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Long-Term Effects of Soldering By-Products on Nickel-Coated Copper Wire

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October 2008

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LIST OF ACRONYMS

EDS	energy dispersive spectroscopy
FTIR	Fourier Transform Infrared

NOMENCLATURE

C-H	carbon-hydrogen bond
C-O	carbon-oxygen bond
O-H	oxygen-hydrogen bond
R	generic organic
R-COO-Cu-OOC-R	copper abietate
R-COOH	carboxylic acid

TECHNICAL MEMORANDUM

LONG-TERM EFFECTS OF SOLDERING BY-PRODUCTS ON NICKEL-COATED COPPER WIRE

1. INTRODUCTION

Rosin, a material obtained from conifer trees, is a flux material typically incorporated into the cores of solder wire. This material has been used for years to aid in the solder wetting process by removing surface oxides from the materials to be soldered. The primary constituent of commercial rosin is a carboxylic acid known as abietic acid.¹ The removal of surface oxides occurs when this carboxylic acid is converted to a metal ester called abietate. The reaction that leads to the abietate form is illustrated in figure 1.² Although soldering requirements usually state that the rosin residue should be removed from soldered areas, usually cleaned using isopropyl alcohol, it is possible that the material can be wicked under the insulation of wires by capillary action. The alcohol itself can assist with the wicking and subsequent deposition of flux residue under the insulation. Excessive wicking is prohibited by soldering requirements, but many times the wicked material is not detected due to the opaqueness of the insulation. When copper wire is part of the soldering process the reaction product is copper abietate, which is green in color. However, copper (II) chloride is also green in color and is an unwanted, corrosive contaminate. The purpose of this study is to determine the composition of the green material found on thirty-year-old, down graded flight cables and to establish the long-term corrosive effects of this material on nickel-plated copper wire.

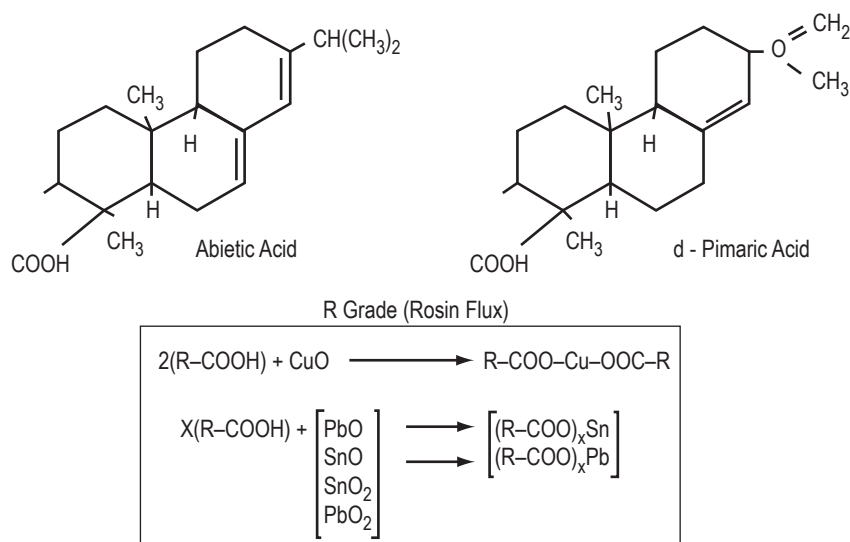


Figure 1. Reaction of copper oxide with abietic acid produces a water-insoluble copper soap called copper abietate.

