Direct Measurement of Trace Contamination on Metallic Surfaces Using ATR/FTIR

Introduction

Hydrocarbon surface contamination on rolled aluminum sheets is generally caused by lubricants used to control friction during processing. Despite de-oiling treatments, oil residues may remain and its detection is often paramount prior to downstream processing. In the case of radiators, condensers, and evaporators, oil contamination on aluminum prevents proper brazing. When this occurs, these types of manufactured components are prone to leak. Therefore, a fast method to evaluate contamination on aluminum that requires no sample preparation is desirable.

Materials and Methods

Aluminum samples, 300 microns thick, were treated and analyzed via ATR/FTIR. Four sample treatments were investigated: 1) as received from the aluminum manufacturer; 2) after cleaning with dichloromethane; 3) after lightly coating the inside surface with typical lubricating oil, heating the sample for about 1 hour at 150 ºC ; and 4) subjecting treatment #3 to additional heating for 1 hour at 350 ºC. ATR measurements were taken on the inside surface of the aluminum.

Figure 1. PIKE GladiATR (shown with optional heating controller).

The PIKE GladiATR (Figure 1) with monolithic diamond crystal was used in this investigation. The aluminum sample was placed over the diamond crystal, and high pressure was applied. Spectral range covered 4000 cm^{-1} to 400 cm^{-1}. Spectra were collected at 4 cm^{-1} spectral resolution using 1 minute data collection times. Using ATR, no sample preparation was required.

Results

ATR provides versatility in the laboratory environment as it is suitable for analyzing a wide variety of materials including powders, liquids, and solids. Using this technique, it is critical that intimate contact be made between the ATR crystal and the sample. Clearly, this is not an issue when testing liquids. Example spectra of two brands of aluminum cutting fluid measured using the GladiATR are shown in Figure 2. However, achieving suitable contact between an ATR crystal and a rigid material is challenging. The GladiATR design addresses this issue by delivering extremely high pressure, greater than 30,000 psi, to the sample while the diamond crystal, brazed in place, is able to withstand such pressures. Other benefits of the monolithic diamond are its extreme hardness, its suitability for all pH values, and its full spectra range.

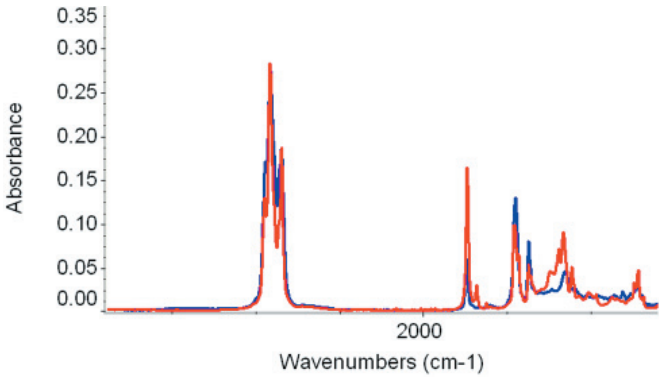

Figure 2. Spectra of two aluminum cutting fluid brands.

In this study, the effect of applied pressure on the spectral quality was evaluated by applying full pressure (>30,000 psi) and half pressure (estimated at 15,000 psi) to the aluminum sample treated with a thin layer of oil and subjected to 150 ºC for about 1 hour (treatment #3). Varied pressures were obtained by manually adjusting the high pressure clamp knurl. Significant enhancements in the hydrocarbon absorbance bands between 3000 to 2800 cm^{-1} were observed when maximum pressure was applied, which are especially important when analyzing trace amounts of residue on metal surfaces (Figure 3).

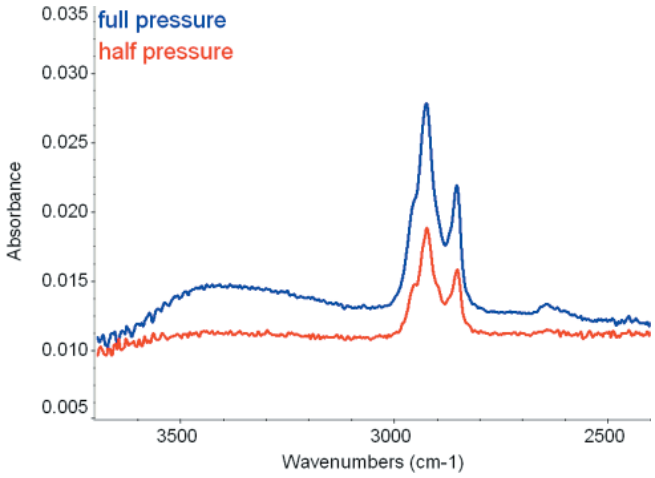

Figure 3. Effects of varying applied pressure (full and half) on spectral quality.

Figure 4 compares spectra from each treatment. Hydrocarbon bands are present for each sample; peak heights are listed in Table 1. Treatment #1 showed slight hydrocarbon residue on the aluminum as recieved. Cleaning with dichloromethane, as performed in this experiment, had minimal effect upon removing the residue as determined by a reduction in peak height of 0.4 mAU. These data show that ATR/FTIR could be used as a screening quality tool for incoming materials or as a tool to evaluate de-oiling processes in manufacturing, for example, by setting an acceptable peak height limit at 2925 cm^{-1}.

In this study, additional heat treatment (350 ºC for 1 h) after applying a thin oil film diminished the

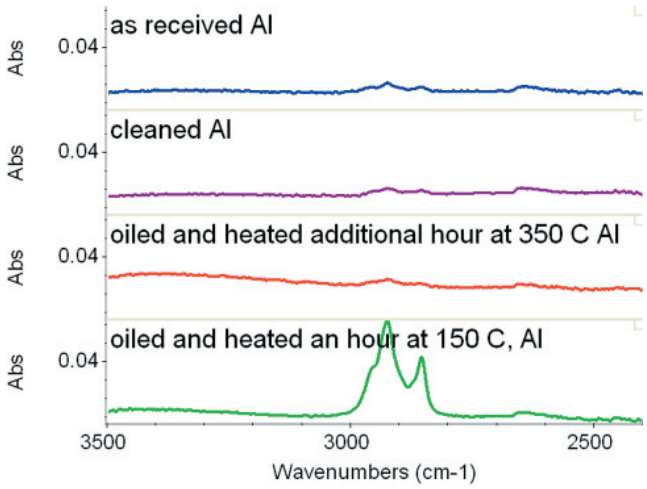

Figure 4. Spectral comparison of aluminum sample treatments, shown at identical y-scaling.

residue considerably resulting in a peak height reduction of 19.6 mAU between treatment #3 and #4. The level of contamination after subjecting to high heat was similar to that of cleaning with dichloromethane or as that received directly from the manufacturer.

Summary

Using the GladiATR, aluminum samples were analyzed for hydrocarbon surface contamination without the need for sample preparation. *The high pressure applied to the sample, a key feature of the GladiATR, was imperative to the success of this application.* This sampling technique offers fast results to assess residual oil contamination. The methodology presented could easily be adapted as an incoming materials screening test, or as a processing evaluation tool.

Table 1. Peak height of hydrocarbon band located at 2925 cm^{-1}.

Treatment #	Description	Peak Height (mAU)
1	As received	2.0
2	Cleaned	1.6
3	Oiled; heat at 150 ºC	21.0
4	Oiled; heat at 350 ºC	1.4