primer is 15 to 23  $\mu\text{m}$  (0.6 to 0.9 mils) and the DFT of the polyurethane topcoat is 43 to 58  $\mu\text{m}$  (1.7 to 2.3 mils). This produces variability in total DFT of the coating system from 58 to 81  $\mu\text{m}$  (2.3 mils to 3.2 mils).

After curing for 14 days at room temperature, the panels were placed in a QUV exposure chamber<sup>4</sup> using UV-B bulbs with continuous peak output at 310 nm. The QUV test method is a common method for simulating environmental exposure and characterizing polyurethane topcoat exterior coatings. The method utilizes commercial, ultraviolet lamps and cycles of water condensation to emulate outdoor weathering. In this experiment, the exposure cycle consisted of 8 h of light with no humidity at an irradiance of 0.63  $\text{W}/\text{m}^2$  and a black panel temperature of 60 °C followed by 4 h in the dark with 50% relative humidity. The panels were

exposed for a total of 2016 h in the cabinet, which is a total of 168 cycles of UV light exposure followed by condensation. The total irradiance received by a 76 × 152 mm panel exposed for 2016 h is 9.83  $\text{W}/\text{m}^2$ .

## 2.2. Proposed metallographic method

The coatings were sectioned into 25 × 6 mm samples using a shear and then vacuum mounted in epoxy using a leg clip at one end. The configuration of the mounted sample is shown in Fig. 2. The samples were ground using 240, 320, 400, and 600 grit SiC papers with water. During each step of the grinding process, the sample was rotated 90° and observed using a light optical microscope (LOM) to ensure that scratches induced from the previous step were removed. The grinding process was continued using a rotating wheel with 800 grit SiC paper at 100 RPM for 15 min with water continuously applied to complete the rough polishing. Light pressure was applied manually. The best results for degraded samples were obtained by reducing the amount of time spent on 800 grit SiC paper to 10 min.

## 2.3. Method justification

Over the course of this study, several grinding and polishing techniques were explored. LOM was used to determine the damage during preparation. Table 1 identifies exposure conditions for the samples.

The proposed preparation method of grinding through 800 grit resulted in minimal damage to the coating system for both the before and after QUV exposure, as shown in Figs. 3 and 4. Continuing the grinding process on 1200 grit SiC paper at 100 RPM resulted in the attack of the primer layer at the interface of the aluminum as shown in Fig. 5.

Table 1

Sample ID	MIL specification	Exposure to QUV (h)
A	MIL-C-85285	0
B	MIL-C-85285	756
C	MIL-C-85285	2016

<sup>4</sup> Q Panel QUV-B exposure chamber (Q-Panel Lab Products, Cleveland, Ohio).

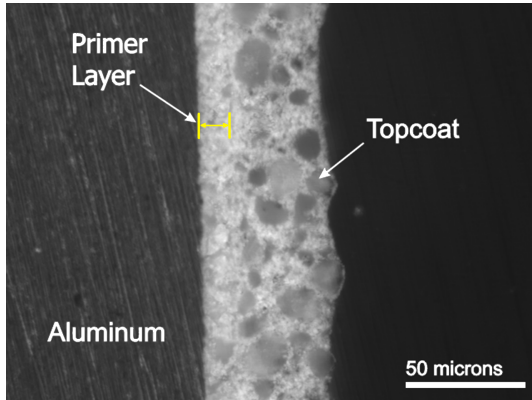


Fig. 3. Sample A (no QUV exposure) after proposed metallographic preparation.

Several attempts were made with diamond lapping films under different conditions. Diamond lapping films consists of precision graded diamond particles resin bonded to a polyester film backing to achieve a finer finish. In the extreme case, the sample was ground using 400 and 600 grit SiC papers with water and then polished using 30 and 15  $\mu\text{m}$  diamond lapping films at 150 RPM. The primer layer smeared into the aluminum, creating a “double” primer layer as shown in Fig. 6.

Although there was a concern over possible contamination of the coating from the polishing solution, sample preparation utilizing this fluid

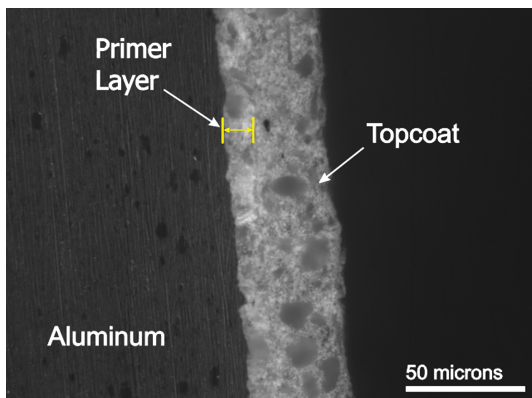


Fig. 4. Sample C (2016 h exposure to QUV) after proposed metallographic preparation.