Two wire assemblies were analyzed during this study. Photographs of both wires were taken and the images are shown in figure 2. The shrink-wrap tubing that protects the soldered area was removed along with the wire insulation to ascertain the location and length of green discoloration. The shrink-wrap tubing and insulation were removed from the wire and a high magnification image of clean and contaminated wire is shown in figure 3. The location of the contamination shows that a significant amount of wicking had occurred. The shorter wire has serial number 009 and was designated with two other identifiers: 5104123-5 and P48-55. The nickel-coated copper overall shield portion of this assembly was approximately 2.5 inches long and each of the wire lead-outs was approximately 2.5 inches long making a total length of 5 inches. The contamination on this wire was found to extend from the soldered end to 0.75 inches down the length of the wire. The other wire has a designator of BTB6 and is much longer. The designation identifies the location where the cable is terminated in the box and was used to differentiate the two wires. The total length of the shielded portion of BTB6 is approximately 24 inches and each of the wire lead-outs was approximately 3.5 inches long making the total length 27.5 inches long. The contamination on this wire was found to extend from the soldered end to approximately 1 inch down the length of the wire. Fourier Transform Infrared (FTIR) spectra were acquired from the insulation and from the metal itself, both of which were coated with a green material. An FTIR spectrum of uncontaminated insulation was also taken for comparison. The bare wire was also placed in a scanning electron microscope to conduct an energy dispersive spectroscopic (EDS) analysis.

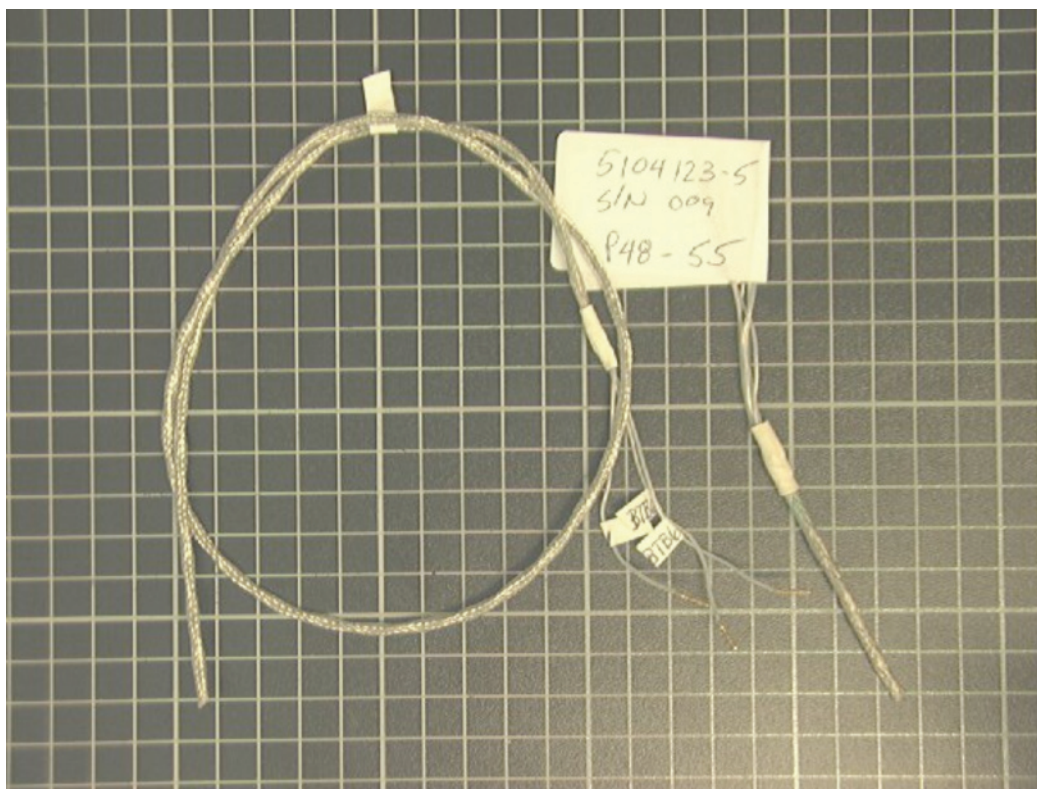


Figure 2. Photograph of wires provided for analysis of corrosion. The smallest boxes that make up the grid in this photo measure 1×1 cm.

