Analysis Technologies for Quality Improvement in Magnet Wires of Electrified Vehicles

Kentaro OKAMOTO*, Yugo KUBO, Yutaka HOSHINA, and Toshiyuki KOIZUMI

Magnet wire is an electrical wire coated with an insulation film. We analyze its constituent elements such as a conductor, insulation film, and the interface between them, in order to improve the quality and performance of the wire. It is increasingly important to develop novel and advanced analysis technologies and use them depending on the purposes of analysis. This paper reports on our unique technology and approach to the analysis of the aforementioned elements.

Keywords: magnet wire, conductor, insulation film, interface, analysis technology

1. Introduction

There has been growing demand for magnet wires in line with the growth of the global market for electrified vehicles. The Analysis Technology Research Center has been working to accelerate the development of new products and improve the quality of magnet wires by utilizing analysis technology to enable Sumitomo Electric Industries, Ltd. to achieve further growth of its magnet wire business and promote enhancement of its manufacturing capability.

Magnet wires refer to an electrical wire that consists of a conductor and an insulation film. High-quality and high-performance materials are required for conductors and insulation films that make up magnet wires to meet rigorous usage requirements for electrified vehicles including heat-resistance property, excellent workability, and insulation property.[(1),(2)] Magnet wires also require technologies to achieve the adhesion between dissimilar materials when a conductor is coated with an insulation film. For improvement of the quality and performance of magnet wires, the targets of analysis are conductors, insulation films, and the interface between the conductors and insulation films (dissimilar materials). It is necessary to develop analysis technologies and utilize cutting-edge analysis technologies depending on the targets (Fig. 1). This paper reports on our unique technology and approach to the analysis of conductors, insulation films, and the interface between them.

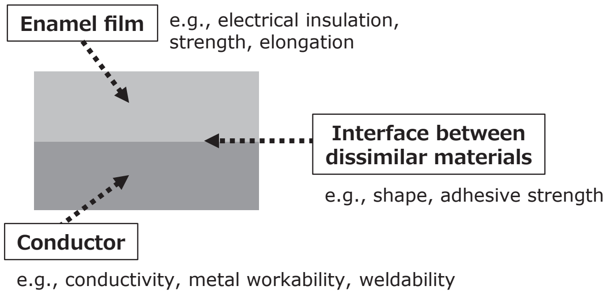

Fig. 1. Targets of analysis

2. X-ray CT*1 Observation of the Internal State

Magnet wires are electrical wires coated with an insulation film. In the material evaluation, it is often required to observe three-dimensionally the internal structure and state change in the longitudinal direction. Mechanical polishing and ion cross section milling are sample preparation techniques to observe the internal structure, but these techniques have three major difficulties shown below in meeting the aforementioned needs.

(1) The internal state cannot be checked from the outside, so it is impossible to process parts accurately.

(2) Reworking is impossible. Thus, these technologies cannot be used for scarcely available samples.

(3) Samples must be processed and observed repeatedly for three-dimensional observation, which is time consuming.

At our center, we have been promoting the use of X-ray CT to achieve nondestructive three-dimensional observation of the internal structure in order to overcome these difficulties.

Figure 2 shows the working principle of the X-ray CT measurement. The X-ray irradiated from an X-ray source penetrates a specimen. The fluoroscopic image captured by the detector is used to acquire data while gradually changing the angle of the specimen. A large amount of data acquired by rotating the specimen 360° are calculated by a computer to produce a three-dimensional image and tomogram on an arbitrary surface. These images are created based on the amount of X-ray transmission. Thus, X-ray CT is a technology for reproducing the internal structure

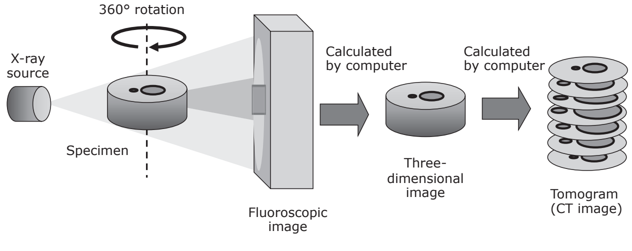

Fig. 2. Working principle of X-ray CT

while reflecting the density difference and constituent atoms of a material in a nondestructive manner.

