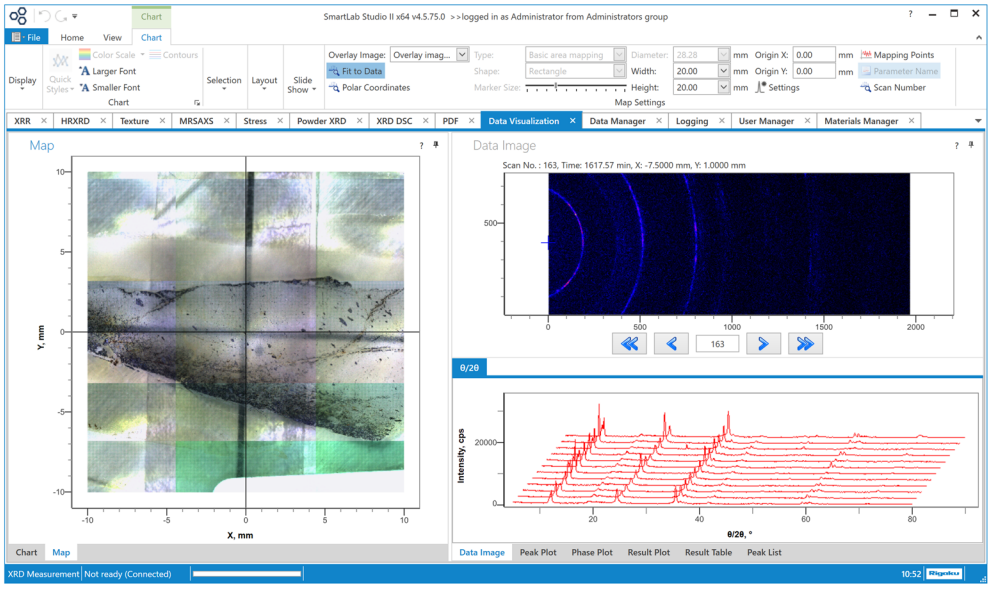
SmartLab Studio II

Data Visualization plugin

—Analysis examples of micro area XY mapping using CBO-μ—



1. Introduction

XY mapping measurements to obtain physical values for your analysis purpose is one of the best ways to analyze the state distribution of a substance, the strength distribution of a material or a device, etc. The significant progress in X-ray sources, optical devices and detectors has enabled the irradiated area to be narrowed and low X-ray intensities to be detected. It is becoming possible to evaluate smaller (or smaller areas of) materials without high-cost optical devices or attachments. This article describes the following experiments and analysis:

- The sample was moved in the X and Y directions step by step using an XY attachment;
- The X-ray beam was conditioned by an optical device called CBO-μ and irradiated onto a small area;
- The X-rays diffracted from the sample were collected using the HyPix-3000 2D hybrid pixel array detector;
- The collected data sets were analyzed using the Data Visualization plugin of the integrated X-ray analysis software SmartLab Studio II.

2. Features of the Data Visualization plugin

The Data Visualization plugin analyzes several data sets together and visualizes the data sets and analysis results in an easily comprehendible manner. Using the SmartLab system, X-ray measurements with changing temperatures or humidity variations, as well as XY mapping measurements, can be made by adding various external equipment. A single scan or exposure sometimes takes less than one minute and the number of data sets collected has increased considerably; hundreds or thousands of data sets are often obtained for just one analysis purpose. In the past, conventional analysis software, together with SmartLab Studio, used a batch process based on a preset analysis template; however, it took considerable time. Also, changes in physical parameters versus time or temperature could not be visualized graphically. An XY mapping display program called “Data Mapper” was able to visualize analysis results obtained from XY mapping measurements, but not all physical parameters were supported; only layer structure parameter values from GlobalFit and peak search results from PDXL were available.

The Data Visualization plugin in SmartLab Studio II provides various powerful features such as plotting physical parameters versus time or temperature, displaying XY mapping results, etc. This article describes the rich functions of the XY mapping display based on a couple of analysis examples.

3. Incident Optics and XY Mapping Measurements

The SmartLab system provides several optical devices to create a small X-ray beam. In this article, the CBO-μ unit is introduced. The CBO-μ unit conditions the X-ray beam so that it irradiates an area of around 100 μm in FWHM on a sample surface. The CBO-μ unit is installed on the incident arm (θs axis) at the position of the incident parallel slit adapter; that is, just after the CBO unit that generates the parallel X-ray beam.

Fig. 1. CBO-μ; an optical device for micro area measurements.

