BL43IR
Infrared Materials Science

1. Introduction

BL43IR is dedicated to infrared (IR) microspectroscopy in the wavenumber region from 10,000 cm^{-1} to 150 cm^{-1}. The beamline has three microscopes: a high-spatial-resolution microscope, a long-working-distance microscope, and a magnetooptical microscope. The microscopes are used with a Fourier transform spectrometer. We are developing new instruments for microspectroscopy to upgrade equipment already in operation but also to recruit new users for various sample environments.

2. Upgrades of the microscope and related gadgets

For the continuous maintenance of the long-working-distance (LWD) microscope, we have prepared new aperture parts, which are 1000 μm square and 1500 μm circular ones. There are two different optical alignments depending on the samples, one of which uses position- and size-adjustable apertures while the other uses position- and size-fixed ones. These new aperture parts are for the position- and size-fixed alignment and are useful in experiments that require the analysis of the chemical composition or physical properties in areas of the same size throughout the experiments.

We also replaced the dried air purging components to improve the operability. The purge system of the LWD microscope is shared with the magnetooptical microscope and thus it was previously necessary to change the dried air piping by hand when the microscope used was switched. We introduced a Y-connector and two valves to switch the flow more easily. Using these gadgets increases the efficiency of the measurements. We also changed the purge cover over the optics located between the microscope and the interferometer to fill the optical path with dried air. The new cover is made of transparent acrylic, which increases the visibility and efficiency of the optical alignment.

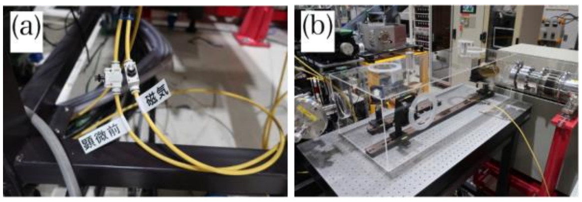

Fig. 1. (a) Y-connector and valves for flow control. (b) Purge cover for efficient optical alignment.

3. Attenuated total reflection (ATR) measurement using a tensile stage

ATR spectroscopy is a technique for measuring IR spectra using evanescent waves. In ATR measurements, the IR light passes through an ATR crystal whose refractive index is sufficiently higher than that of a sample and is reflected from the inner surface in contact with the sample. At the reflection point, evanescent waves emanating from the ATR crystal interact with the sample, which is pressed against the crystal, enabling spectral measurements. ATR spectroscopy is a widely used technique because of its advantages, such as simple sample preparation. For transmission measurements, the thickness of the sample should be only several microns (~5 mm, for example), but there are many cases where it is difficult to prepare such a thin sample. ATR measurements are performed by pressing the crystal against the surface of the sample, so there is no thickness limit, which is an advantage of the method. In 2022, we installed a tensile stage (LINKAM, TST350) on the IR microscope (BRUKER HYPERION2000) at the high-spatial-resolution microscope station and prepared equipment to perform ATR measurements, as shown in Fig. 2. Figure 3(a) shows a photograph of the tensile stage. The film-like sample is clamped by the parts indicated by the arrows and is stretched by moving the parts in the directions of the arrows. Figure 3(b) is a side-view schematic of the ATR crystal pressed against the sample fixed on the tensile stage. The ATR crystal, represented by the green hemisphere, is pressed against the sample for spectral measurement. The starred position is where the ATR crystals are pressed. The part of the stage indicated by the cross has a structure that protrudes in the vertical direction. When the sample is placed directly on the tensile stage, the holder of the ATR attachment hits the cross-marked area, and the ATR crystal cannot be pressed against the sample. Therefore, we placed small blocks, indicated by red diagonal lines in Fig. 3(b), under the sample so that the ATR crystal could be pressed against it. As a result, it became possible to measure the spectrum of a sample in a stretched state. Since it is an ATR measurement, even samples with a thickness of several hundred micrometers can be measured, expanding the range of user applications.

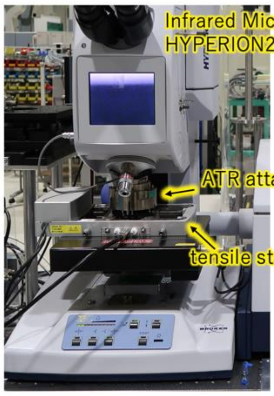

Fig. 2. Photograph of the tensile stage installed on the IR microscope at a high-spatial-resolution microscope station.

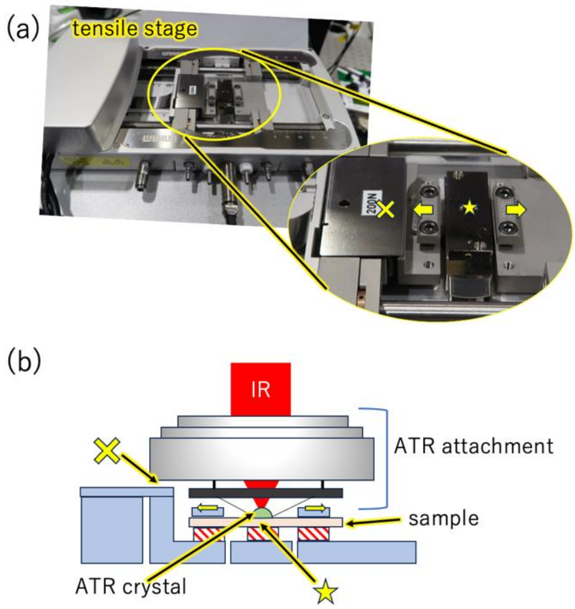

Fig. 3. (a) Photograph of the tensile stage and (b) side-view schematic of the ATR crystal pressed against the sample fixed on the tensile stage.

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