Material Analysis Technology R&D Efforts

H. KUWABARA

Material analysis technology has become a core technology indispensable not only for R&D and product development but also for securing product quality. This report describes the roles and organizational positioning of material analysis technology in JTEKT and provides examples of element/compound composition analysis and nano-configuration measurement in the field of surface analysis as well as examples of visualization and identification of organic structure in the field of organic analysis.

***Key Words**: material technology, material analysis, nano-scale analysis, visibility techniques*

1. Introduction

In recent years, the need for compact and lightweight products has been increased due to environmental problems such as global warming and resource depletion. These requirements raise expectations for the field of material technology. Material analysis has become an indispensable core technology not only for R&D and product development but also for guaranteeing product quality.

Nm-order structural analysis or visualization, once difficult, has been become possible, with improved performance and increasing automation of analytical instruments.

This report explains JTEKT's material analysis technology, with focus on specific examples.

2. Positioning of Material Analysis Technology and Analytical Instruments

Material analysis technology used to be regarded as a supporting technology for R&D and product development. However, in view of the current complexity of product functions, materials, and operating environment, a lot of material phenomena cannot be analyzed well without the advanced technologies of material analysis. For this reason, material analysis technology can be positioned as a core technology in the material technology field.

In the field of material analysis, it is important to understand not only methods of analysis and operating technique of analytical instruments, but also material properties; to investigate and analyze the material phenomena in all aspects. The general material analysis process is shown in **Fig. 1**.

Before using an analytical instrument, configuration, colors, and appearance of the sample material should be carefully observed by visual inspection and/or a stereomicroscope, which may provide useful hints for subsequent analysis. Sampling and pretreatment methods of a sample are also an important process. And it is important to accumulate such know-how (or form a standard manual) and pass down our skills to the next generation. Information on material analysis technology has been gathered not only internally at JTEKT but also from all other sources such as universities, material manufacturers, academic societies and our affiliated companies. Based on such information, the application of our analysis methods has always been improved to best fit the characteristics of our products.

In recent years, remarkable progress has been made in the computerization of analytical instruments incorporating a one-touch function or a black-box structure. Therefore, it is easy for every user to obtain the data, but in order to gather reliable data, it is essential to use analytical instruments with sufficient knowledge of the principles of operation and the characteristics of the instruments. Analysis results used to be shown only in a very complicated form of spectral data, but it is now

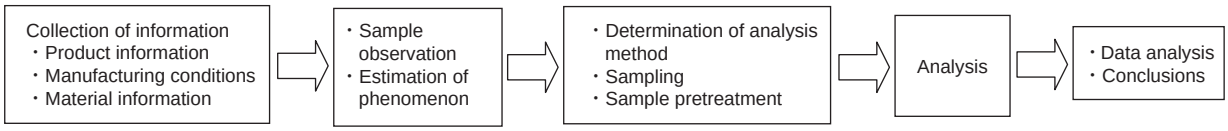

Fig. 1 Process of material analysis

Table 1 Main analytical instruments

Classification	Abbreviation	Name	Application
Element analysis	ICP	Inductively coupled plasma atomic emission spectrometer	Element in metal, Element in water
	Frameless AA	Frameless atomic absorption spectrometer	Element in metal, Element in water
	IC	Ion chromatograph	Ion volume in water
Surface analysis	SEM	Scanning electron microscope	Observation of surface shape
	EPMA	Electron probe micro-analyzer	Analysis of surface element
	FE-EPMA	Field emission electron probe micro-analyzer	Analysis of minute surface element
	XPS	X-ray photoelectron spectrometer	Analysis of surface structure
	XRD	X-ray diffractometer	Analysis of crystal structure
	FIB	Focused ion beam	Fine machining of test piece
	TEM	Transmission electron microscope	Analysis of minute structure
	LR	Laser Raman spectrometer	Analysis of inorganic structure
Organic analysis	GC-MS	Gas chromatograph mass spectrometer	Analysis of organic structure
	FT-IR	Fourier transform infrared spectrometer	Analysis of organic structure
	I-FT-IR	Imaging fourier transform infrared spectrometer	Analysis of organic structure
	GPC	Gel permeation chromatograph	Molecular weight distribution

possible to show the results in the form of two- or three-dimensional visualized data like mapping or imaging, which can be easily understood by design engineers and others who do not specialize in material analysis. **Table 1** shows a list of main analytical instruments at JTEKT.