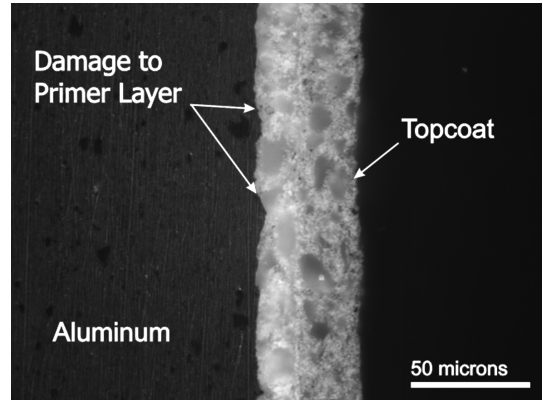


Fig. 5. Damage to primer layer as a result of grinding on 1200 grit SiC paper; Sample C.

was attempted. The resulting sample surface flatness was not good. The solution removed some components of the coating and not others.

### 3. Results and discussion

#### 3.1. Microstructural characterization

Images of the coatings were captured using a JEOL scanning electron microscope (SEM) in addition to a Reichert-Jung LOM. Samples prepared for SEM were cleaned several times with soap and water before coating them with carbon. Then

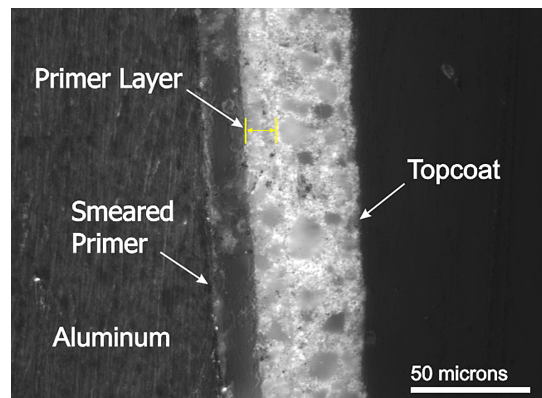


Fig. 6. Cross-section of coating polished by diamond lapping; Sample C.

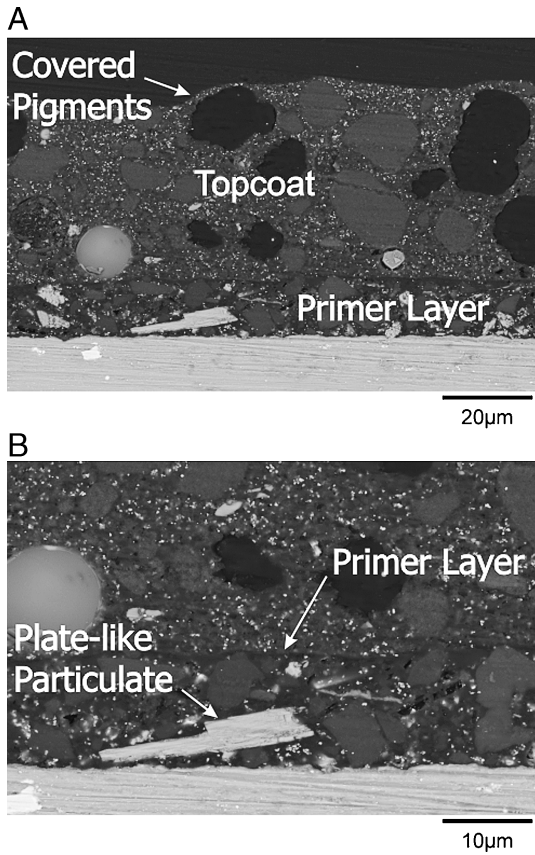


Fig. 7. Backscatter secondary electron SEM images; Sample A, no QUV exposure: A) and B); same area at two magnifications.

