# NONDESTRUCTIVE CHARACTERIZATION OF CORROSION PROTECTIVE COATINGS ON ALUMINUM ALLOY SUBSTRATES

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#### **ABSTRACT**

This paper describes the initial phase of the development of a nondestructive, multisensor approach for detecting, quantifying and monitoring degradation of organic coatings applied to aluminum surfaces. Descriptions of the purposes and chemical compositions of layered coatings used on aircraft structures are provided. The discussion then concentrates on ultrasonic thickness measurements. One is the well-established pulse/echo scanning acoustic microscopy and, as a proposed alternative, continuous acoustic waves measurements with a probe in contact to the sample. Advantages and disadvantages of the two methods and their potential as in field applications are discussed.

## INTRODUCTION

Nondestructive characterization of corrosion protective coatings is of exceptional importance to enhance the usability of the coating system and thus to reduce the costs of aircraft maintenance and to avoid extensive environmental pollution. The possible failure of the coating leads to insufficient corrosion protection and finally to the failure of the Al alloy. Determination of mechanical and physical properties of the coating and the condition of the interface are required, both under different types of load and environmental exposure. Furthermore, it is necessary to detect corrosion damage in the aluminum alloy below the coating [1].

To achieve this, the employment of high-resolution nondestructive evaluation (NDE) techniques is necessary. The NDE includes thermal, acoustic and eddy current techniques as possible in-field applications. Each of these techniques is sensitive to different material defects and properties. Generally NDE measurements of layered structures are highly influenced by the thickness of the examined material. Thus, to start the NDE characterization, thickness evaluation has to be carried out to calibrate the system. Different acoustic techniques are employed.

#### **EXPERIMENTS**

## **Coating System**

Military aircraft coatings have to be multi-functional. Only a multi-layer coating system can satisfy all requirements. Currently used coating systems consist of surface pretreatment, primer layer and topcoat. The schematic of such a coating system is shown in Figure 1. First a solution is sprayed on the Al substrate, which contains chromates as corrosion inhibitors. The preexisting (and for Al alloys porous) oxide-film changes from Al<sub>2</sub>O<sub>3</sub> to a mixture of Al<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub> and CrO<sub>2</sub>. The developed film is called chromate conversion coating. Chromates are especially used because of their self-healing nature. However, chromium is known to be a toxic

substance. Future research concentrates on finding substitute inhibitors with little or no environmental pollution [2].

The primer coating acts as a physical barrier between substrate and environment. Hard thermosets, especially epoxy resins are used providing good adhesion to the substrate material and are capable to withstand mechanical loading during service. Furthermore, epoxies are highly chemical resistant and can contribute to corrosion protection.

The topcoat's main purpose is to provide protection against erosion and mechanical abrasion. Thus, both sufficient flexibility and hardness are needed. It is also used to achieve a decent cosmetic appearance. Furthermore, the topcoat material must be water-repellent and UV radiation resistant. Certain polyester-urethanes (PUR) meet all these requirements the best [2].

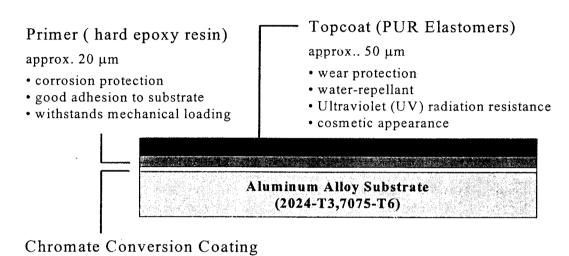


Figure 1: Schematics of coating system

In the scope of this work a series of Al 2024-T3 specimens with different epoxy primer-thickness have been examined. Thus, we first concentrate on a single layer system.

• improves corrosion protection of natural Al<sub>2</sub>O<sub>3</sub> by adding corrosion inhibitors

## Scanning Acoustic Microscopy

• self-healing

The characterization of multi-layered structures using Scanning Acoustic Microscopy is classically performed by time-of-flight spectroscopy in the pulsed regime. The method is very easy to apply. An ultrasonic wave generated by a piezoelectric transducer is sent into the test material; the same probe detects the reflected waves [3,4]. The coupling between transducer and sample is achieved by submerging both in a water bath (figure 2). In this experiments a focused 140 MHz was used. Focusing and high frequency of the excited wave and the scanning technique provide the possibility of high-resolution measurements (approx. 10 µm).