3. Examples of Analysis

As key technologies related to materials, JTEKT covers tribology and other technologies such as metallic materials, organic materials and functional materials, and has a wide variety of analytical instruments and methods of analysis. This report shows some examples of analysis with typical analytical instruments available for use at JTEKT.

3. 1 Elemental Analysis of Melting Diffusion Area of Micro TIG Welding by EPMA (Electron probe micro analyzer)

Micro joining is being introduced for electronic components of automobiles, aimed at cost and size reduction. Quality of the joining has been guaranteed mainly through various strength tests such as static breaking strength or fatigue strength test. In addition, JTEKT has verified the joining of the micro-joining section by elemental analysis using EPMA (Electron probe micro analyzer) to guarantee the reliability. **Figure 2** shows a schematic drawing of micro TIG welding (Tungsten inert gas welding) of brass and copper, and **Fig. 3** the result of cross-sectional analysis by EPMA. As seen from the zinc diffusion behavior in the brass, fusion of the both materials is completed by heat at the time of TIG welding, which shows proof supporting sufficient joint quality in terms of not only strength but also chemical characteristics.

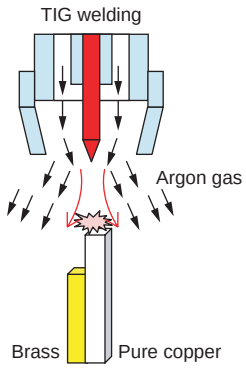

Fig. 2 Micro TIG welding of brass and copper

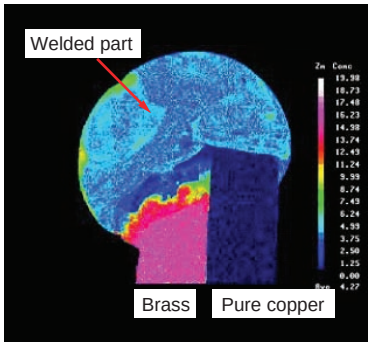

Fig. 3 EPMA result of zinc diffusion state

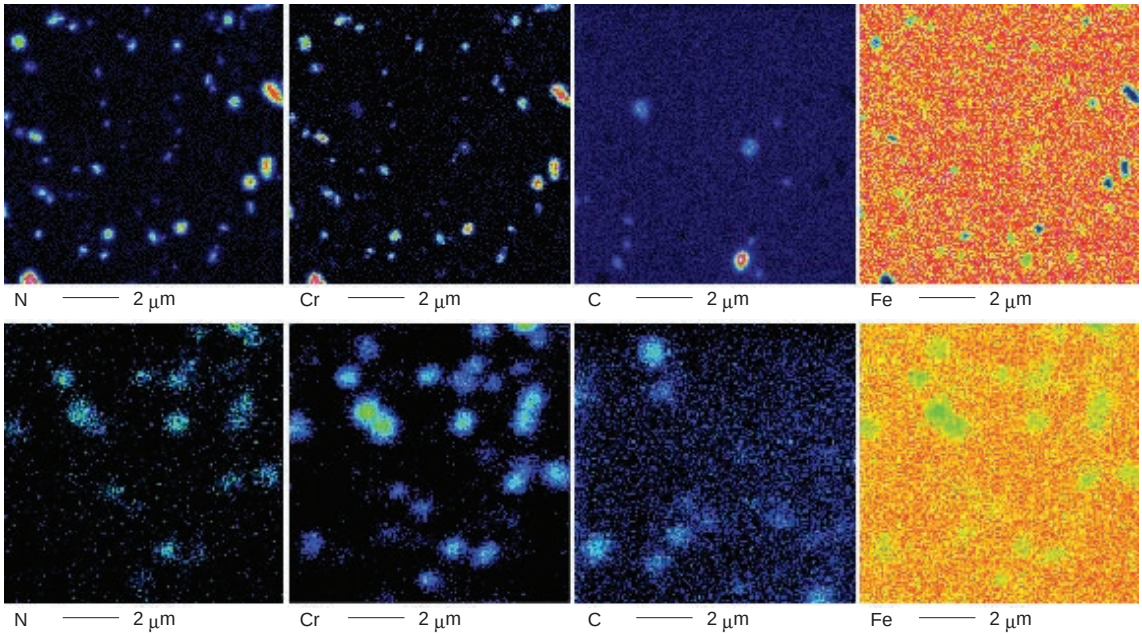

Fig. 4 Comparison of FE-EPMA and W-EPMA results
Upper: FE-EPMA Lower: W-EPMA

3. 2 Elemental Analysis by FE-EPMA (Field emission-EPMA) in nm-order Area

In the development of rolling bearing products, longer fatigue life of bearing steel is critical. Various approaches, such as the observation of microstructure, have been developed for analyzing the mechanism of long life. As an approach, precipitates have been analyzed and investigated by EPMA using W (tungsten) filament. But as EPMA has difficulty in detecting the distribution of micro precipitates in the nm-order area, FE-EPMA (Field emission electron probe micro analyzer) using a field emission gun has been introduced. Examples of the analysis are shown in **Fig. 4**, where the mapping images of precipitates of carbonitrided bearing steel are given. FE-EPMA allows it to clearly detect the distribution of micro CrN precipitates that are impossible for EPMA to observe.

3. 3 Configuration Measurement in nm-order Area by SPM (Scanning probe microscope)

With the recent growing needs for compactness of products, evaluation technology of configuration and physical properties in the nm-order area such as surface configuration, modulus of elasticity, frictional force and electrical characteristics is required. At JTEKT, SPM (Scanning probe microscope) is used to obtain necessary information and data on configuration and physical properties in the nm-order area. **Figure 5** shows the AFM (Atomic force mode microscope) image (uneven image) of the metal surface processed with a JTEKT nano-processor. It is now possible to measure both pitches and surface roughness by nm order. JTEKT has promoted the development of processing technology and elemental technologies regarding processing machines, reflecting the measured results by SPM on processing conditions.