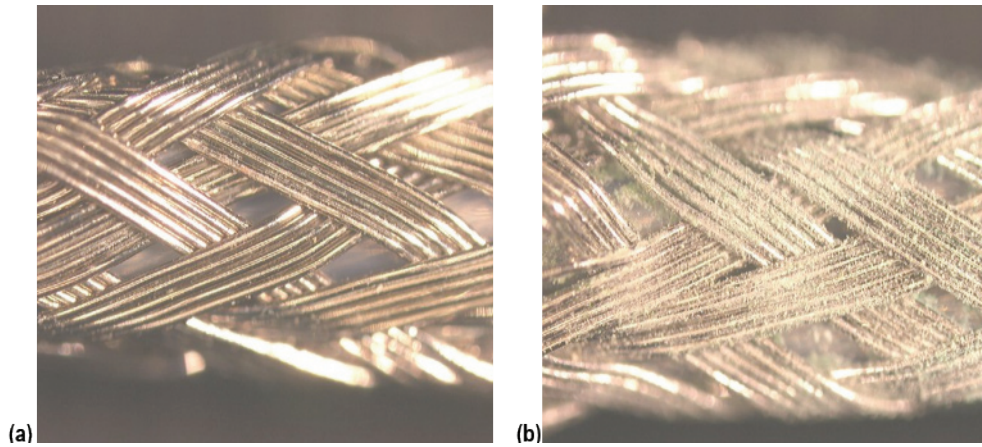


Figure 3. Optical photographs of (a) Clean wire and (b) Wire covered in green material.

A cross section of each section of contaminated wire was performed along with an uncontaminated portion of the same wire. The wires were mounted such that the axial surface was exposed for metallographic preparation. This position was necessary in order to determine if any corrosion was present by measuring the nickel coating thickness in areas exposed to the green material. The wires were potted in a two-part epoxy (Epo-Resin® and Epo-Hardener®, both by Buehler) and allowed to cure overnight without heat or pressure. A standard grit regimen was used to grind the potted samples, namely 240-grit, 400-grit, 800-grit, and 1,200-grit. Grinding was followed by polishing with 6-micron diamond and 0.05-micron alumina.

2. ANALYSIS

Fourier Transform Infrared spectra of the wire insulation and the underlying green material are shown in figure 4. The top spectrum is from an area of insulation where the green material was sandwiched between the insulation and the nickel-coated copper wire. The spectrum reveals distinct polytetrafluoroethylene (PTFE), which is the insulation, fingerprints at 1,147 and 1,201 cm^{-1} . The bottom spectrum is representative of green material from both the insulation and the nickel-coated wire. The green material exhibited characteristic peaks at 1,398 cm^{-1} and 1,606 cm^{-1} that point to an ionized carboxylate stretching mode.^{3, 4} The small peak at 1,249 cm^{-1} results from a C–O stretch that is also associated with the carboxylate structure.⁴ These peak positions are clear evidence that the bulk of the green material is the abietate form. The peak at 1,703 cm^{-1} indicates that unreacted abietic acid may also be present because this position is typical for the carbonyl stretch of a free carboxylic acid.⁵ The typical O–H stretch band for free carboxylic acids is in the 2,700–3,300 cm^{-1} area, but the size and shape of the signature from the green material is not indicative of a strong O–H stretch mode in this band. The O–H stretch peak of free carboxylic acid is generally very strong and broad and in most cases overshadows the C–H stretching peaks, which in this case is at 2,867 and 2,925 cm^{-1} .^{3, 4} The lack of a strong O–H stretch peak is compelling evidence that only small amounts of carboxylic acid are present. In addition, the weaker O–H stretch appears to be shifted upward to between 3,100 and 3,600 cm^{-1} , which is associated with changing hydrogen bonding owing to a decrease of free carboxylic acid moieties in the green material.⁵

Scanning electron microscope images of the material are shown in figures 5 and 6. The material is clearly insulating due to the amount of charging. Energy dispersive spectroscopy was used to analyze the elemental makeup of the green material, as shown in figure 7. The energy dispersive spectrum clearly shows that no chlorides exist. This evidence coupled with the FTIR data strongly indicates that the green material is copper abietate with some small, unreacted abietic acid and not copper (II) chloride.

Cross sections of the wires were obtained so that nickel thicknesses could be measured, as demonstrated in figures 8–10. Typical nickel thickness measurements, acquired using a National Institute of Standards and Technology traceable calibrated optical system, are shown in figure 10. The minimum nickel thickness of 50 microinches (1.27 microns) as called out by the drawings to which these assemblies were built (40M39526, Sheet 5, Section 3.3.2.1) and American Society for Testing and Materials Specification B355 is clearly met. Areas where the contamination was prevalent showed no signs of gross degradation or corrosion even after almost 30 years of residing on the wire surface.