We applied the X-ray CT-based nondestructive observation technology to the internal observation of conductors. The observation results were utilized to investigate the quality of conductors. X-ray CT-based nondestructive observation found low-density parts (voids) inside conductors. Void distribution images as shown in Fig. 3 were created based on the three-dimensional data obtained. Visualization of such distribution has made it possible to check the concentration of voids and determine the quality of conductors through quantification. When a conductor material is drawn to reduce the diameter, defects may result in manufacturing problems. Thus, it is important to accurately determine the distribution of internal defects such as voids, cracks, and segregations when achieving stable manufacturing and developing products of better conductor quality.

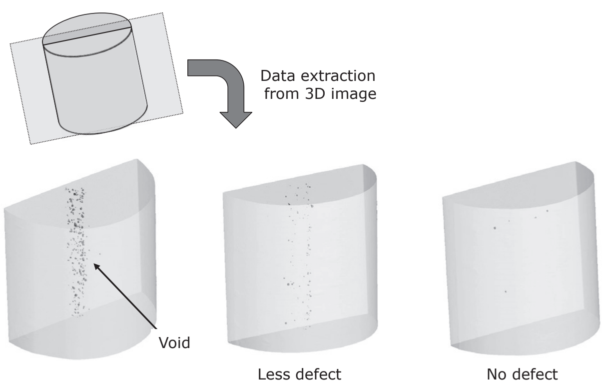

Fig. 3. Observation results of the internal state of a conductor

It is also possible to investigate internal defects more closely by processing the defective position identified in nondestructive observation. X-ray CT is also highly useful as pretreatment for destructive observation as shown in the procedure of Fig. 4.

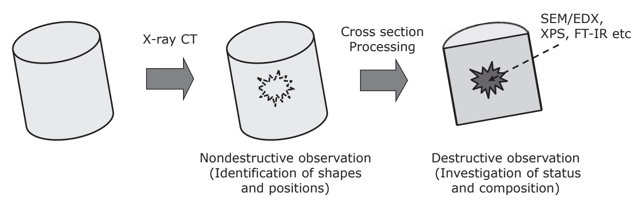

Fig. 4. Observation and analysis of internal structure by utilizing X-ray CT

3. Evaluation of the Degree of Cure Based on Evolved Gas Analysis

Polyimide (PI) is a thermosetting resin excellent in heat-resistance property, workability, and insulation property. It is used for insulation films of magnet wires for electrified vehicles.(1) The curing reaction of PI is shown in Fig. 5. The substructure of PI molecules changes after reaction. The reaction amount (degree of cure) is an important index for evaluating cured products. However, it was difficult to evaluate the degree of cure with sufficient accuracy. At our center, we focused on the water molecules generated during curing and developed a proprietary technique for evaluating the degree of cure by utilizing the evolved gas analysis mass spectrometry (EGA-MS).*2

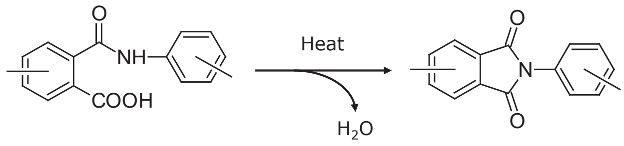

Fig. 5. Curing reaction of polyimide

Figure 6 (a) shows the behavior of water molecules generated from uncured PI by heating. We confirmed that water molecules are generated gradually from 100°C by gradient heating when unreacted substructures remain in the PI molecules.

The results of Fourier transform infrared spectroscopy (FT-IR)*3 in Fig. 6 (b) indicated peaks derived from imide bonds after gas analysis (C=O stretching vibration 1712 cm^{-1}, C-N stretching vibration 1373 cm^{-1}). It was found that the curing reaction of polyimide had progressed during the gas analysis.