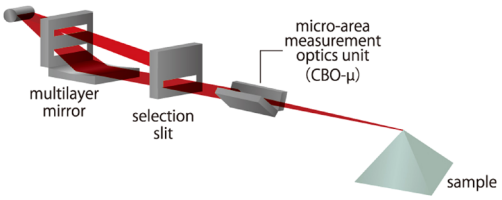


Fig. 2. Incident optics for micro area measurements using CBO-μ.

In the following XY mapping measurements, (1) the XY-20 mm attachment (where the X and Y axes can move in the range of -10 mm $\sim$ $+10$ mm) was used, (2) the sample was moved in the X and Y directions step by step (the step size was 1 mm), (3) 2D scan was performed using HyPix-3000 at each measurement point. The scan conditions and mapping conditions (ranges and the number of points) are described below.

4. Analysis Examples

4.1. Rusty iron plate

This article describes the features of the Data Visualization plugin with a couple of analysis examples. The first target sample was a rusty iron plate, where the distribution of the substances on the sample surface was analyzed. A photo of the iron plate is displayed on the left part of Fig. 3. In this way, the Data Visualization plugin can load an image that was taken by the sample attachment camera and display it on the analysis window. The brown areas are considered to mainly consist of iron oxides, and the gray areas are considered to mainly consist of iron. Measurements were made on a total of 225 points on the surface of the iron plate (the X and Y ranges were both from -7 mm to $+7$ mm, and the step size was 1 mm). The $\theta/2\theta$ scans in 2D mode were performed from 20° to 60° with a scan speed of 5°/min. One of the 2D data sets collected using a HyPix-3000 detector and the 2θ profiles converted from the 2D data sets are displayed on the right part of Fig. 3. Fluorescent X-rays from the iron contained in the sample were reduced using XRF reduction mode, and good quality data with low background were obtained.

The Data Visualization plugin displays a photo of the sample and overlays the measurement points in the Map panel on the left, and displays a 2D image and 1D profile(s) in the Data Image panel on the right. 1D profile data sets are usually loaded and displayed, but there is another option allowing you to convert loaded

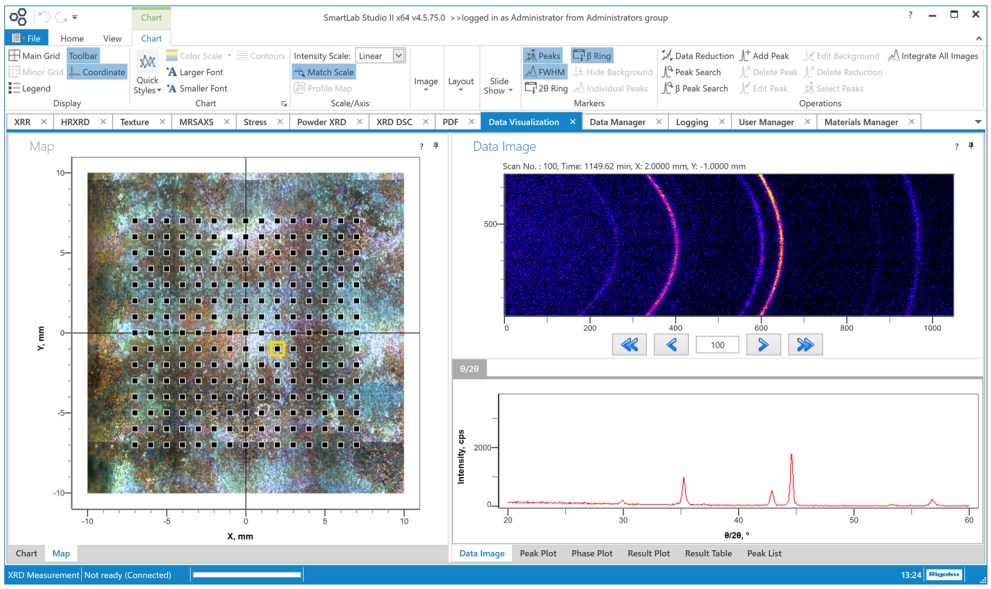


Fig. 3. Data Visualization plugin: Map panel (left), Data Image panel (right).

2D data to 1D profiles under the conversion conditions you have set. Figure 3 shows that measurement point No.100 is selected (highlighted by the yellow rectangle) in the Map panel on the left, and that the corresponding 2D and 1D data collected at the measurement point are displayed on the right side. You can select the measurement points one by one or all points inside a specified area or along a specified line. When multiple points are selected, the corresponding 1D profiles are displayed in the bottom-right graph.