The acoustic wave pulse travels through the different material as indicated in figure 2. The time difference between the detection of two reflected waves, the so-called time-of-flight, is measured. For known sound velocity, the thickness can be determined with equation 1.

$$d_{coat} = \frac{1}{2} v_{coat} \cdot tof \tag{1}$$

Employing own measurements on specimens with known thickness we determined the sound velocity of epoxy to be 2250 m/s and that of 2024-T3 Al-alloy to be 6240 m/s.

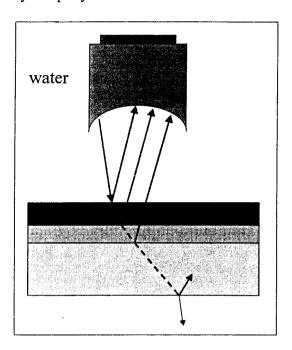


Figure 2: Schematics of pulse/echo scanning acoustic microscopy

## **Continuous Acoustic Waves**

In our experiment a network analyzer was used to generate, receive and analyze the continuous acoustic waves. A sample and transducer combination was connected to the network analyzer. The instrument treats the sample-transducer combination as an electrical network. One of the parameters of the network,  $S_{11}$ , defined as the ratio of the reflected energy to the input energy, is measured as the frequency is swept across the bandwidth of the transducer. At certain frequencies, corresponding to a resonance of the specimen, minima in the amplitudes of the reflected waves can be observed. Resonance occurs, if the total sample thickness is a multiple of half the acoustic wavelength  $\chi/2$ . Then a standing wave is created, as illustrated in figure 3.

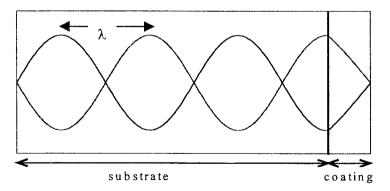


Figure 3: Schematics of standing wave in coated sample for certain resonance frequencies  $f_{res}$ , if total thickness is multiple of  $\lambda/2$ 

At the substrate/coating interface the wave changes its velocity. Several minima are observed within the bandwidth of the transducer, superposed with the transducer response. The

total thickness can be determined according to equation 2, with the known, average velocity (averaged over Al-alloy substrate and coating velocity) and by measuring the difference between two successive resonance frequencies (successive minima),  $\Delta f_{res}$ . From this equation, the coating thickness can be derived (equation 3).

$$d_{tot} = \frac{1}{2} \overline{v} \cdot \frac{1}{\Delta f_{res}} \qquad \text{with} \qquad \overline{v} = \frac{2 \cdot d_{tot}}{\left(\frac{2 \cdot d_{alu}}{v_{alu}} + \frac{2 \cdot d_{coat}}{v_{coat}}\right)} \qquad (2)$$

$$d_{coat} = v_{coat} \cdot \left(\frac{1}{2 \cdot \Delta f_{res}} + \frac{d_{alu}}{v_{alu}}\right) \qquad (3)$$

For the continuous wave measurements a flat 10 MHz-transducer was used. The transducer was bonded to the sample with a thin layer of grease. In the measurements, corrections to the bond thickness have not been included. The polished substrate surface has thickness variations of approx. 2  $\mu$ m, which are not negligible. However, the averaging over areas in the mm²-range compensates this influence.

## **RESULTS**

Figure 4 shows the amplitude of the reflected wave as a function of time. Increasing coating thickness can be evaluated by increasing time-of-flight between the peak reflection from the coating surface and the peak-reflection from the substrate/coating interface. Furthermore, a decrease of amplitude for the interface reflection can be observed.