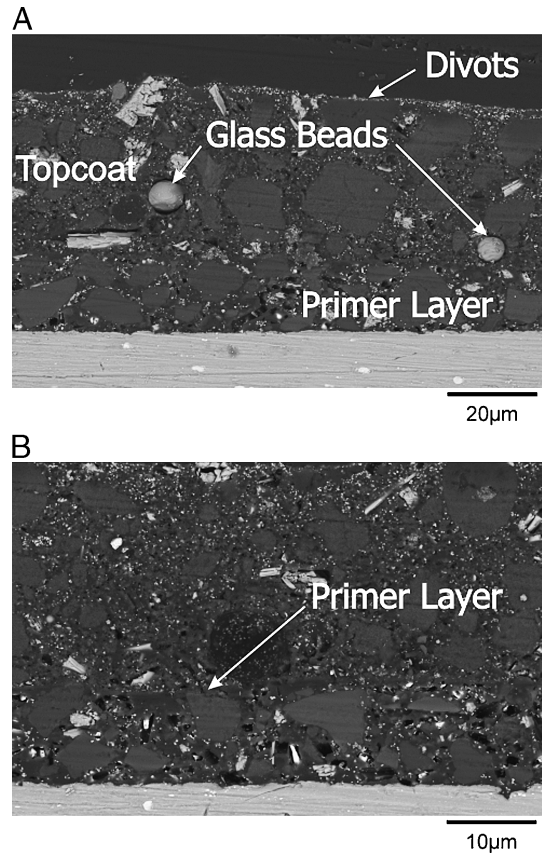


Fig. 9. Backscattered electron SEM images; Sample B, 756 h exposure to QUV: A) and B); different areas at two magnifications.

conductive paint was applied to the epoxy mount to create a conductive surface for viewing in the SEM.

SEM images were obtained using backscatter imaging. This technique is useful to identify the microstructure in organic-based coatings containing

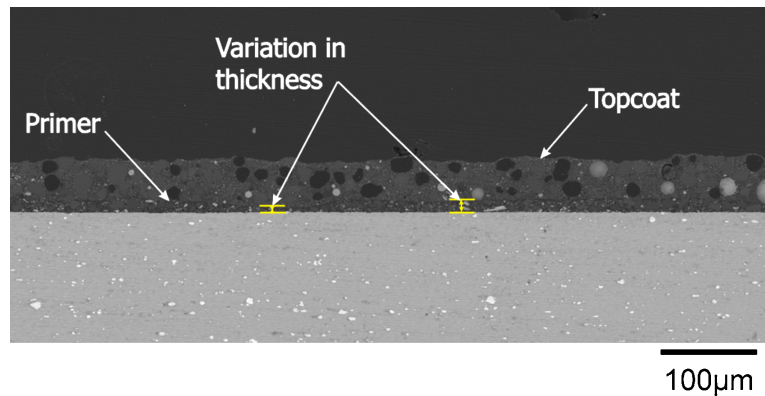


Fig. 8. Backscatter SEM image viewing typical coating microstructure; Sample A.

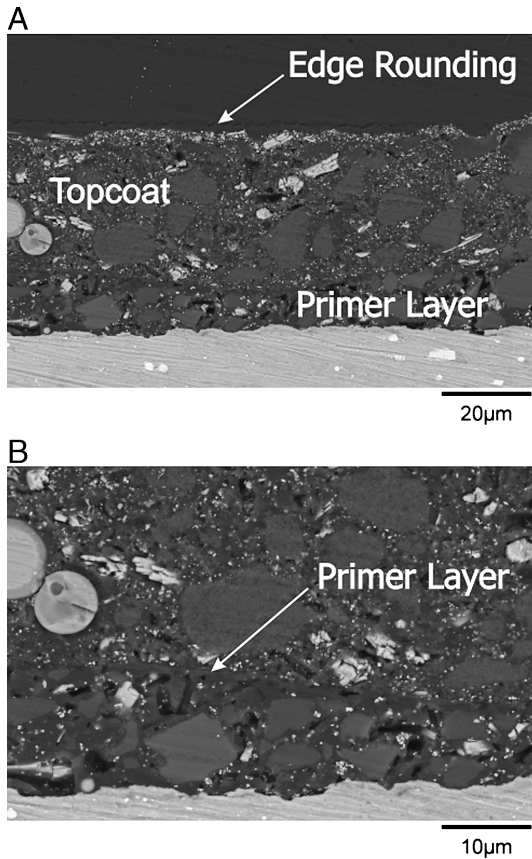


Fig. 10. Backscatter secondary electron images; Sample C, 2016 h exposure to QUV.

inorganic additives because the resulting image contrast is based upon differences in atomic mass. Lighter atomic mass components (e.g., polymers,

carbon and boron) appear darker in the image; higher atomic mass components (metals, glasses) appear lighter. The primer layer and topcoat are clearly evident in addition to the precipitates of the aluminum alloy.