Next, we describe how to analyze data and how to display results. The simplest case is to do peak search for 1D profiles and compare the distributions of peak intensities and widths with the sample surface state. Click the **Peak Search** button on the ribbon, set the peak search conditions in the displayed dialog box, then click the **Run** button to do peak search. In this example (Fig. 4), peak search was done after setting the peak search range since we wanted to analyze the peak at around 44.7°, which is derived from iron.

Figure 5 shows the Data Visualization plugin tab after peak search. Based on the conditions shown in Fig. 4, the peak search was performed in the specified range (around 44.7°) of each profile. On the left side of the plugin tab, distribution maps of the peak search results—such as peak positions, peak heights, FWHMs, integrated intensities, etc.—can be overlaid on a sample photo. A dotted color map is sometimes preferable because the normal topo color map hides the sample photo. The size of the dots can be changed.

The Data Visualization plugin also analyzes a series of data sets all together using the functions of other analysis plugins*[1]. In this example, Fe and Fe_3O_4 were found as the main crystalline phases contained in the

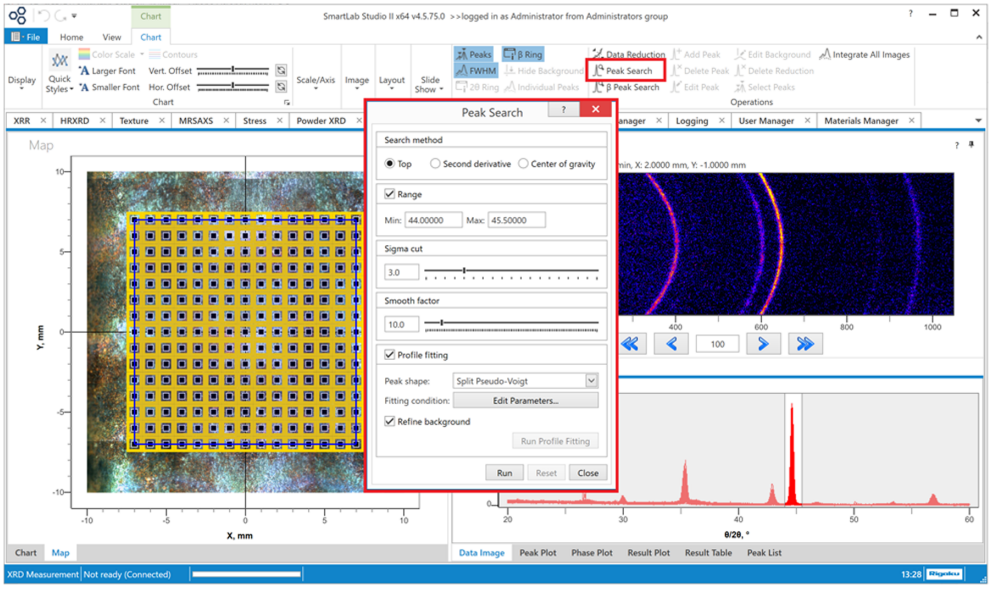


Fig. 4. Selecting measurement points (left) and setting peak search conditions (center).

