Identification of Contamination on Welding Wires Using Cross-Platform Techniques in SEM and Mass Spec

Introduction

A batch of contaminated welding wire received from a vendor by a customer was causing problems in a manufacturing process. Visual comparison of the clean and contaminated wire did not show any obvious differences, but the contamination was readily observed on backscatter electron images obtained with the JEOL IT300 scanning electron microscope (Figure 1).

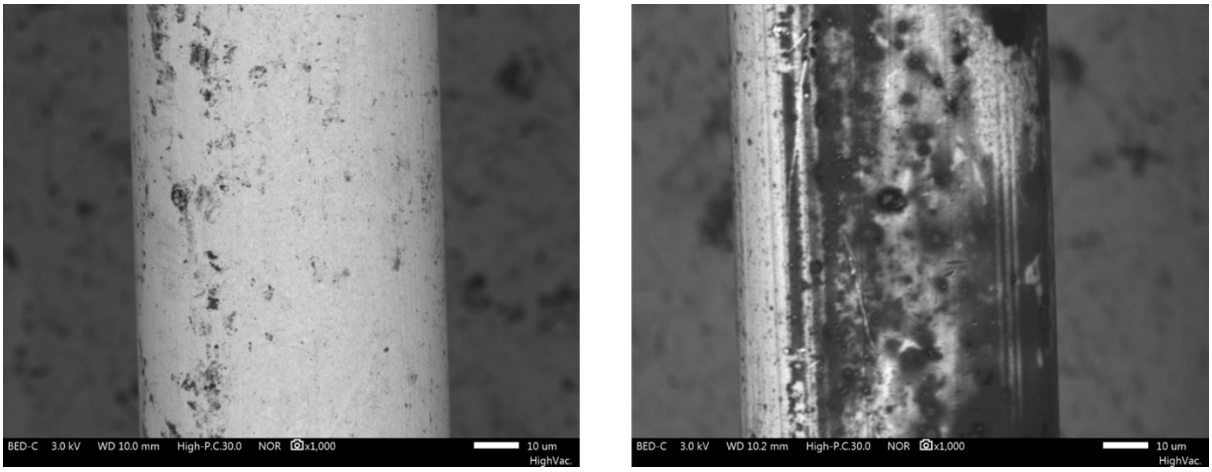


Figure 1. Backscatter electron images obtained at 3 kV of clean welding wire (left) and contaminated wire (right).

A JEOL *AccuTOF™-DART®* mass spectrometer equipped with a Biochromato *IonRocket™* thermal desorption and pyrolysis attachment were used to determine the chemical composition of the wire contamination. The *IonRocket* includes an oven and disposable sample holders that permit controlled heating of samples from ambient temperature up to 600°C. The resulting thermal desorption profiles facilitate the identification of volatile compounds and outgassing. At higher temperatures, pyrolysis products permit the identification of materials such as polymers.

Experimental

Mass spectra were acquired in positive-ion mode with the DART ion source on the JEOL *AccuTOF-DART 4G* high-resolution time-of-flight mass spectrometer. Mass spectra were acquired with JEOL Mass Center software and data processing (mass calibration, spectral averaging, background subtraction, and mass chromatogram displays including 3D plots) was accomplished with TSS Unity software (Shrader Software Solutions, Detroit, MI). Mass spectral interpretation and compound identification were carried out with Mass Mountaineer software (mass-spec-software.com).

Scissors were used to cut small segments of wire (a few millimeters in length) that were placed onto the disposable copper sample stage for the *IonRocket.* Figure 2 shows the wire segments on the surface of the copper sample stage both before and after heating. The *IonRocket* was oven was positioned directly under the DART ion source, and a glass tee was used to sample the gases desorbed from the sample into the DART helium gas stream. The *IonRocket* was programmed with a temperature ramp to heat the sample from ambient temperature to 600°C in 6 minutes.

Figure 2. Segments of welding wire placed on the surface of the copper sample stages for the IonRocket. The left figure shows the wires placed on a fresh, clean sample stage, and the right side shows wire segments on the surface of a copper sample stage that was oxidized after heating to 600°C.

Results

The 3D plots for clean and contaminated wires shown in Figure 3 provide an overview of the mass spectral analysis. The x-axis for each 3D plot indicates time and temperature. At a heating rate of 100°C min^{-1}, 2 minutes is equivalent to 200°C, 4 minutes corresponds to 400°C, and 6 minutes corresponds to 600°C. The y-axis represents the mass-to-charge ratios (*m/z*), and darker spots indicate higher relative abundance for the detected ions.