The SEM images for the control Sample A reveal a non-porous topcoat with surface pigments covered by a thin layer of polymer shown in Fig. 7. The primary pigments of the topcoat are titanium dioxide (10–20 wt.%) and carbon black (3–5 wt.%). Carbon black is added to the topcoat to produce the gray color. The remaining contents include siliceous filler, a small amount of flattening agent and organic pigment. Fig. 8 provides the overall view of Sample A exhibiting the variation in thickness of the topcoat and primer over the substrate. Large black pigments dispersed throughout the topcoat are visible. Fine scratches are observed across large pigments at higher magnifications. Glass beads are intact and scattered randomly throughout the topcoat. It is believed the coating manufacturers include these beads to physically roughen the surface and impart a low gloss finish. Microscopic inspection during this study found the distribution of these spheres throughout the topcoat, not necessarily at the surface. The primer appears to be heavily loaded with a variety of particulates of a different morphology than the topcoat. More specifically, non-spherical, plate-like particles (mica) are abundant within the primer. The primer also contains strontium chromate (25–30 wt.%) and siliceous filler.

A different surface texture is revealed when different QUV exposures are compared. The surface

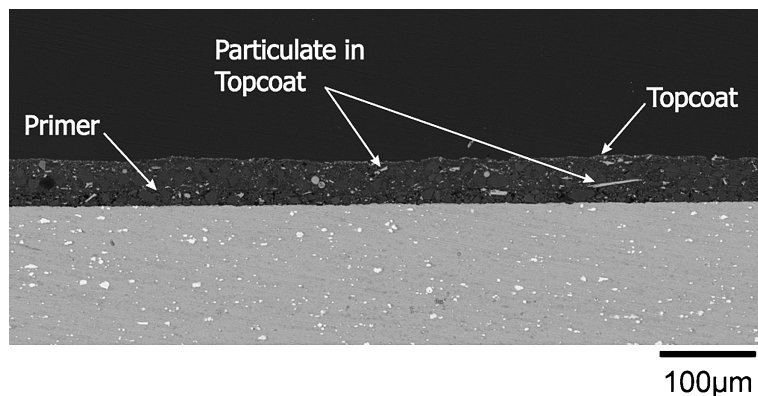


Fig. 11. Backscatter SEM image viewing degraded microstructure; Sample C.

appears to be rough; however, the roughness differs from the control in that divots are created at the surface shown in Fig. 9. The surface appearance is due to the degradation of the polyurethane resin system and release of pigments through chalking. Large black pigments are not as prevalent. Some of the

particulate from the primer has migrated into the topcoat.

Sauer describes the effects of polishing thermal spray coatings, and in particular, the problem of edge rounding observed for non-metals [7]. Edge rounding is a preparation artifact that becomes more