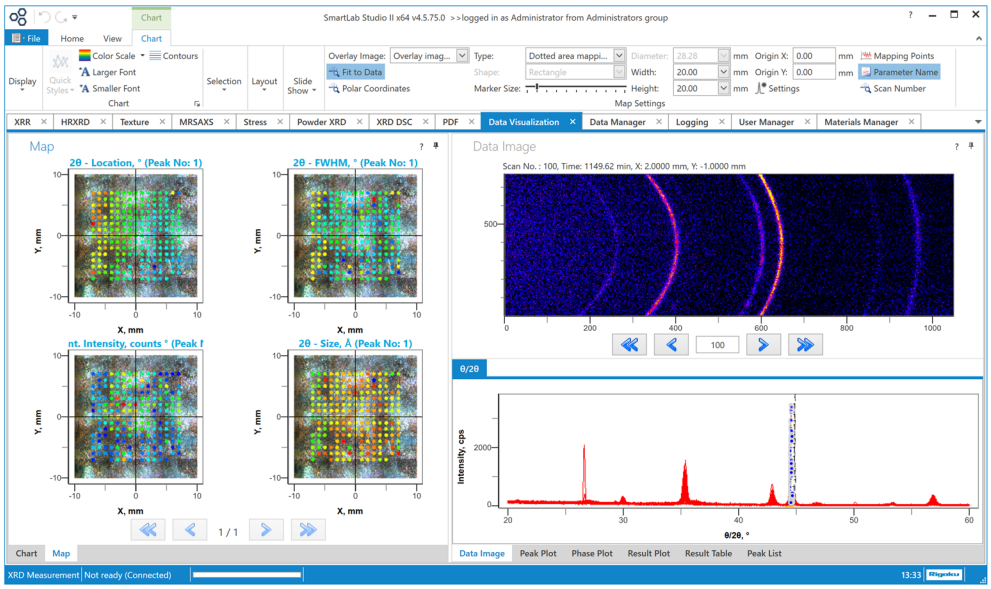


Fig. 5. Overlaying peak search results on the image of the iron plate (dotted color map).

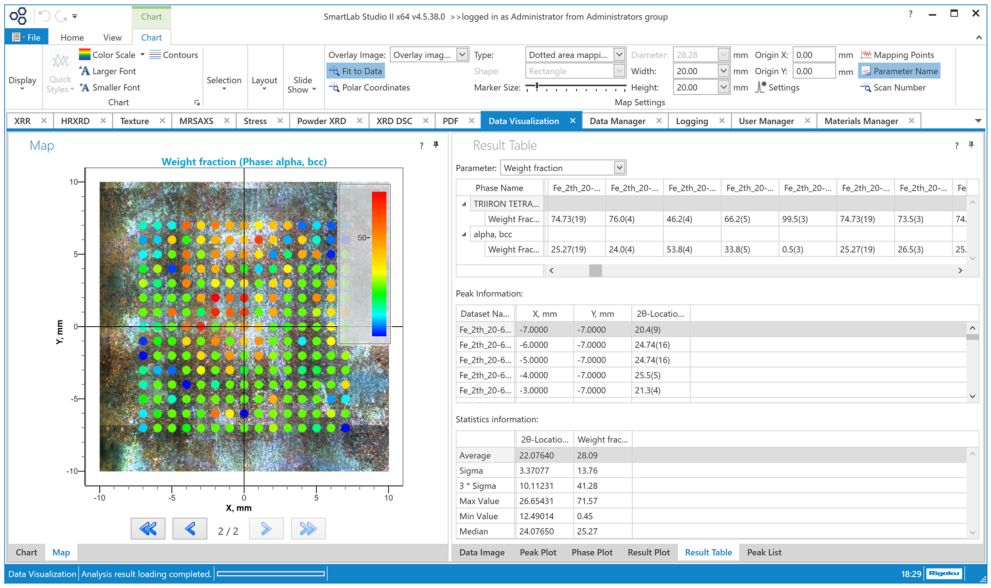


Fig. 6. Overlaying the distribution map of weight fractions of iron on the photo of the iron plate (dotted color map).

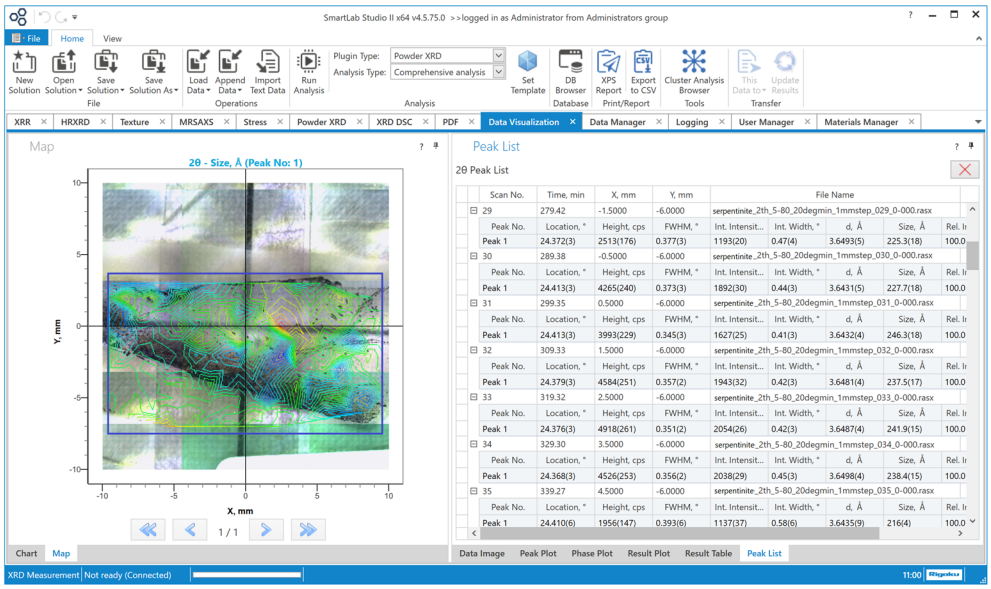


Fig. 7. Crystallite size distribution map overlaid on the photo of the serpentinite flake.