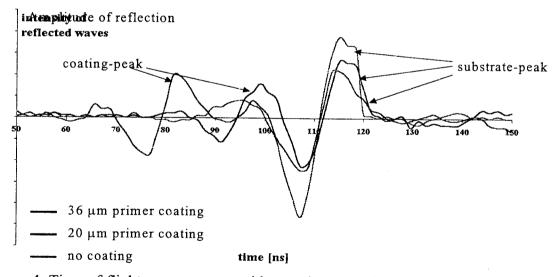


Figure 4: Time-of-flight measurements with scanning acoustic microscopy

However, the different reflections can only be distinguished down to a certain coating thickness. This can be demonstrated in figure 5. Here, so called B-scans for specimens with primer coating of 36  $\mu$ m, 20  $\mu$ m and 10  $\mu$ m are presented. A B-scan shows the time-of-flight along one scanning direction. The 10  $\mu$ m coating can no longer reflect a separable peak.

An additional disadvantage is the need to submerge both probe and specimen in water. This disqualifies the method as in field application. Thus, new efforts are focused on the

continuous wave technique with a sensor in contact to the sample. Figure 6 shows the amplitude of the reflected waves as a function of wave frequency. For decreasing coating thickness the resonance frequencies change and  $\Delta f_{res}$  increases.

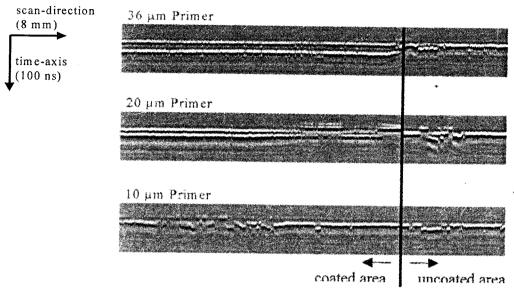


Figure 5: B-scans for primer coatings of different thickness (white lines indicate peak reflections from sample)

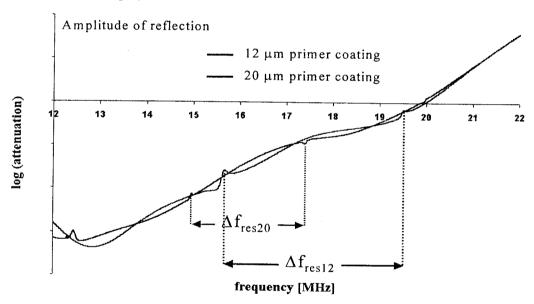


Figure 6: Amplitude of reflected waves as function of frequency for continuous wave measurements

With equation 3, we can calculate the coating thickness from the measured  $\Delta f_{res}$ -values. The thickness as function of  $\Delta f_{res}$  is presented in figure 7. An almost linear relation can be observed. Further calculations revealed that above a certain thickness (60  $\mu m$  in our experiment for the epoxy) resonance occurs within the coating, i.e. the thickness crosses the  $\chi$ 2-value. The additional standing waves interfere with those for the complete specimen and the measurements are no longer reliable. Thus, an upper thickness limit for each coating layer has to be considered.

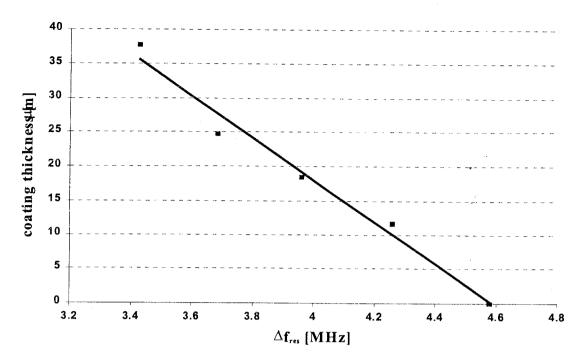


Figure 7: Primer coating thickness as function of  $\Delta f_{res}$ 

#### **CONCLUSIONS**

Continuous acoustic wave measurements can be considered to be a good alternative for acoustic measurements in the pulsed regime. Since no inconvenient water bath is needed and the measurements are valid for the most interesting thickness range below 50  $\mu$ m, they have a much higher potential as in field application. Future work has to concentrate on an improvement of the lateral resolution. Furthermore multi-layer-coating systems will be examined.

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