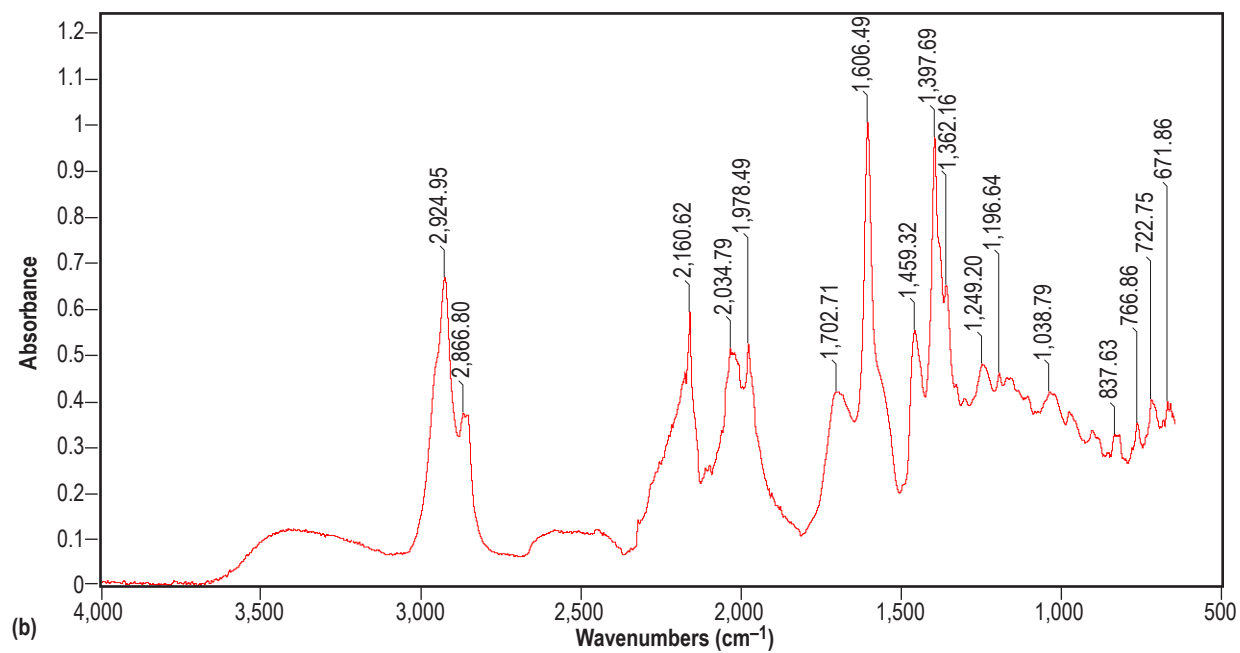
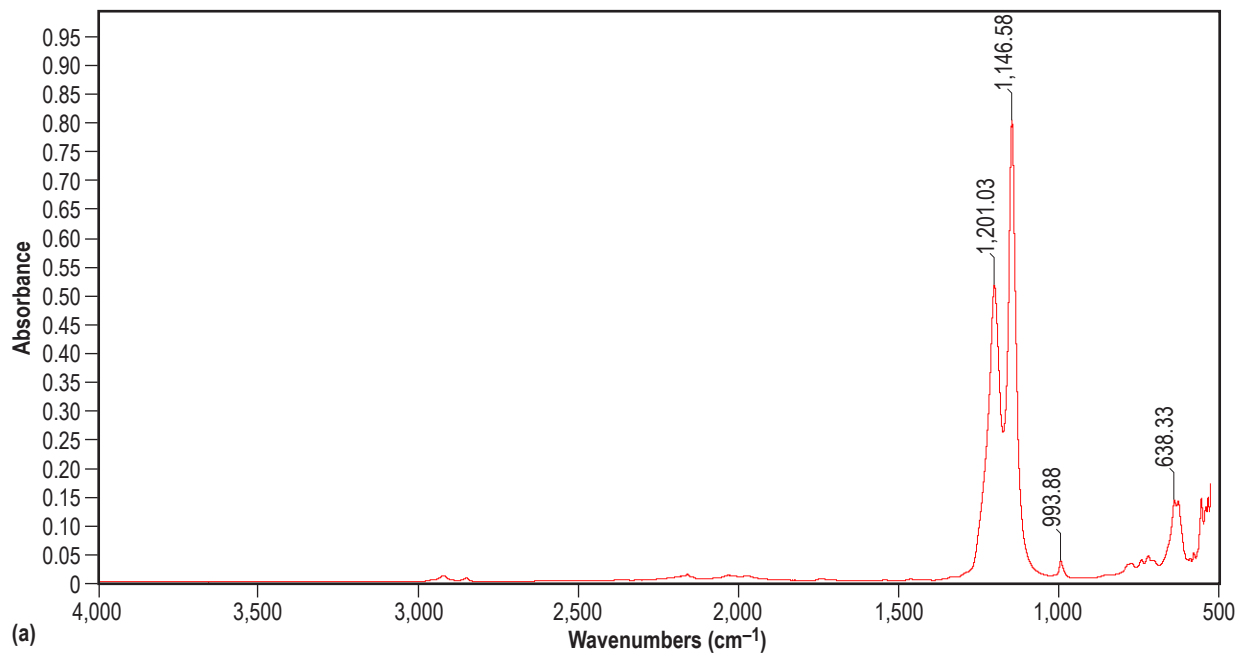


