講演番号	300

文献番号	20225300

Observation of Aluminum-Adhesive Interface using Scanning Transmission X-ray Microscope

Qiuyi Yuan　Takanori Itoh

NISSAN ARC, LTD., Analysis PF Development Department
1, Natsushima-cho, Yokosuka, Kanagawa 237-0061, Japan (E-mail: t-ito@nissan-arc.co.jp)

KEY WORDS: **Materials, Adhesive, Test/Evaluation,** X-ray absorption, STXM [D3]

In recent years, studies on joining dissimilar materials has attracted attention as an indispensable element for reducing the weight of automobiles. Understanding the interface bonding mechanism is essential for improving bonding technology. For this purpose, a method that can observe the chemical state and morphology with submicron resolution is ideal. Scanning Transmission X-ray Microscopy (STXM) can obtain a chemical bond state with high spatial resolution (several 10 nm), has lower sample damage than electron beams, and has a field of view exceeding 10 μm. Since it can be observed, it can be said to be a powerful method for analyzing the mechanism of the bonding interface. In this study, the Al-adhesive interface is processed with a focused ion beam (FIB), the sample is about 100 nm, and STXM measurements are performed at the carbon, oxygen, and K-edges, and the Al-adhesive interface is observed. We discussed the state of carbon and oxygen in the Al-adhesive interface.

A adhesive was applied on the Al substrate, and the dried sample was FIB processed to make the sample thickness about 100 nm. The entire STXM system is installed in vacuum or in He gas. It is possible to squeeze soft X-rays to a minimum of 30 nm x 30 nm using a Fresnel zone plate, which has made it possible to produce soft X-rays that have entered through the Si_3N_4 window due to recent advances in fine wire technology. The transmitted X-ray is detected by a photodiode. The measurement is fixed with a certain energy, the sample is moved, the transmitted X-rays at each point in the area squeezed by the Fresnel zone plate are measured, and the next energy is measured after the measurement is completed at all points. The measured energy region is shown below. Measurements were performed at about 100 points in each energy region at 1 to 2 ms per point.

Two methods were used for STXM analysis. The methods are Singular Value Decomposition (SVD) analysis and clustering analysis. For SVD analysis, NEXAFS spectra are extracted from specific regions in the measurement image at three locations, the entire measurement image is linearly combined by the three NXAFS spectra, and the ratio of the three NEXAFS spectra is mapped. Clustering analysis maps differences in NEXAFS spectra by clustering similar vectors from the NEXAFS spectral shape. In SVD analysis, the intention of analysis works, but in clustering analysis, judgment is made simply by the shape of the NEXAFS spectrum. However, it is not possible to know whether the clustering analysis can be compared with the same number of clusters in the STXM image that you want to compare. Perform and consider both basic, SVD analysis, and clustering analysis.

Figure 1 shows the mapping of carbon and oxygen K-edges by SVD analysis and clustering analysis. In the SVD analysis, NEXAFS spectra were extracted from the main part of the adhesive (red), the non-uniform part of the adhesive (green), and the Al-interface part (purple), and the entire image was linearly combined and mapped. From the image of both carbon and oxygen K-edges, it can be seen that the inside of the adhesive is non-uniform. In particular, it can be seen that there is a region in the adhesive where the carbon state is different at about 200 nm from the K-edge of carbon. On the other hand, even at the K-edge of oxygen, regions with different oxygen states exist non-uniformly, but they do not match the regions with different carbon states. In addition, it is expected that the state will be different at the Al-adhesive interface at both carbon and oxygen K absorption ends. Figure 2 shows the spectra at the interface and near the interface layer. Comparing the NEXAFS spectra at the carbon and K absorption edges, it can be seen that both π * and σ * have high intensities at the interface closest to Al. At the oxygen and K absorption edges, the π * at the interface shows almost the same strength as the base material of the adhesive. These results suggest that the electronic state of carbon has changed.

This is based on results obtained from a project commissioned by the New Energy and Industrial Technology Development Organization (NEDO)【JPNP14014】.

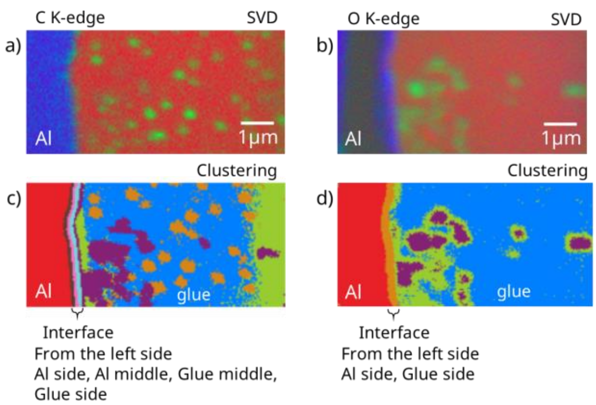

Fig.1 STXM imaging SVD analyses
a) C, K-edge, b) O, K-edge,
Clustering analysis
c) C, K-edge, d) O, K-edge.

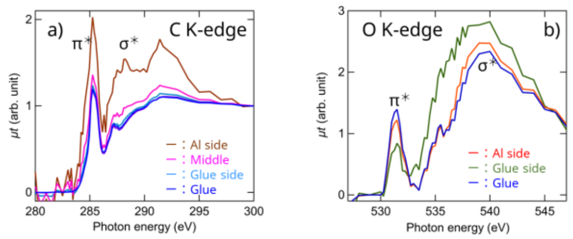

Fig.2 NEXAFS spectra
a) C, K-edge, b) O, K-edge