Problems for quantitative absorbance measurements by micro-FTIR spectroscopy

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Infrared spectroscopy has several exclusive advantages that this method is able to determine water contents of minerals (e.g. Paterson et al., 1982; Libowitzky and Rossman, 1997) and to approximate active orientation of the oscillator (e.g. Shinoda and Aikawa, 1994; Libowitzky and Rossman, 1996). Fourier transform infrared microspectrometer (micro-FTIR), especially, displays its greatest force for the measurement of the water contents of hydrous minerals synthesized at high pressure, because high resolution spectrum are obtained for a short time in spite of a small sample. In general, the absorbance normalized by the thickness of the sample is used for the determination of water contents, based on the Beer-Lambert law that absorbance is in proportion to the thickness of the sample. However, light path length in the sample equal to the thickness of the sample in only cases where the direction of the incident light normal to the sample plane. In micro-FTIR, the incident light normal to the sample plane does not exist, because the incident light is focused by the Cassegrainian condenser mirror. Therefore, in order to obtain correct absorbance of the sample, it is important to consider effect of the deviation of the incident light form normal to the sample plane in the case of micro-FTIR measurement. In this study, we investigated how much the difference between normal and not normal, in other words focused, incident light effects on the absorbance.

The doubly polished section of a andesitic standard glass (hota1, water contents: 1.24wt%, the thickness around center of the sample: 105micrometer) synthesized by Kawamoto et al. (2003) was used as a optically isotropic body. The thin section cut parallel to (001) of topaz from Gilgit, Pakistan as a optically anisotropic body as samples was used. IR spectra were measured by Fourier transform infrared spectrometer (Perkin Elmer: SPECTRA GX). Glober lamp as a light source, KBr beam spliter, and MCT detector were used in this spectrometer. The resolution of obtained spectra was 4cm-1. The measurable wavenumber range was 750-7800cm-1. On the measurement with macro optical system the incident light is normal to the sample plane, because direct beam was a incident light. On the other hand, on measurement with micro optical system the incident light is not normal to the sample plane, because the sample is set on a stage between a pair of the Cassegrainian condenser mirrors. Aperture sizes were phi100micrometer for micro optical system and phi200-500micrometer for macro optical system. IR spectra were almost obtained by 100 scans, but IR spectra of the standard glass using by macro optical system were obtained by 20000 scans in order to obtain sufficient good S/N. Unpolarized light as an incident light was used for all measurement.

The absorbance of OH stretch of the standard glass used focused light is 0.92-1.05, one of topaz used the incident light normal to the sample plane is 0.86. The latter is about 7-22% larger than the former. This difference would be derived from the light path length transmitting in the sample. The refractive index of the standard glass is supposed to be 1.55, the light path length in the sample in the case of focused light is larger than that of using the incident light normal to the sample plane about 3-9%. In the case of topaz, as well as the standard glass, the ratio between absorbance obtained by both optical systems is 180%. These results indicate the significant effect of the direction of the incident light on the absorbance.