Figure 4. FTIR spectra of (a) Wire insulation and (b) Green material.

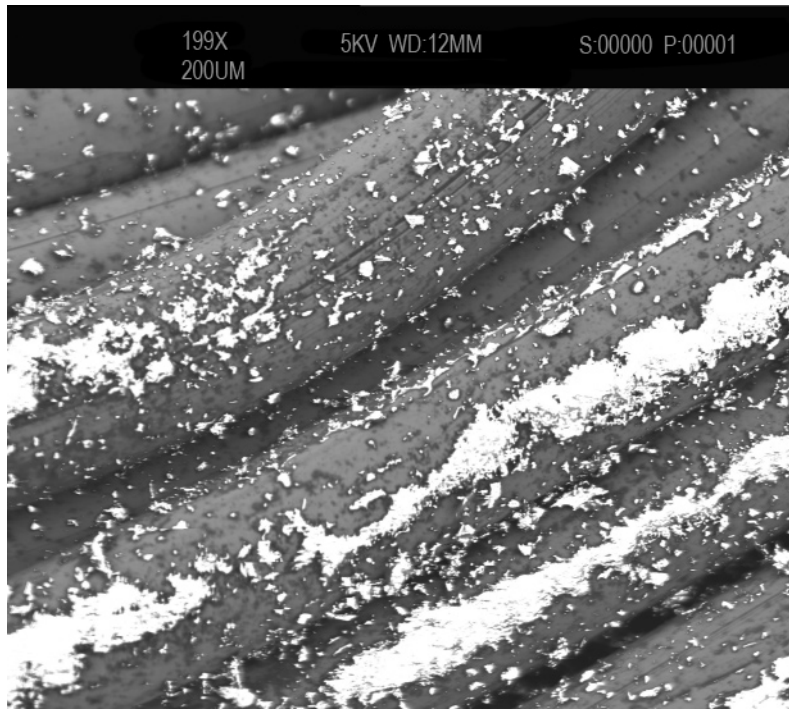


Figure 5. High-magnification image of wire showing the presence of contamination that is low molecular weight and insulating as indicated by the large amount of charging.