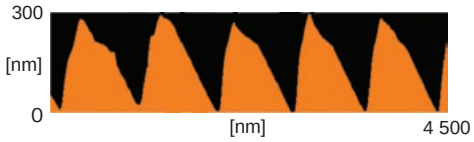

Fig. 5 AFM image of surface processed with nano-processor

3. 4 Structure Analysis of Organic Compounds by Imaging IR (Infrared spectroscopy)

Though FT-IR (Fourier transform infrared spectrometer) is the most basic analytical instrument for organic analysis, visualization of organic structure distribution has been much behind the visualization of inorganic and metallic element distributions. In recent years, as linear array detectors have become increasingly sophisticated, the imaging speed of FT-IR has been remarkably improved, and imaging IR is now in widespread use. At JTEKT, imaging IR is used to visualize the data of organic structure distribution so that a user who requires such data can easily understand

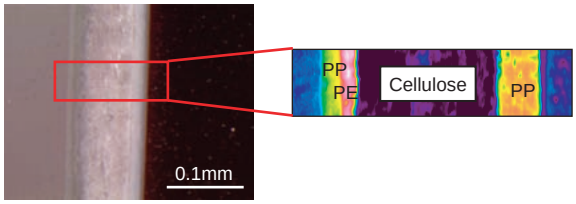

Fig. 6 IR imaging of multilayer film

it without detailed knowledge of the analysis work. With composite sample analysis, infrared data used to be collected carefully one by one. With the introduction of the imaging IR, data can be automatically collected in a wider range and then necessary IR data in the targeted area can be selectively analyzed. This helps analysis efficiency to be improved. Analysis result of multilayer structure of a resin film is shown in **Fig. 6**, where the multilayer structure is visualized as being composed of single-layer films (10 μm or so) of PP (polypropylene) and PE (polyethylene).

3. 5 Analysis of Corroded Part in Micro Zone by Laser Raman spectrometer

There are a wide variety of cases where steering gears, rolling bearings and automotive driveline products suffer from corrosion. Even if corrosion has no influence on the function of the products, customers often reject such products for cosmetic reasons. In the analysis of corroded parts, samples are getting so small that it has often become difficult to detect the corrosion by conventional analysis methods such as X-ray analysis or infrared spectroscopy. At JTEKT, corroded part in the micro zone analyzed by Laser Raman spectrometer with the use of lasers as a source of light. **Figure 8** shows comparative data for the Laser Raman analysis result for discoloration (**Fig. 7**) on the surface of rolling bearings and for the standard chart of the corrosion. The result confirmed the discoloration part as α-Fe_2O_3, while the mapping result showed the distribution of α-Fe_2O_3 in that zone (**Fig. 9**). Laser Raman spectroscopy has an analysis feature for areas as small as μm and requires little pretreatment of test pieces, which makes it possible to easily identify the compound state in the atmosphere.

