Metal coatings analysis using the handheld Agilent 4100 ExoScan FTIR

In situ anozidation thickness measurement

Application Note

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Abstract

Infrared (IR) spectroscopy provides a non-destructive means of identifying and quantifying anodized coatings, as well as detecting if parts have been powder or Teflon coated. The anodized aluminum oxide surface provides a strong IR signature that can be easily quantified and, depending on the anodization process employed, the IR spectrum of the aluminum oxide coating changes significantly.

Since FTIR spectroscopy is typically carried out in a laboratory, measurement of the anodized layer by reflection IR spectroscopy has not provided a significant advantage over other analysis techniques for articles that are very large. These oversized samples need to be destructively cut up.

But Agilent has developed the portable, handheld 4100 ExoScan FTIR, capable of measuring anodization thickness and type on large parts in situ. The handheld nature of the 4100 ExoScan enables even large parts to be measured in any orientation. Customized optics are designed to obtain an optimum focus when the 4100 ExoScan is placed in contact with the sample, facilitating easy measurements.

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With the 4100 ExoScan, large parts can be easily, non-destructively qualified, ensuring that they meet their design criteria in critical applications.

Introduction

Nearly all metal surfaces are coated in order to increase corrosion resistance, improve bonding to other surfaces or improve hardness. As the use of new alloys grow, the need to protect those materials from oxidation using the correct type and thickness of coating gains increasing importance. Anodization is a process that is used to protect metal surfaces. By increasing the thickness of a natural oxide layer, the anodizing process increases corrosion resistance and wear resistance of a metal surface. It is often used on aluminum parts; the aluminum oxide formed by the electrostatic passivation provides a smooth, durable layer. This is especially important for high strength aluminum alloys, due to the increased corrosion caused by the other metals present. Other metals such as titanium, zinc and magnesium are also anodized to improve specific properties.

Anodized metals have very different properties than the un-oxidized base metal. These properties are crucial for the metal part to meet its design criteria, especially when used in a high performance application. One factor that determines these properties is the thickness of the oxide layer. The current applied and the time over which it was applied determines the thickness of the resulting coating; therefore, it is important to determine that the correct coating thickness has been obtained on critical parts. Additionally, there are several different types of anodization based mostly on differences in the electrode baths in which the processes take place. Examples include chromic acid anodization, sulfuric acid anodization or borate sulfuric acid anodization. Each process produces slightly different properties, but may not result in a visible difference between parts. Additionally, parts are often post-treated after anodization. They may be powder coated, Teflon coated or hardened to achieve specific properties. Many of these post-anodization processes are not detectable by visible inspection only. There is a need to verify that the anodization process has been carried out correctly and that any further modification of the surface has been properly completed before using a part in a critical application.

Typically parts are destructively analyzed in order to identify and determine the thickness of an anodized coating. Atomic spectroscopy is the primary method used to determine whether parts were sulfuric acid or chromic acid anodized. This is done by looking for peaks due to contaminants from the acid bath remaining in the coating. Unfortunately, alloys contain small amounts of chrome and sulfur unrelated to the passivation process, making this technique non-definitive. Various techniques are available to determine the thickness of the anodization. One popular method is a gravimetric technique. The coating is first dissolved in acid, and then the amount of coating is determined by weighing the part before and after the coating has been dissolved. This analysis is time consuming, error prone and uses hazardous chemicals. Due to the destructive nature of these tests, they are often not carried out even though the wrong thickness or process could greatly reduce the serviceability of the metal part.

Anodized surface measurement by the Agilent 4100 ExoScan FTIR

The small size and portability of the 4100 ExoScan FTIR enables measurement of the sample directly in the field. The 4100 ExoScan has two available sample interfaces. The internal reflectance interface (ATR) is used for highly absorbing or non-reflective samples. For analysis of anodized coatings, the external reflectance sample interface is used. The IR light from the 4100 ExoScan is reflected from the sample at an angle of 45 degrees and then collected by the sampling optics. Samples can be measured over the full mid-infrared range from 4000 to 650 cm^{-1} at a maximum resolution of 4 cm^{-1}. For the anodization coating measurements in this study, 8 cm^{-1} resolution was used.

