FEATURE ARTICLE

The Horiba Application Center Provides Solutions: The X-Ray Analytical Microscope

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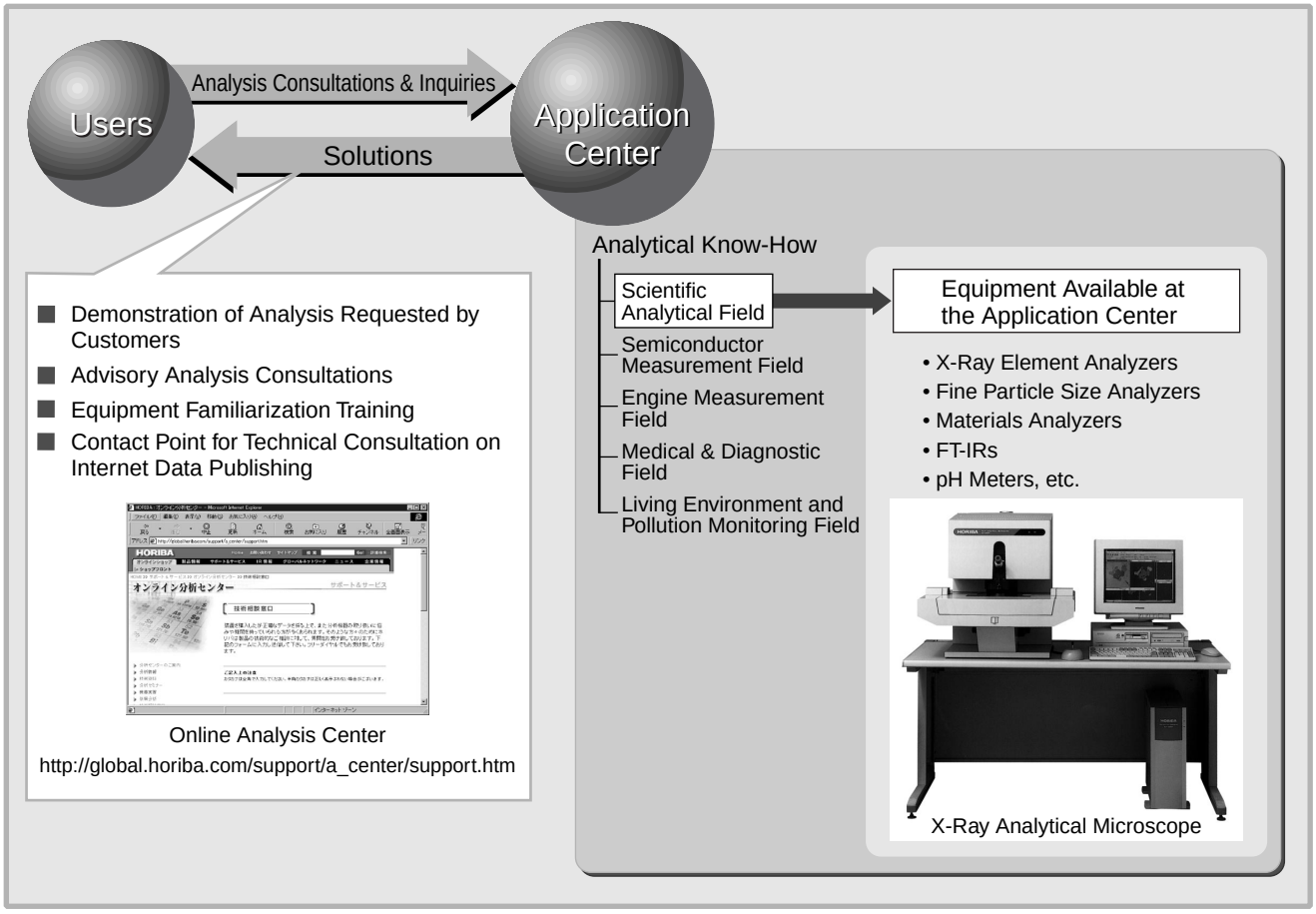

Abstract

Recently, the request from a customer to the analytical instruments maker is not only to supply the good products but also to offer the know-how for analysis. An inexperienced engineer will be able to get the precise and reliable data using these know-how. The Horiba Application Center has been endeavor to accumulate the analytical know-how for various samples using Horiba's products. In this paper we will report roles of the Application Center through the customer service of the XGT-2700W X-ray analytical microscope that is very useful for elemental analysis.