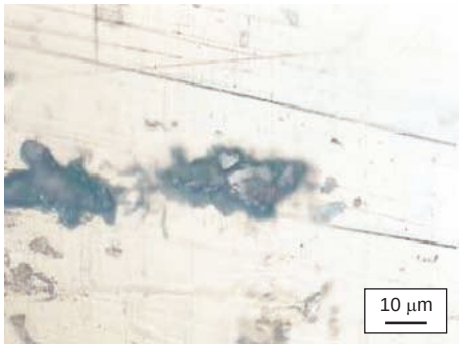

Fig. 7 Bearing discoloration part

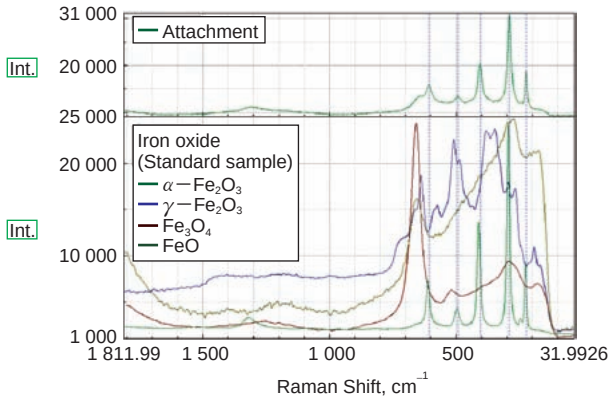

Fig. 8 Result of discoloration part analysis using Laser Raman spectrometer

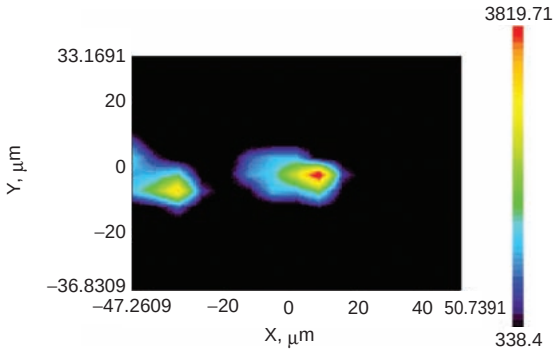

Fig. 9 Result of corroded part mapping using Laser Raman spectrometer

3. 6 Analysis of Resin Deterioration by GPC (Gel permeation chromatography)

Various resin materials are used for components of rolling bearings such as retainers. The surface of resin components is usually in contact with a lubricant (grease or oil), and therefore, there is always a risk of deterioration due to the lubricant on the resin surface. As a way of seeing how much a resin has deteriorated, it is common to check how much the strength or elongation of the resin after its immersion in a lubricant has decreased. In addition, molecular weight distribution in the depth direction is measured and used as a data for analyzing deterioration mechanism. As an example, for obtaining information in the depth direction of a nylon sample immersed in a lubricant, the sample was sliced by a microtome and turned into a derivative by trifluoroacetic acid anhydride. And then, molecular weight distribution was measured by GPC. The measurement method is shown in **Fig. 10** and the results in **Fig. 11**. Significant decrease of molecular weight was observed in the zone close to the surface of nylon which was in contact with the lubricant. **Figure 12** shows the measured results of molecular weight distribution and elongation retention in the surface zone (0~50 μm) with the nylon samples immersed in the lubricant for different hours. There was a