The 4100 ExoScan software provides multiple levels of user interaction. The Administrator level allows full use of the system to develop methods, including advanced data processing. The system is designed to wirelessly communicate with either a PDA for data collection, or a laptop computer. Method development personnel can collect the data on either the PDA or laptop. If data was collected with the PDA, it can be wirelessly transferred to the laptop for advanced processing. Once the method has been developed, the Operator level of software allows simple data collection, automated data analysis and presents easy to understand results that are displayed on the PDA. This allows the system to be used with very little training. The methods can be set up to give users a simple yes/no answer when looking for a specific type of coating or thickness.

Aluminum anodization thickness

Five samples of 2024 aluminum that were treated with borate sulfuric acid anodization (BSAA) were measured with the 4100 ExoScan FTIR. The aluminum oxide surface concentration ranged from 66 to 374 mg/ft^2. The overlaid spectra of the five samples are shown in Figure 1. The strong aluminum-oxygen stretching band is observed at 1128 cm^{-1}. Both the height of this band and the area of this band using local baseline points at 1390 and 995 cm^{-1} were plotted with respect to aluminum oxide concentration. Both plots are easily fit with quadratic equations, each producing a correlation of 0.995. The calibration plots are shown in Figure 2.

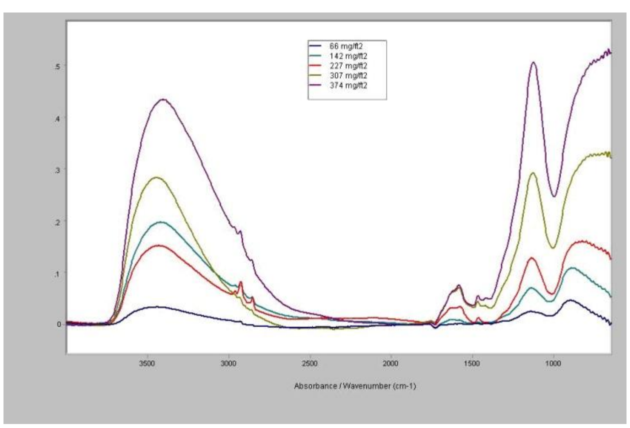

Figure 1. Spectral overlay of BSAA on 2024 aluminum calibration standards. Spectra were measured with 32 scans at 8 cm^{-1} resolution with the Agilent 4100 ExoScan FTIR using external reflectance