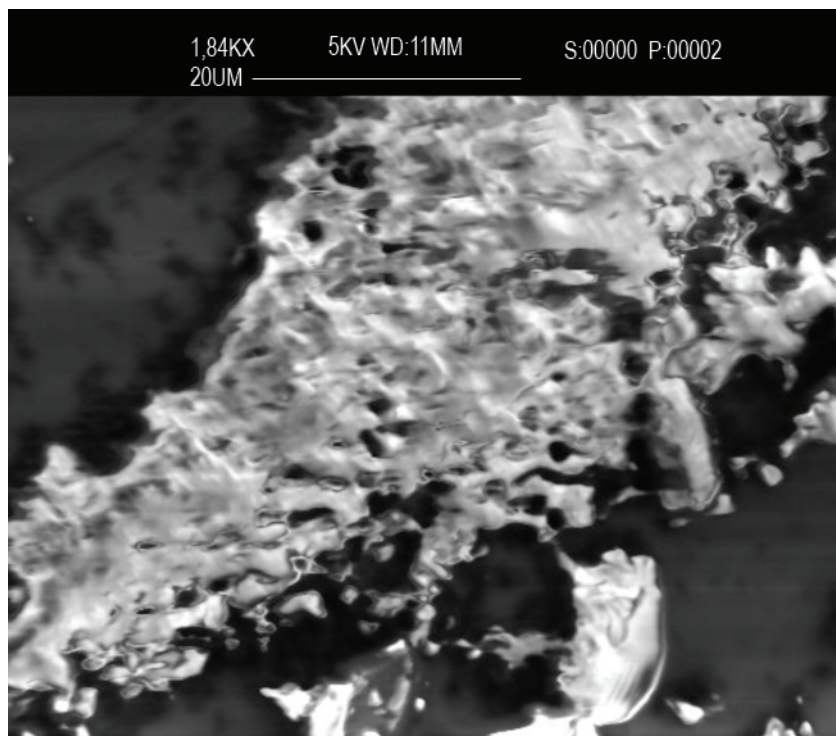


Figure 6. High-magnification image of the contamination from which an EDAX spectrum was taken.

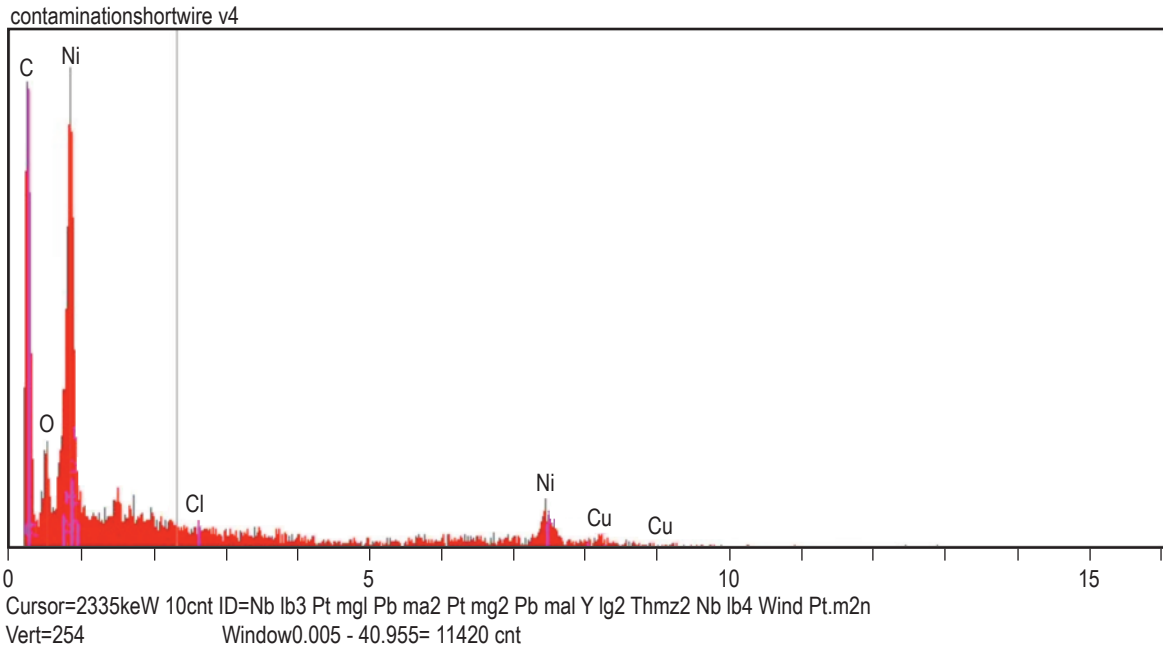


Figure 7. EDS spectrum indicating an organic material and no chlorine contamination. The location of chlorine, had there been a peak, is marked with its symbol, Cl.