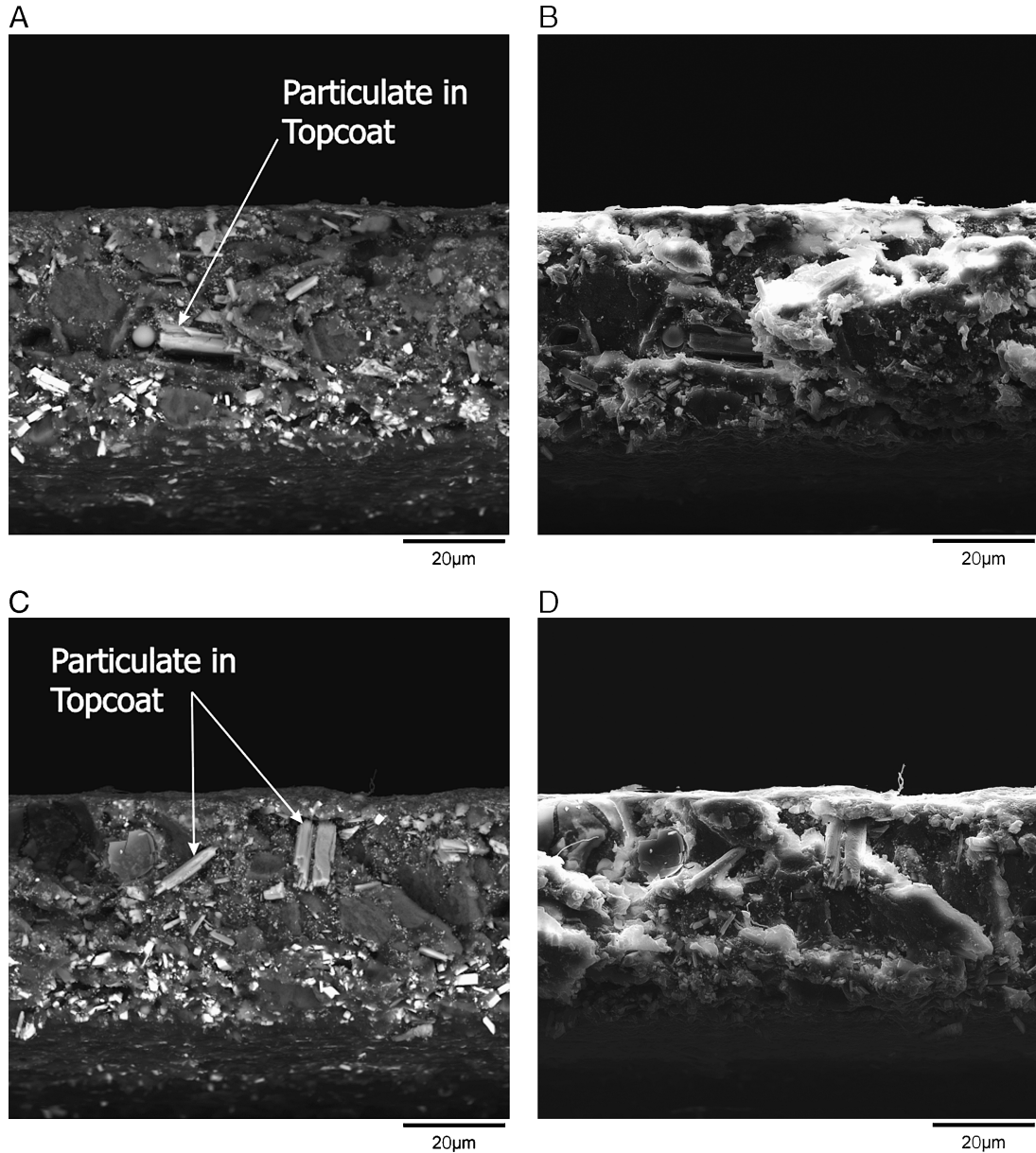


Fig. 12. SEM images of the edge of Sample C. A) Backscattered electron image. B) Corresponding secondary electron image. C) Backscattered electron image, second area. D) Corresponding secondary electron image, second area.



apparent in degraded samples as shown in Fig. 10. The bulk of the topcoat and primer coatings appears to be substantially degraded when compared to images of Sample A. It is evident in Fig. 11 that some of the plate-like particulates from the primer have migrated into the topcoat. This observation is not considered to be the result of the preparation method. There are no signs of drag marks through the coating and increased porosity observed using secondary mode indicates that smearing is not a factor. The mechanism for the migration of these particulates from the primer to the topcoat after aging is not understood.

Some degree of increased porosity is expected to result from UV exposure due to chalking. However, before making quantitative judgments about the porosity of the coating, the relationship between increased porosity and the true microstructure must be further investigated.

In order to verify that the particulate movement is not due to the preparation method, a non-polished

sample was examined. This sample was sheared from an exposed section of the panel and then carbon coated on the long edge to conduct electrons. The sample was placed vertically in a clamp allowing the edge to be viewed in the SEM.

In both pairs of SE and BSE images shown in Fig. 12, the particulate is surrounded by the topcoat effectively holding the particulate in position. These images of the edge confirm that the particulate movement is not due to the preparation method. This observation suggests that the UV radiation penetrates the topcoat and causes the degradation of the underlying primer. The UV transmissivity of the topcoat was therefore examined.