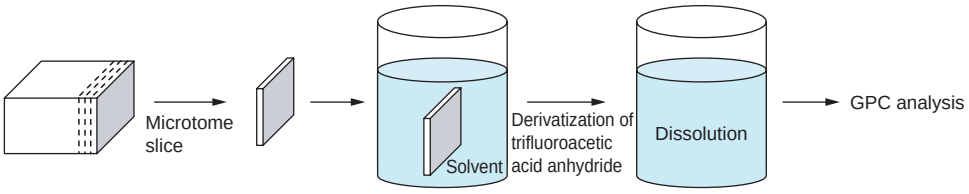

Fig. 10 Method of measuring nylon molecular weight in depth direction

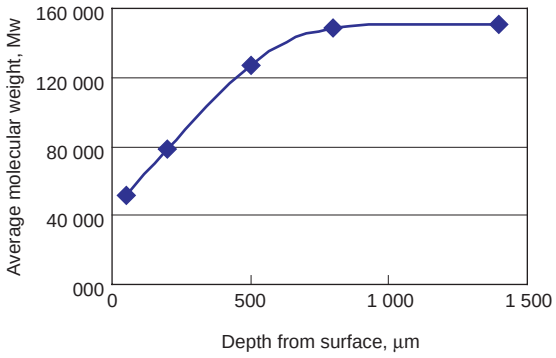

Fig. 11 Results of nylon molecular weight analysis in depth-direction

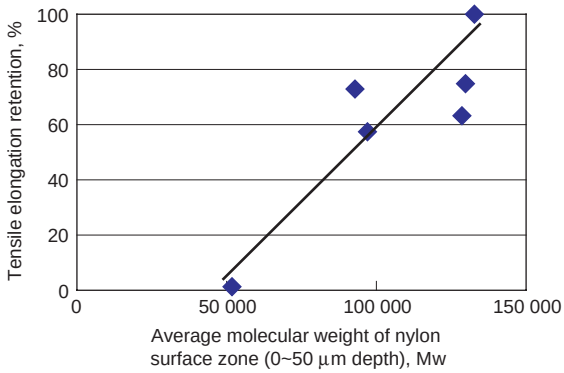

Fig. 12 Relation between tensile elongation and average molecular weight

correlation between the decrease of molecular weight and the decrease of tensile elongation. This is a useful basic data for development of resin materials.

3. 7 Analysis of Metallic Soap by Derivative Pyrolysis GC-MS (Gas chromatograph mass spectrometer)

A new high efficiency cutting fluid has been developed for high-speed machining applications. A unique problem with cutting fluids is when metal ions dissolved from machine tool components reacts with the cutting fluid to form soaps or sludge which can lead to damage of the individual machine tool components. We recognized the importance of this issue early on and studied the reaction of metal ions with fatty acids contained in the cutting fluid using both material analysis and reproduction tests. In the process of analysis of the metallic soap, TMAH reagent (Tetramethylammonium hydroxide) is reacted with the fatty acid component of the metallic soap in a pyrolysis furnace to form a derivative (methylesterified) as shown in the derivative reaction equation in **Fig. 13**. And then, the type of fatty acid is identified by the use of GC-MS (Gas chromatograph mass spectrometer), so that the cause of the trouble is determined. The test method for metallic soap reproduction has been established as shown in **Fig. 14** and the same reaction as caused on machine tools was reproduced in the laboratory. Through the studies and tests made at our laboratory, it has been revealed that it is difficult to design an affordable machine tool that will work with any type of cutting fluid. As a result, it is best to let customers select a cutting fluid that is capable of satisfying primary performance (machining) requirements as well as secondary performance requirements, such as foaming, clearance penetration, seal/rubber deterioration, corrosion and metallic soap formation. Optimal cutting fluids considering not only primary performance but also secondary performance are shown in our recommendation list of cutting fluids.

$$R_1COOM + TMAH \Rightarrow R_1COOCH_3$$

TMAH: $(CH_3)_4NOH$

M: Metal

R_1: CH_3- $(CH_2)_n$-

Fig. 13 Derivative reaction

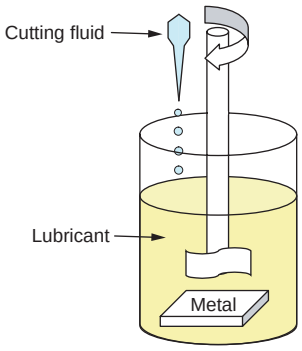

Fig. 14 Metallic soap reproduction test method

4. Conclusion

Several examples of JTEKT's R&D efforts regarding material analysis technology are presented. For the future of analysis technology, instruments and technology that satisfy super-micro and ultra-high sensitive area will be needed. At the same time, it becomes important to develop technology that makes it possible to in situ observe and catch a phenomenon dynamically and simulate it. At JTEKT, further efforts will be made for improving technology to contribute to R&D and product development.

Reference

1) JEOL: New Technical Information (2004).

H. KUWABARA*

* *Material Engineering R&D Dept., Research & Development Center*