1 Application Center

The Horiba Application Center has undergone consecutive expansions since it was established at the head office of the Horiba Group in Kyoto in 1982. We presently provide application services at two locations, including the Technical Plaza in Tokyo. The equipment in the Center encompasses a wide range of the Horiba group's analytical instruments, with a particular emphasis on scientific equipment. The Center is staffed by experts in X-ray analysis, infrared spectroscopic analysis, materials analysis, and fine particle measurement who engage in day-to-day customer services and development work in analysis technology. The photographs in Fig.1 show scenes of activity at the Analysis Center at our head office and the Technical Plaza in Tokyo.

a) Kyoto Analysis Center

b) Tokyo Technical Plaza

Fig.1 Application Center

Horiba does more than simply manufacture and market products. We endeavor to give our customers true satisfaction through assistance with everything from selection of the most appropriate equipment models, proper measurement methods, and data analysis, to the resolution of new problems as they arise. To that end, we carry on activities in the following areas:

(1) Pre-Marketing Services

The Center provides a full package of pre-marketing services. When customers are considering the purchase of a Horiba product, for example, we encourage them to evaluate it by conducting a sample analysis. We also encourage them to visit the Center and operate the equipment for themselves to check its performance and usability.

(2) After-Sales Service

Center staff members are available to consult with customers who obtain questionable results in the course of their regular analysis operations. We work with our customers to resolve new problems and issues that may arise. The Center also welcomes suggestions for improvements in product functions for incorporation in succeeding models.

(3) Online Application Service

Accurate analysis depends on the proper handling of equipment and the correct interpretation of the results obtained. Horiba has established the Online Analysis Center (http://global.horiba.com/support/a_center/apl0.htm) to serve as a contact point for immediate assistance to any of our customers at any time.
The Online Analysis Center provides comprehensive application services over the Internet, including (1) information on the newest analysis technology, (2) consulting and seminars on analysis, (3) accepting customer orders to perform analysis on a commercial basis, and so on.

(4) Showroom

The Application Center also has a full range of Horiba products on display, ranging from the pH meters that were our founding product, to the latest U-20 series multifunction water-quality monitoring systems, as well as their component parts. The Center thus serves as a showroom that gives a deeper understanding of Horiba products and technologies. Each analysis display section is furnished with related technical data that are made available to Center visitors.

Horiba conducts activities like those described above at the Application Center in order to provide added value to our customers. The next section will take a closer look at the Center's activities using the example of the X-ray analytical microscope, which is utilized in fields ranging from research and development to quality control.

2 The X-Ray Analytical Microscope (XGT-2700)

The XGT-2700 is an X-ray fluorescence element analyzer that irradiates a sample with a micro-focused X-ray beam to measure the element distribution in a small area. Fig.2 gives an exterior view of the XGT-2700W, and Fig.3 shows the measurement principles involved.

Fig.2 X-Ray Analytical Microscope XGT-2700W

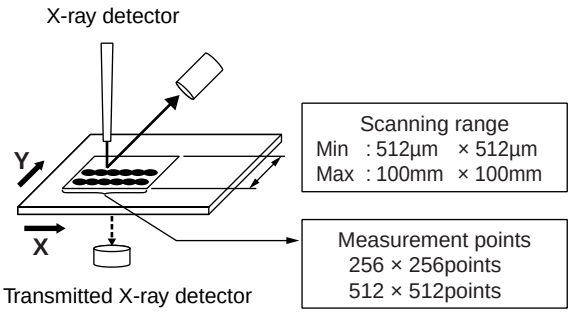

Fig.3 Measurement Principles of the X-Ray Analytical Microscope

The most distinctive feature of this device is that the X-ray emitted from an X-ray generator forms a high intensity microbeam with 10 to 100 µm diameter. The X-ray is led to the X-ray Guide Tube (XGT), which Horiba developed based on the research at the Frontier Research Center Institute for Metals of the former Science and Technology Agency, then creates a microbeam by being fully reflected inside of the tube. This microbeam is directed onto the sample, where it generates fluorescent X-rays that are detected by a silicon semiconductor detector, and the resulting spectra are used for element analysis. By moving the sample stage in the X and Y axial directions, element distribution can be measured over an area ranging from a minimum 512 µm × 512 µm to a maximum 100 mm × 100 mm, and can be displayed two-dimensionally.