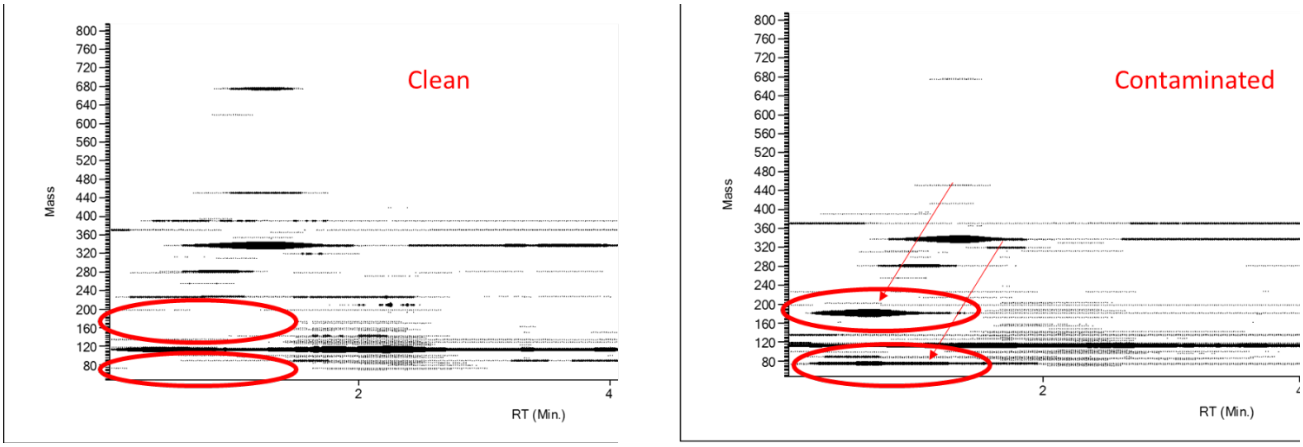


Figure 3. Comparison of the 3D plots for thermal desorption of clean (left) and contaminated (right) wires shows the presence of peaks at nominal m/z 76 and m/z 182 in the contaminated wires that are not present in the clean wires.

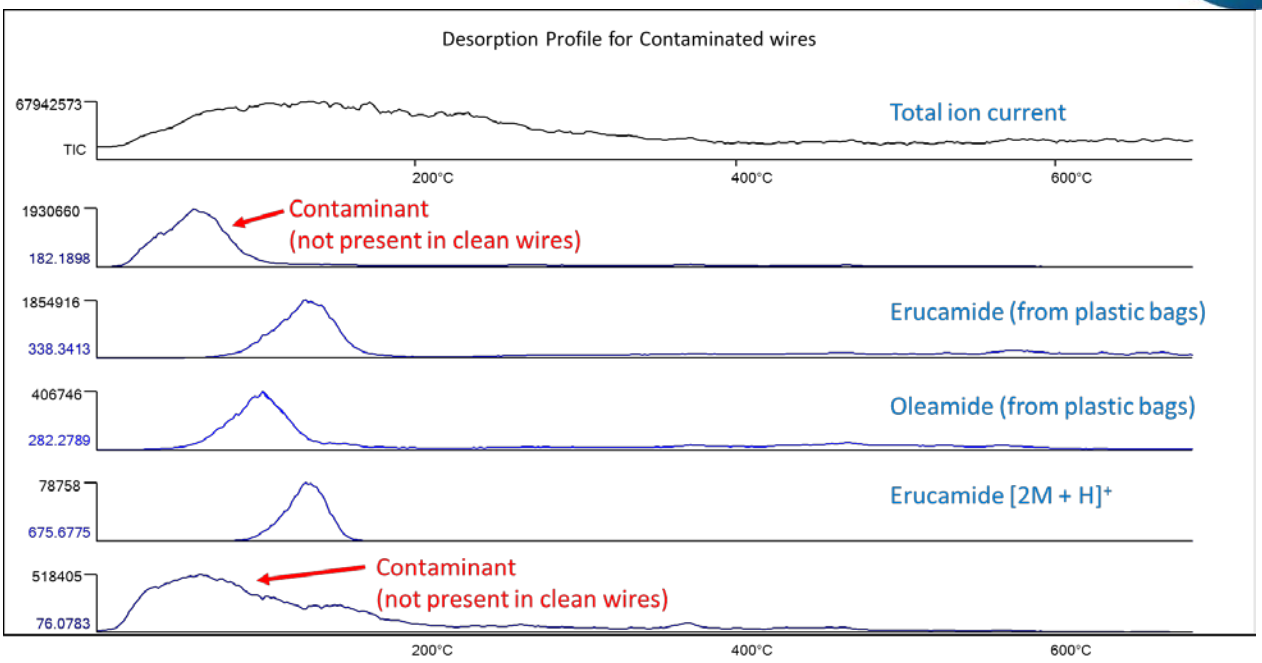


Figure 4. Thermal desorption profiles for selected components. Erucamide and oleamide are slip agents found on both clean and contaminated welding wires from the plastic bags used to ship the wires. The peaks at m/z 76.0783 and m/z 182.1898 were observed on the contaminated wires, but not on the clean wires.