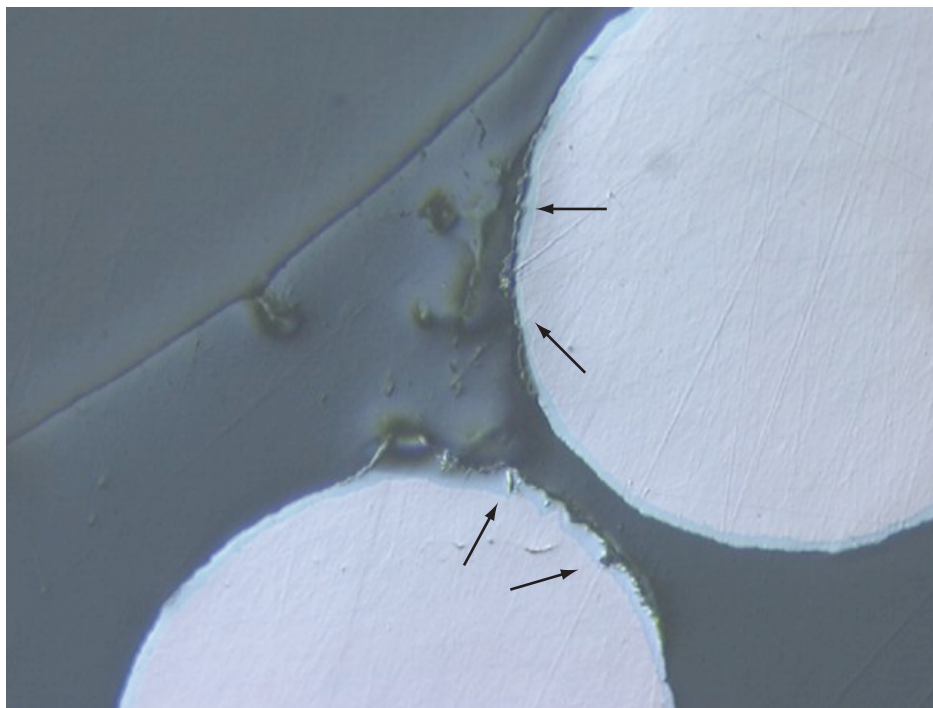


Figure 8. Polarized microscopic image of contaminated area showing an intact nickel coating (black arrows) and no copper degradation ($\times 500$ magnification).