This device is further capable of using an NaI scintillator to detect X-rays that are transmitted through the sample. This transmitted data makes it possible to extrapolate the internal structure of the sample.

The XGT-2700W enjoys the above superior functions in addition to the unique XRF capability for performing non-destructive analysis out of vacuum. Users are, therefore, applying it in a variety of fields, ranging from work with semiconductors, ceramics, and other inorganic materials, to the biological study of plant and animal samples.

3 Examples of Application

Having the proper hardware in perfect operating order is of course important for obtaining correct analysis results, but it is also essential to apply pretreatment that is suited to the sample properties, to set up the optimum measurement conditions, and to analyze the measurement results correctly. Here we will introduce actual cases in which the Center used of XGT-2700W analysis to provide valuable service with failure analysis in circuit boards that use lead-free solder, and with research into ulcer treatment using rats.

3.1 Failure Analysis in Electronic Components

There is a move toward the use of solder that contains no lead (lead-free solder) in electronic components, for occupational health and other reasons. In this connection, we received an inquiry from Professor Katsuaki Suganuma of Osaka University, who wondered if our XGT-2700W could be used to check the condition of connections on circuit boards that use lead-free solder (containing Sn, Bi, Ag, and Ge). We performed a trial analysis at the Center. Fig.4 shows the transmission images, Fig.5 shows the fluorescent X-ray images, and Fig.6 presents a schematic cross-sectional view of a circuit board. The transmission images showed the presence of voids in the board connections, and the results of area analysis by X-ray fluorescence confirmed that copper (Cu) migration was taking place. The cause of this migration appears to have been the influence of residual flux and detergent where some copper was exposed because the copper pads on the board were not completely covered with solder[1].

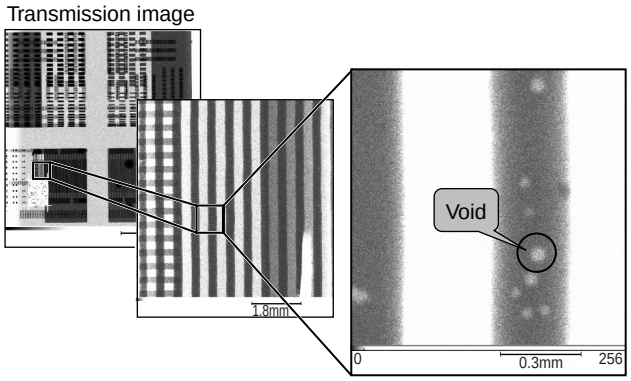

Fig.4 X-ray Transmission Images of the Connecting Area on a Print Board Using Lead-Free Solder

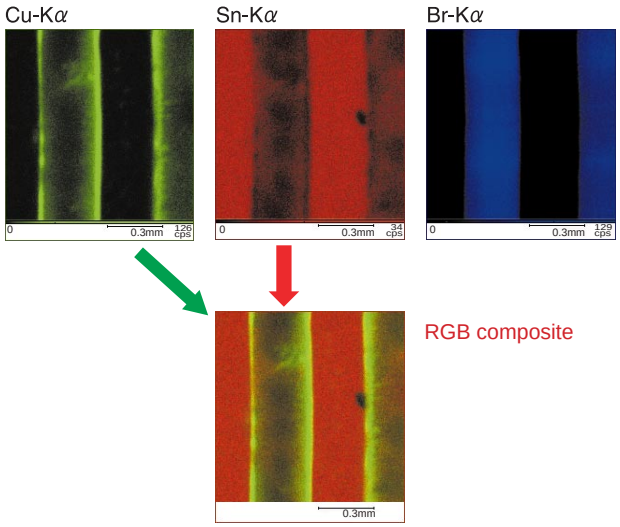

Fig.5 X-ray Fluorescent Images of the Connecting Area on a Print Board Using Lead-Free Solder

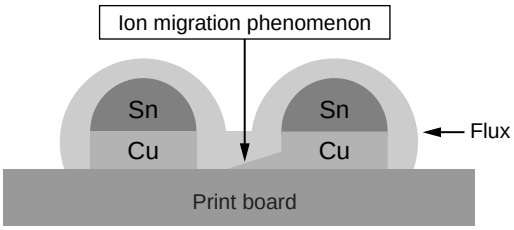

Fig.6 Schematic View of a Print Board Cross-Section

Circuit boards are generally coated with organic materials, and up to now a non-destructive means of seeking out elemental data under this organic material, where the cause of such failure is located, was not thought to be within easy reach. With the XGT-2700W, however, we combined the use of images from X-ray transmission and X-ray fluorescent analysis to obtain data that are useful in conducting failure analysis of electronic materials. In the future, we will take positive action to communicate these findings to customers who experience similar problems, for their reference in exploring new and expanded applications for this equipment.