### 3.2. Free film transmissivity

Free films of the polyurethane topcoat were cast at thicknesses below and above the desired dry film thickness (DFT) at 40.6 (1.6 mils) and 76.2  $\mu\text{m}$

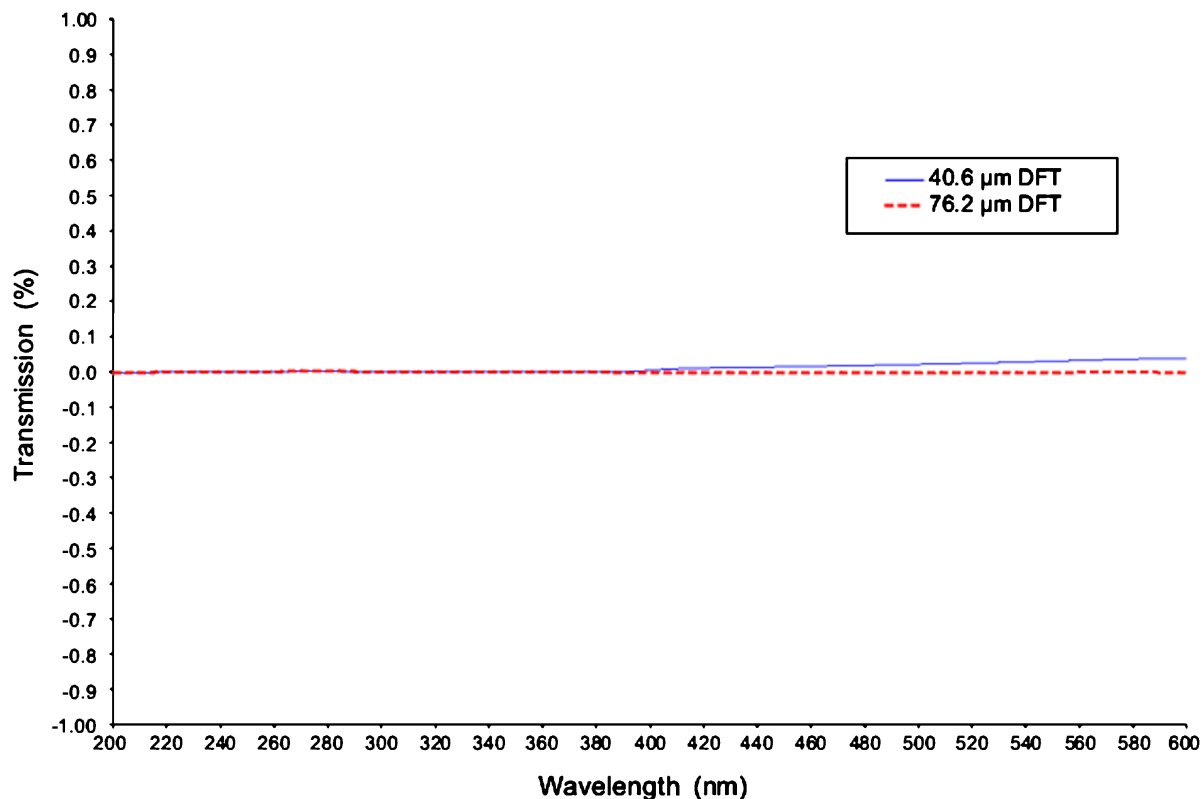


Fig. 13. Free film UV transmission spectra for two dry-film thicknesses (DFTs).

