Determination of the feasibility of using attenuated total reflectance Fourier transform–infrared spectroscopy to evaluate thermal ageing of enamel-coated magnet wire

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A study was initiated to determine the feasibility of employing attenuated total reflectance Fourier transform–infrared spectroscopy (ATR/FT–IR) to detect changes resulting from thermal ageing in the enamel of copper magnet wire. Polyamideimide (SX-81002) was cured on a zinc selenide (ZnSe) internal reflection element (IRE) coated with a thin film of metallic copper. The coated IRE was inserted in a Circle cell housed in a heating jacket and maintained at 250 °C on the optical bench of an infrared spectrometer to simulate thermal ageing of enamel-coated magnet wire. Evaluation of the infrared spectra in the fingerprint region suggested that the polymer experienced chemical degradation within a 23 day period of thermal ageing. Through comparisons with controls containing no copper coatings, and ageing studies carried out at 28 °C, it was determined that ageing at elevated temperature caused more pronounced chemical changes in the polymer than did exposure to the copper. These results indicate that ATR/FT–IR may be a useful tool to detect enamel fatigue after a short period of thermal ageing.

1. Introduction
The failure of copper wire at elevated temperatures, has been reported to result from the formation of copper oxides which react with the polymer coating surrounding the wire to cause dielectric failure [1]. Current procedures used to determine the stability of new polymer coatings involve ageing of the copper wire for periods of up to 2 years before testing its suitability for release to the customer. The development of a rapid technique to verify the stability of the polymer coating will enable quality assurance programmes to determine the suitability of a polymer coating in a shorter time frame. Furthermore, a knowledge of copper oxidation rates and polymer stability will facilitate the evaluation of corrosion/oxidation inhibitors incorporated in the coating process.

Fourier transform–infrared (FT–IR) reflection–absorption spectroscopy (RAS) has been used effectively to investigate both the effect of thermal ageing on polymers deposited on to copper surfaces [2, 3] and the behaviour of surface oxide layers of copper upon heating [4]. RAS is generally used to study films which are less than 100 nm thick [5], therefore the technique is not able to provide information on changes within a polymer film deposited on to a copper substrate and on changes occurring simultaneously in the valence state of the copper surface.

Attenuated total reflectance (ATR) infrared spectroscopy has been used extensively to obtain information on substances at solid/solid interfaces [6]. A modification of this technique is the deposition of a thin metal film on to the surface of the internal reflection element (IRE) [7–9]. If the metallic film is thin enough, the energy transmission of the IRE remains sufficiently high to allow the detection of substances, such as polymers, at the metal interface [10–13]. A further advantage of this technique is that the penetration depth of the infrared into the sample is less than 1 μm, so only changes in the polymer that occur at the copper/polymer interface are recorded.

The aim of this research was to investigate the feasibility of using attenuated total reflectance Fourier transform–infrared spectroscopy (ATR/FT–IR) to (1) study the oxidation of metallic copper in contact with polymer coatings at elevated temperatures; (2) determine chemical changes that occur in the polymer during thermal ageing; (3) relate the observed chemical changes in the polymer coating to the copper oxidation reaction.
2. Experimental procedure

2.1. Curing of a polyamideimide polymer on a ZnSe IREs

An enamel polymer (SX-81002), commonly used in the production of enamel-coated magnet wire, was obtained from Dr. François Lavalle (Essex Group, Inc, Fort Wayne, IN). A coating and curing regime was developed so that the polymer coatings on ZnSe IREs were similar to coatings of SX-81002 on copper magnet wire. Two factors were critical during the polymer coating; the temperature regime during curing of the polymer on to the IRE, and the thickness and uniformity of the polymer.

Coating and subsequent curing of the polymer on the IRE was complicated by two factors. Firstly, the end cones of the IRE through which the infrared radiation entered and exited had to be free of coating, and secondly, the ZnSe IRE could not withstand temperatures above 250 °C, which was 50 °C lower than the normal minimum curing temperature of SX-81002.

To facilitate polymer coating of the IRE, a Teflon holder was constructed which held two IREs and prevented coating of the end cones (Fig. 1). For logistical reasons, the development of a low-temperature polymer-curing regime was carried out using glass rods instead of ZnSe IREs. The glass rods were dipped in various concentrations of polymer diluted with a 1:1 mix of N-methyl-2-pyrrolidone and xylene and subjected to a variety of curing regimes. The following coating and curing procedure produced a coating similar to the polymer on copper wire; ZnSe IREs were positioned in the Teflon holder and dipped into a container of undiluted SX-81002. The holder and IREs were then immediately placed in a muffle furnace at 200 °C for 24 h. The temperature was subsequently increased over a 10 min interval to 225 °C and maintained at 225 °C for 20 min, then further increased over a 10 min interval to 250 °C. After 20 min at 250 °C, the muffle furnace was turned off and left to cool overnight (16 h). Polymer coatings produced in this manner had a glass transition temperature of 552 °C and a thickness of between 0.01 and 0.0039 in (0.0254 and 0.0099 cm), both parameters were within the normal range for this polymer on copper wire [14].

2.2. Performance of ZnSe IREs at elevated temperatures

Two Circle cells (Spectra Tech, Stamford, CT), each containing a ZnSe IRE, were aligned in separate paths of a dual-beam Perkin-Elmer model 1800 FT-IR spectrometer (Fig. 2). One of the Circle cells was contained within a heating jacket (Spectra Tech). The FT-IR was equipped with a 2 mm diameter L-alanine-doped deuterated triglycine sulphate (DTGS) detector. Operating parameters were adjusted to achieve a maximum signal/noise ratio over the spectral range of 4000–500 cm⁻¹. The parameters depended on the energy throughput and were as follows: the interferometer OPD velocity was set at either 0.1 or 0.05 cm s⁻¹ and the Jacquinot stop used was either 3 or 6, depending on whether or not the IRE had been copper-coated.

All interferograms were double-sided and apodized with a medium Beer-Norton function prior before the fast Fourier transformation using the Model 1800 computer software. Scans were taken over the range of 4000–450 cm⁻¹ with an interval of 2 cm⁻¹ and a nominal resolution of 4.0 cm⁻¹. A single-beam spectrum was calculated from 100 averaged scans.

To determine the effect of heating on the energy throughput of a ZnSe IRE, the temperature of the Circle cell contained within the heating jacket was increased in 25 °C increments to a maximum of 250 °C. After each change in temperature, a spectrum was obtained from the heated Circle cell, as well as from an unheated Circle cell, which served as a control. The energy throughput and the signal/noise ratio at each temperature were also monitored. After the IRE had been heated to 250 °C for 20 min, it was cooled to 25 °C and an additional spectrum was obtained.