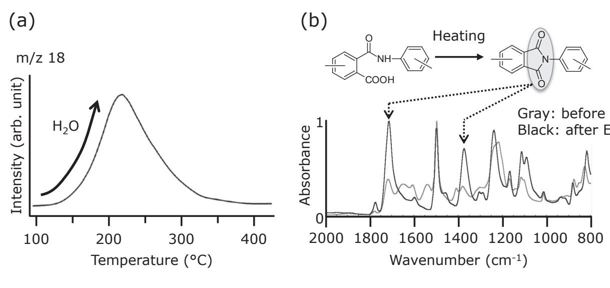

Fig. 6. (a) Temperature profile of water molecules detected by EGA-MS
(b) FT-IR spectra before and after EGA-MS

The behavior related to the generation of water molecules in EGA-MS is attributed to the curing reaction of PI. We found that, by evaluating the amount of water molecules as an index of the curing reaction, it is possible to estimate the amount of unreacted substructure contained in the PI molecules and the degree of cure which indicates the progress of the curing reaction.

Figure 7 shows the results of evaluating the degree of cure of insulation films manufactured under different conditions using the newly developed technique. It is common to use thermal analysis and FT-IR when evaluating the degree of cure of resin materials.(3),(4) However, it is difficult for these techniques to determine the differences in the degree of cure after most of the molecules are cured. Given that cured products are insoluble, the degree of cure is often evaluated based on the unreacted monomeric materials and low molecular weight compounds collected by the solvent extraction method,(5) but this cannot be applied to PI in which polymers contain unreacted substructures. To determine the slight differences in the cured material such as insulation films, the newly developed technique is highly useful because it can evaluate even trace amounts of water molecules. We have been working to utilize the technique to investigate the quality of products and seek conditions for non-defective products.

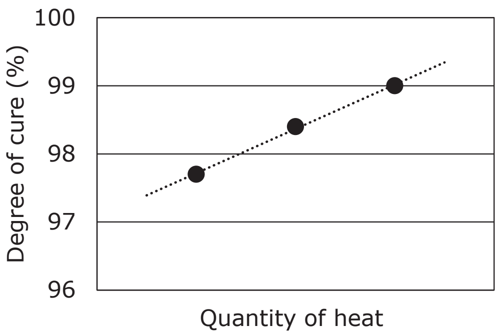

Fig. 7. Application example

4. Chemical State Analysis of the Resin/Metal Interface by Synchrotron Radiation

It is important to analyze the interface between dissimilar materials (e.g., resins and metals) to understand chemical interactions and adhesions at the interface. Since, the chemical interactions and adhesions are derived from multiple factors, combination various analytical approaches are required. We are promoting the use of hard X-ray photoelectron spectroscopy (HAXPES)*4 as one of the approaches for the interface. HAXPES is a type of X-ray photoelectron spectroscopy (XPS), owned by a synchrotron radiation facility, SPring-8, that uses the synchrotron X-ray as an excited X-ray (Figure 8). The analysis depth of HAXPES is significantly larger than that of our laboratory. For this reason, HAXPES enables us to investigate the chemical state of buried interface nondestructively. This section describes HAXPES results, for the investigation of the chemical bonding state between a resin and a metal by fabricating a specimen that simulated the interface between PI (used as an insulation film) and copper.

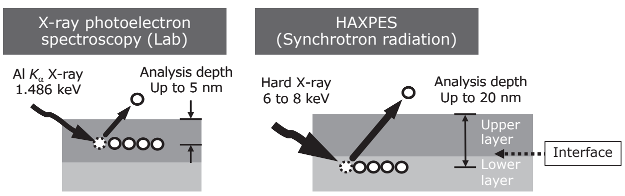

Fig. 8. Working principle of HAXPES

Figure 9 shows the procedure to fabricate the specimen for HAXPES measurement. Films of SiO_2 and copper were formed, in this order, on a GaAs substrate whose surface was flat. A PI precursor was coated on the thin copper film and cured by heating to create a PI/copper interface. Subsequently, the GaAs substrate and SiO_2 film were removed from the thin copper film to conduct the HAXPES measurement. The obtained N1s spectra are shown in Fig. 10. The N1s spectrum of the PI-only specimen is shown above that of the PI/copper interface for reference. The peak shape of the simulated specimen was significantly different from that of the PI-only specimen, which indicates that the chemical state at the PI/copper interface was different from that of the PI surface. For the PI-only specimen, a peak derived from imide bonds (400.8 eV)(6) was detected. From the HAXPES results of the simulated specimen, it is considered that the chemical state was an amic acid (i.e., opening of the imide ring) (400.0 eV)(6) at the PI/copper interface. The shoulder peak on the low-energy side (398.6 eV) supports the presence of bonds between nitrogen atoms and the metal (N-Cu).(7) The results suggest the presence of chemical bonds at the PI/copper interface.