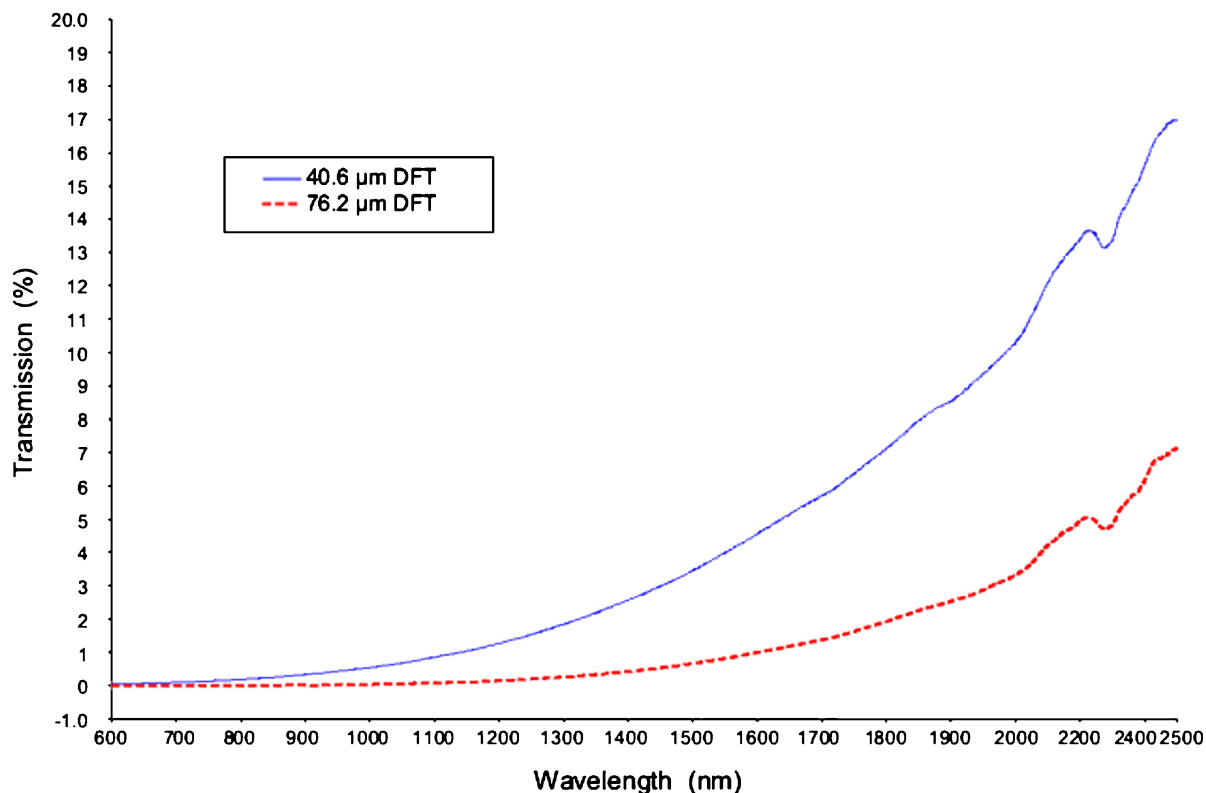


Fig. 14. Transmission spectra of the near-infrared region.

(3.0 mils), respectively. The UV transmission was then measured for each film<sup>5</sup>. The initial UV scans resulted in 0% transmission for the 40.6 and 76.2 μm free films indicating that the topcoat is opaque to UV irradiation. However, when the transmission scale is plotted from -1% to +1%, minute transmission in the visible region of the 40.6 μm film is revealed.

Fig. 13 is the UV transmission spectra with the expanded scale showing increasing transmission above 400 nm. The transmission maximum for the 40.6 μm film is 0.04% at 600 nm.

The spectrum yielded significant transmission of near-infrared energy as shown in Fig. 14. It also illustrates the transmission as a function of thickness. The 40.6 μm film has a transmissivity of about 16% at 2500 nm while the 76.2 μm film has approximately half this level. A greater transmissivity for the

40.6 μm film is expected due to the smaller thickness of the film.

The transmission spectrum in the mid-infrared region was obtained using Fourier Transform Infra-Red spectroscopy (FTIR) shown in Fig. 15. Spectral peaks are observed and denoted by wavenumbers. A broad transmission band from approximately 2780 to 1780  $\text{cm}^{-1}$  dominates the spectra.

The free film data suggests that it is possible for UV irradiation to penetrate the topcoat and cause damage to the underlying primer. However, it is more likely that the degradation observed in the epoxy primer is caused by thermal radiation indicated by the IR spectra.

#### 4. Summary

It is important to obtain a true microstructure of aircraft coatings for the determination of degrada-

<sup>5</sup> A Perkin-Elmer Lambda 900 was used.