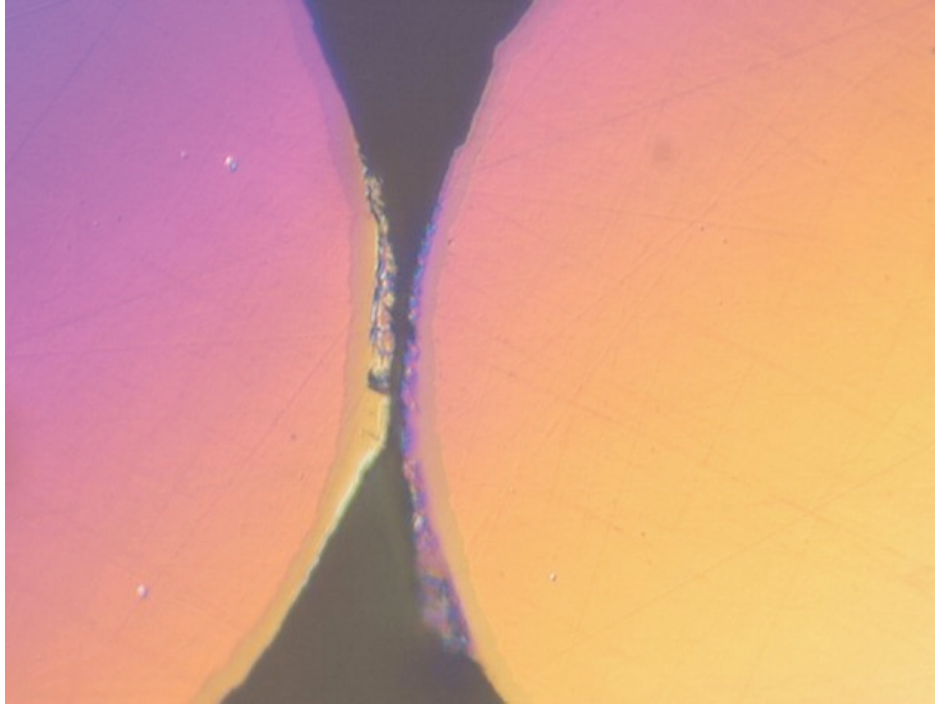


Figure 9. Polarized microscopic image of contaminated area showing an intact nickel coating and no copper degradation ($\times 1000$ magnification).

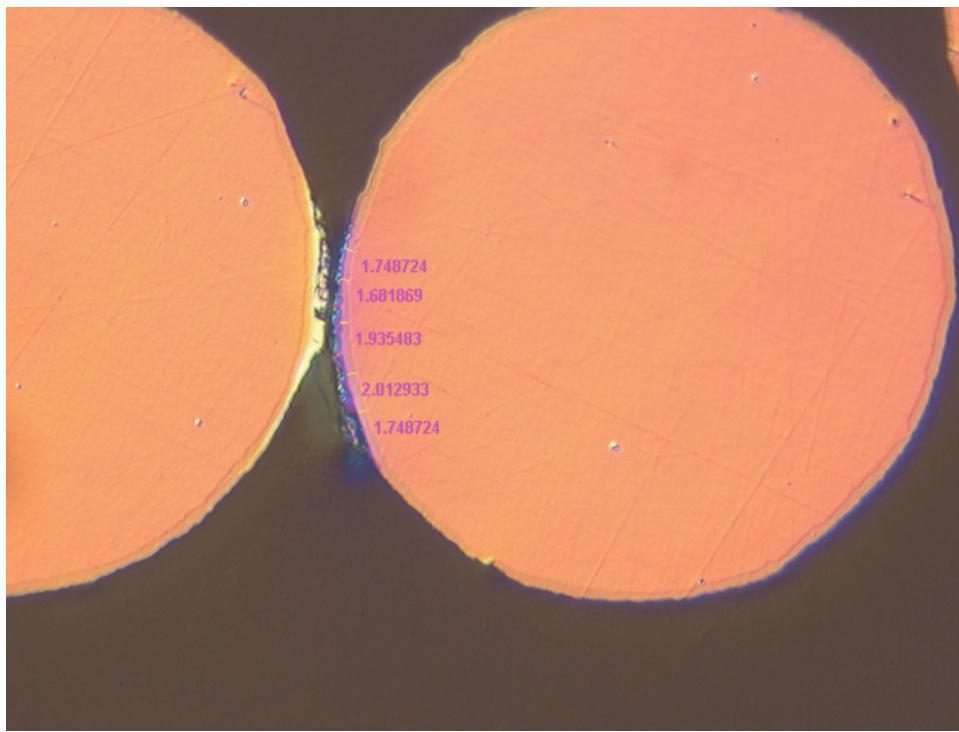


Figure 10. Polarized microscope image of contaminated wire showing minimum nickel thickness is met by calibrated measurement (numbers reported in microns).

3. CONCLUSIONS

The green material on the surface of the nickel-coated copper wire has been confirmed with EDS and FTIR to be copper abietate. Further analysis using scanning electron microscopy and high magnification optical microscopy confirm that no gross degradation or corrosion of the nickel-coated copper wire has occurred. The thickness of the nickel plating under areas of copper abietate still meets the minimum thickness requirements even after decades of exposure to the copper abietate and small amounts of abietic acid. It should be noted, however, that these particular wires have been under controlled environments. During flight, their time is spent in a sealed box that is back-filled with dry nitrogen and is exposed to ambient conditions during lid removal in a clean room. These test results and observations reflect copper abietate behaviour in those environments.

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