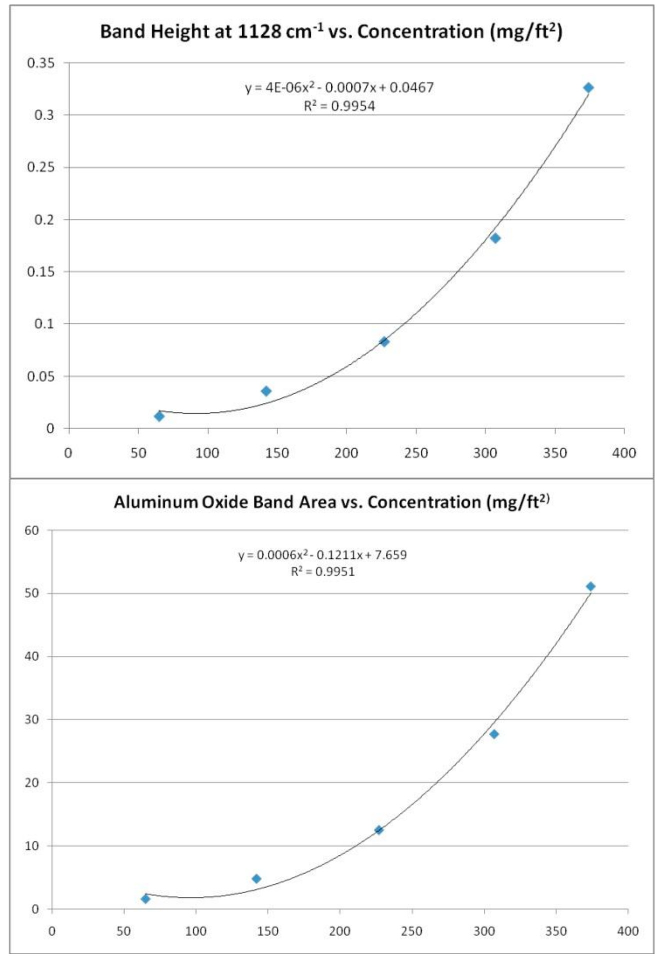

Figure 2. Calibration curves showing the aluminum oxide band height at 1128 cm^{-1} versus concentration (top) and the aluminum oxide band area from 1390 cm^{-1} to 995 cm^{-1} versus concentration (bottom). Both calibration curves are fit by a quadratic equation with an excellent correlation coefficient.

IR spectra of the anodized coating are independent of the aluminum alloy. Figure 3 shows samples of 7075 aluminum that have been treated with the same BSAA process as the 2024 samples above. The calibration in Figure 2 was used to predict the concentration of aluminum oxide; the results are shown in the legend of Figure 3.

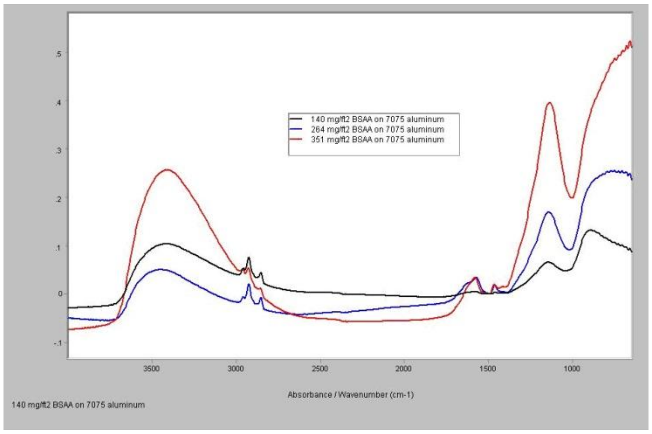

Figure 3. IR spectra of BSAA on 7075 aluminum measured with the Agilent 4100 ExoScan FTIR. Concentrations of anodized coating calculated from the spectra are shown in the legend

Coating type identification

The IR spectrum of the anodized coating is not characteristic of the alloy to which it is applied; rather, the spectrum is characteristic to the coating process. Each coating process produces a slightly different crystal size and shape. These slight differences produce changes in the IR spectra, which can be correlated to the coating type. Using a library search approach, one can determine the anodization process used on a particular part from the IR spectrum. With the 4100 ExoScan FTIR, this can be done on large parts without the need for disassembly or destruction of the part.

Figure 4 shows the spectra of three different aluminum samples that were treated with BSAA. The first sample is 2024 aluminum alloy sheet, the second is 7075 aluminum alloy sheet and the third is a cast aluminum part. It should be noted that the two aluminum alloy sheets had a very smooth surface, but the cast part had high amount of surface roughness. This shows that spectra of the anodization coating can be obtained even from low reflecting surfaces.

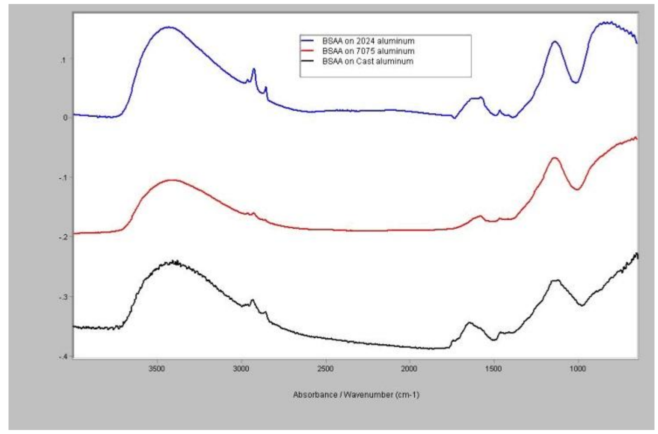

Figure 4. Comparison between spectra of BSAA on 2024 aluminum (blue), 7075 aluminum (red) and rough cast aluminum (black)

Spectra of samples from three different types of aluminum anodization processes were measured. Each process produces slightly different properties in the metal part. The processes used were BSAA, sulfuric acid anodization, and chromic acid anodization. Each anodization produced a distinctly different spectrum as is shown in Figure 4. Figure 5 shows a library search carried out in the 4100 ExoScan FTIR software identifying a BSAA anodization type on a cast aluminum part.