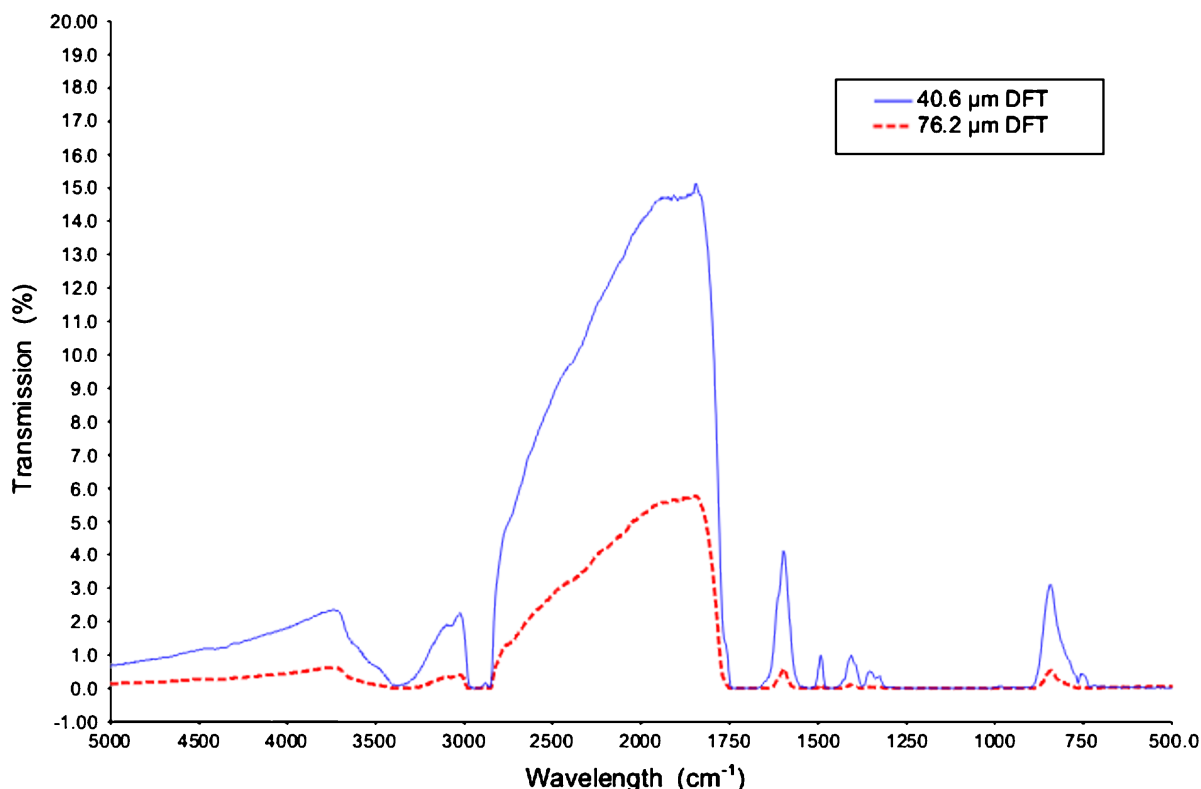


Fig. 15. Transmission spectra of the mid-infrared region for two dry-film thicknesses (DFTs).

tion mechanisms. This is a challenging objective for these multilayer coatings due to the variety of materials incorporated in each layer. The method described in the present paper was effective for the required microstructural characterization. The fine scratches that remain do not prevent the observation and recording of fundamental differences in coating microstructures before and after QUV exposure. Before QUV exposure, the method produces excellent sample flatness and allows the recording of accurate coating characteristics. After exposure, however, some edge rounding is observed using the identical preparation procedure. Overall, the sample preparation method presented does reveal the distribution of pigments and surface roughness within these complex coatings when samples are examined in the SEM.

Microstructures revealed degraded surface pigments after QUV exposure. The microstructures also revealed that some of the inorganic pigments

in the primer migrate into the topcoat after QUV exposure. Minute transmission of ultraviolet radiation through the topcoat was detected at 0.04%. Additional transmission spectra of the topcoat yielded peaks in the IR region suggesting that thermal radiation can also penetrate the highly loaded topcoat and may be a source of degradation of the underlying primer. The effects of humidity are to be examined in a further investigation of the particulate movement phenomenon.

#### Acknowledgements

The authors would like to thank Doug Hufnagle from the Air Force Research Laboratory Coatings Technology Information Office (CTIO) for guidance and analysis of free film UV transmissivity. Also, we would like to thank Luann Piazza for SEM consultation.

## References

- [1] Polmear I. Light alloys—metallurgy of the light metals. Materials Park, OH: American Society of Materials International; 1981.
- [2] Bierwagen G, Tallman D. Choice and measurement of crucial aircraft coatings system properties. *Prog Org Coat* 2001;41: 201–16.
- [3] Wicks Z, Jones F, Pappas SP. Organic coatings: science and technology. 2nd ed. New York: John Wiley & Sons; 1999.
- [4] U.S. military specification—chemical conversion coatings on aluminum and aluminum alloys, MIL-C-5541E (AS);1990 (30 November).
- [5] U.S. military specification—polyurethane coatings for aircraft and support equipment, MIL-PRF-85285C (AS);1997 (30 April).
- [6] U.S. military specification—epoxy, high solids primer coatings, MIL-PRF-23377G (AS);1994 (30 September).
- [7] Sauer J. Metallographic preparation of thermal spray coatings: coating sensitivity and the effect of polishing intangibles. Materials Park, OH: American Society of Materials International; 1996.