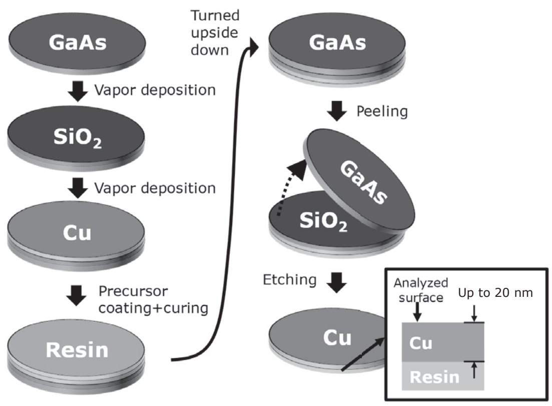

Fig. 9. Procedure to fabricate a specimen

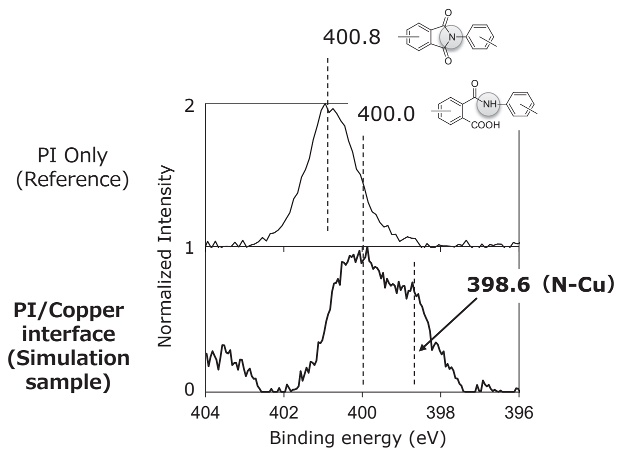

Fig. 10. HAXPES results of the PI/copper interface (N1s)

As discussed above, HAXPES is a technique that enables us to investigate the interface nondestructively while maintaining the adhesion state between dissimilar materials. Conventional analytical techniques of the interface require destruction such as peeling, cutting, or sputtering. HAXPES is a highly significant analytical technique because it enables us to analyze the real condition of the interface.

This section describes the analysis using the specimen that simulates a magnet wire. We will further promote the study to apply the HAXPES to actual products, for the elucidating the resin/metal interfacial adhesion mechanism and improving the interfacial adhesion.

5. Conclusion

We reported our efforts related to analysis technology while discussing the latest analysis examples of conductors, insulation films, and the interface composed of them. This report has focused on analysis technology utilizing analysis equipment. We also utilize CAE*5 for various purposes from material design to manufacturing process. To continue to meet various needs to overcome technical challenges, we remain committed to developing analysis technologies and improving the quality of magnet wires for electrified vehicles, and expanding the magnet wire business.

6. Acknowledgements

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Technical Terms

*1 X-ray computed tomography (X-ray CT): X-ray CT is an observation technology to capture the image of an object from various directions using X-ray, and rebuild a three-dimensional image of the object using a computer. It enables nondestructive analysis of the internal structure of an object.

*2 Evolved gas analysis mass spectrometry (EGA-MS): In an EGA-MS system, a mass spectrometer is used to measure the gas generated when a specimen placed in a heating furnace is heated gradually. The system enables investigation of the gas generation behavior in the temperature-rising process.

*3 Fourier transform infrared spectroscopy (FT-IR): An FT-IR system irradiates infrared light on specimens to measure the amount of light that penetrates or is reflected by an object. Information about the molecular structures and functional groups can be obtained from the spectra. Effective information related to the identification of substances can be obtained.

*4 Hard X-ray photoelectron spectroscopy (HAXPES): HAXPES refers to photoelectron spectroscopy that uses a hard X-ray beam (about 6 to 8 keV) as excited X-ray. The spectroscopic technology is characterized by a detection depth that is deeper than that of XPS using soft X-ray. The deep detection depth enables analysis of the chemical state of more internal or buried interfaces.

*5 Computer-aided engineering (CAE): The computer-based simulation is aimed at conducting a preliminary study of product design, manufacturing, or process design. CAE is also known as numerical analysis.

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