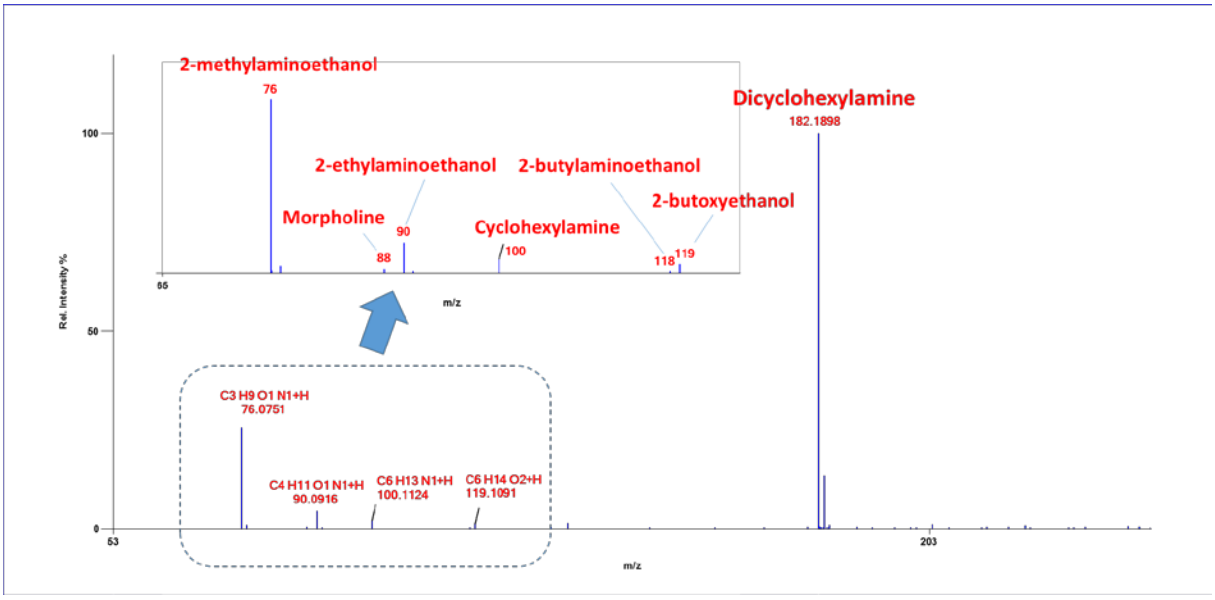
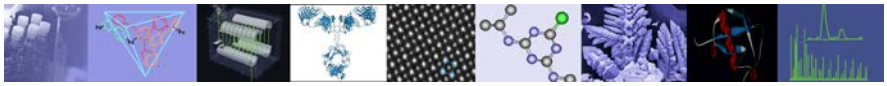


Figure 5. AccuTOF-DART mass spectrum of the contaminated wire at an IonRocket sample heater temperature of 65°C showing the presence of compounds used in metalworking fluids.



The temperature profiles for selected contaminants in the plastic bags containing the welding wires and the compounds found only on the contaminated wires are shown in Figure 4. Examination of the mass spectra at time 0.6 minutes shows that major peaks are present in the contaminated wire, but not in the clean wire, at *m/z* 182.1898 and *m/z* 76.0751. The mass spectrum in Figure 5 shows the peaks present in the contaminated wire that are not present in the clean wire. Exact mass measurements with isotope matching provide elemental compositions for each peak that are consistent with compounds used in metal working fluids. For example, the base peak at *m/z* 182.1898 is identified as protonated $C_{12}H_{23}N$ (dicyclohexylamine), the peak at *m/z* 76.0751 is identified as protonated C_3H_9NO (2-methylaminoethanol) and other compounds are tentatively identified as protonated 2-ethylaminoethanol, 2-butylaminoethanol, morpholine, 2-butoxyethanol, and cyclohexylamine based on their elemental compositions.

Conclusion

The combination of SEM and Mass spectrometry, particularly *IonRocket* and *AccuTOF-DART,* permitted the identification of residual metalworking fluid on the contaminated samples, indicating that the wires were not adequately cleaned after fabrication.

While the contaminants could be detected with DART alone, the *IonRocket* facilitated handling for the small wire samples, permitted the analysis of multiple wire segments in a single measurement, and provided temperature-dependent desorption information about the contaminating compounds.

The Scanning Electron Microscope provides high magnification imaging of samples to identify quality control issues and reveal details that cannot be seen with the naked eye or optical microscope.

The *AccuTOF-DART* mass spectrometer with the *IonRocket* thermal desorption and pyrolysis accessory is a powerful tool for investigating problems related to materials and contamination.