3.2 Research into Ulcer Treatment

Dr. Takeshi Otsuka of Kyoto Prefectural University of Medicine is using rats to conduct research in the effectiveness of a preparation containing zinc for stomach ulcer treatment. Male Wistar rats were injected with acetic acid to induce ulcers, to which the anti-ulcer drug (a preparation containing zinc) was applied. Abdominal incisions were then made and the kinetics of the zinc along the border of the ulcers were observed with the XGT-2700. Comparison with untreated rats confirmed the therapy effect of the treatment.

Fig.7 shows the results from area analysis of treated rats, and Fig.8 the results from untreated rats. These results confirm that zinc was accumulating along the border of the ulcers in the treated rats, producing a healing effect on the ulcers. At the same time, the zinc concentration along the border of ulcers in the untreated rats was also found to be increasing. This report has received notice as supporting evidence for the natural healing action of zinc, which is one of the essential trace elements found in living organisms[2].

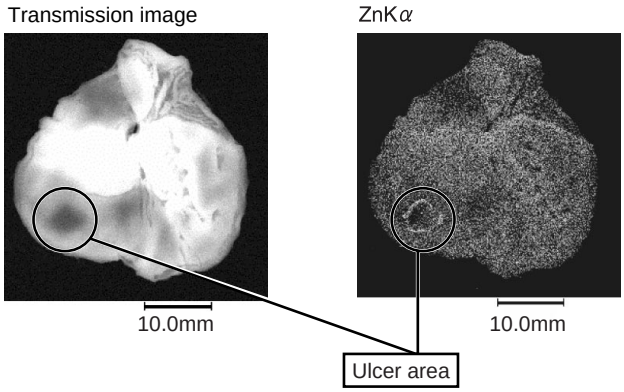

Fig.7 Kinetics of Zinc on the Border of Ulcers in Treated Rats

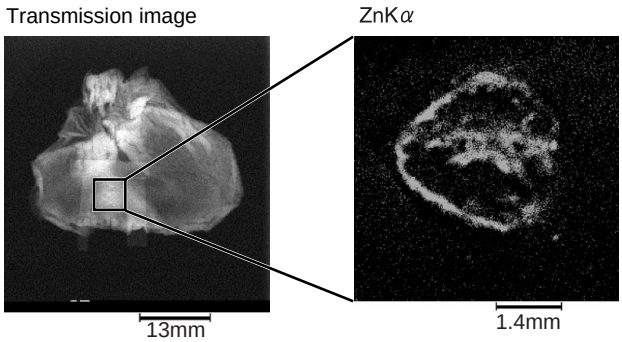

Fig.8 Kinetics of Zinc on the Border of Ulcers in Untreated Rats

In the past, element analysis of biological samples required such pretreatment as drying and dehydration, embedding in resin, preparation of sections, treatment for conductivity, and so on. This made it difficult to check element distribution over a wide area in fresh samples.

Preparation of samples used for this analysis involved making an abdominal incision on the rat, removing the stomach, cutting a section from the middle portion, fixing the section in formalin, enclosing it in an organic film, and applying heat to the ends to seal it. The Application Center examined various pretreatment methods to find those suited for use with the XGT-2700W, which is capable of performing a wide visual range analysis in the air, and so made it possible to observe biological samples in a near-fresh condition.

4 Future Directions for the Application Center

We have described the above cases of actual measurements performed using the X-ray analytical microscope in order to illustrate the kind of role played by the Application Center. As these examples show, the correct analysis of unknown samples that are becoming ever more sophisticated and complex will require that customers and the Center explore solutions together. For the future, those of us who are responsible for the Center are committed to acquiring the most advanced analysis technology, and to further enhancing the Center's resources as an analysis facility for the entire Horiba Group, including its affiliates. We are endeavoring in this way to deepen still further our customers' confidence in us.

In closing, we would like to express our profound gratitude to Professor Katsuaki Suganuma of Osaka University and Dr. Takeshi Otsuka of Kyoto Prefectural University of Medicine, who generously furnished their samples and allowed us to publish them here.

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