sample (iron plate) as the result of phase identification. Therefore, each peak was assigned to Fe or Fe_3O_4 then weight fractions of Fe and Fe_3O_4 were determined. The procedure is (1) Create an analysis template with Fe and Fe_3O_4 as the crystalline phases and save the template in the Powder XRD plugin, (2) Select the saved template in the Data Visualization plugin, (3) Select **Powder XRD** from the **Plugin Type** box, and select **RIR quantitative analysis** from the **Analysis Type** box. (4) Click the **Run Analysis** button on the ribbon. The RIR quantitative analysis will be performed for all the data sets selected in the **Map** panel on the left side, and the quantity values (weight fractions) of Fe and Fe_3O_4 will be displayed on the **Result Table** tab. The distribution map of the weight fractions can also be overlaid onto the sample photo on the left-side Map panel. You can compare the weight fractions with, for example, the colors of the sample surface (Fig. 6).

*1 As of February of 2021, the plugins available for the batch analysis are Powder XRD, XRR, HRXRD, and Stress.

4.2. Small flake of serpentinite

Serpentinite is an ultrabasic rock containing serpentine $(Mg,Fe)_3Si_2O_5(OH)_4$ as the principal mineral. It is produced when peridotite etc. is serpentinized after reacting with water. The mineral flake was measured at a total of 240 points in the X range of −9.5 to +9.5 mm and the Y range of −7 mm to +4 mm with X and Y step sizes of 1 mm. At each measurement point, a 2D scan was performed in the 2θ range of 5 to 80° at a scan speed of 10°/min.

As in Section 4.1, peak search was performed around $2\theta = 24.4°$ and the crystallite size distribution calculated based on the peak is overlaid on the photo of the serpentinite flake (Fig. 7). Here a contour map is applied instead of a dotted color map so that the sample photo can be seen clearly. The contour lines on the black part of the serpentinite are shown in blue, and the lines on the other parts are shown in green, yellow, or red. It indicates that the crystallite sizes of the black part are 50 Å smaller than those of the other part.

When you look at the contour map of Fig. 7 carefully, there are no lines around the bottom-left and top-right corners. As described in Section 4.1, you can confirm the reason from the XRD patterns displayed by selecting the measurement points around the bottom-left and top-right corners. The Data Visualization plugin provides a "slideshow" feature. Each measurement point is selected one by one starting with the No.1 point. Then the corresponding XRD pattern will be displayed and switched to the next in the Data Image panel on the right side. Watching the slideshow, the data sets collected at the points where the contour lines are absent showed amorphous patterns, which indicated the bottom-left and top-right corners were just glass plate without the serpentinite flake.

You can watch a slideshow example at the following link.
https://youtu.be/sAv9LEBK0O8

5. Summary

The Data Visualization plugin analyzes XY mapping measurement data sets together and displays the distributions of various physical parameters on the sample photo. As an example in this article, the XY mapping analysis results for the micro area of the samples using the CBO-μ unit were described. The CBO-μ is a very convenient optical device for making XY mapping measurements with step sizes of 0.3–1 mm and analyzing the distributions of some physical parameters for a sample with a size of 0.5 up to 2 or 3 centimeters square.

Furthermore, the Data Visualization plugin is not just for the sizes of the samples described here, but is also useful for other samples with various sizes, such as large wafers, small capacitors, etc. XY mapping analysis can be performed for data sets collected using various measurement optics, sample stages, etc. We hope that Rigaku diffractometers and the Data Visualization plugin of SmartLab Studio II will be utilized in a variety of purposes such as state analysis, physical property analysis, degeneration analysis, etc. of micro areas.

References

(1) Yuji Shiramata: *Rigaku Journal*, **32** (2016), No. 1, 3–9.
(2) Takeshi Osakabe: *Rigaku Journal*, **33** (2017), No. 1, 15–19.
(3) *Rigaku Journal*, **33** (2017), No. 2, 29–31.
(4) *Rigaku Journal*, **34** (2018), No. 2, 35–36.