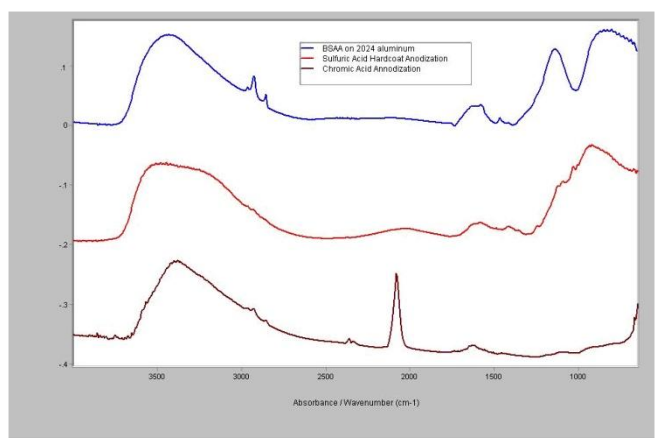

Figure 5. IR spectra of BSAA anodization (blue), sulfuric acid hardcoat (red) and chromic acid anodization (brown)

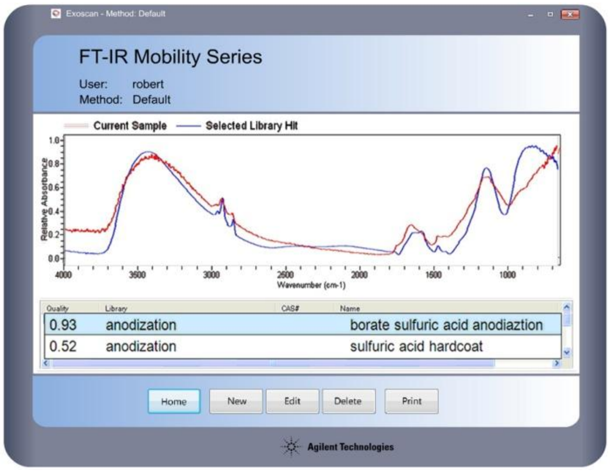

Figure 6. Library search in the Agilelnt 4100 ExoScan FTIR software showing a positive match for the BSAA process

Conclusion

A variety of coatings are used in order to ensure corrosion resistance and durability of metals for high performance applications. In order to ensure that the metal parts will perform as designed, it is important to verify both the type of coating used, and the thickness of that coating. IR spectroscopy can identify many coatings used on aluminum and other metals. Even thin anodized coatings, as shown here, can be both identified and quantified using the 4100 ExoScan FTIR. The 4100 ExoScan can also be used to identify paint and primer coatings[1]. Since the 4100 ExoScan is handheld, portable and designed to be used directly where the article of interest is located, it permits measurement of coatings on these large complex parts without disassembling or destroying the parts. The 4100 ExoScan is a very useful quality control device for ensuring that incoming parts have the proper type of coating with the correct thickness for the intended application.

For more details on paints and primers, see Agilent application note 'First article and incoming product inspection of paints and plastics using the handheld Agilent 4100 ExoScan FTIR'.

In addition to the 4100 ExoScan FTIR, Agilent also offers the 4200 FlexScan. The 4100 ExoScan and 4200 FlexScan both provide easy, handheld FTIR analysis, but with slightly different form factors. The 4200 FlexScan has the same optical components as the 4100 ExoScan, but the optics and electronics are separated by a cable. This makes the handheld component smaller while still providing the spectroscopic performance needed for a wide variety of applications. The 4200 FlexScan has a 3 pound optical head attached to a 4 pound battery and electronics pack. Although the form factor is different, use of the two systems, including the software, is identical. While the 4100 ExoScan provides an integrated, compact package, the 4200 FlexScan has a smaller size to fit into spaces with tight clearances

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