Quantitative analysis of water concentration in melt inclusions by reflectance micro-FTIR spectroscopy

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A technique based on micro reflectance spectroscopy using a vacuum Fourier Transform infrared (FTIR) spectrometer has been developed for the quantitative measurement of water content in melt inclusions in phenocrysts. Water in magma strongly affects how a volcano erupts. Thus it is important to determine the amount of water dissolved in the magma in order to predict how the eruption develops. Previously, a FTIR micro reflectance measurement was proposed to be a promising method to determine the concentration of water in volcanic glasses rapidly because of its great advantage in sample preparation. It requires neither doubly polished wafers nor measurement of sample thickness. However, application of the method to natural samples has been limited so far because the resonance due to the O-H stretching vibration of dissolved water in the glass observed around 3650cm^{-1} is weak. Moreover, requirement of smaller apertures for measurement of melt inclusions with several tens micron in diameter significantly decreases the signal-to-noise ratio. Consequently quantitative analysis of water in such a small melt inclusion was considered to be difficult. We overcome the difficulty by two improvements.

Firstly a vacuum FTIR apparatus was introduced. Evacuation the whole light path by a vacuum pump (<100 Pa) can reduce more efficiently noisy absorption around 3650cm^{-1} due to water vapor than purging dry N_2 gas. Thirty-one glasses with various water content ranging from basaltic to rhyolitic composition were synthesized by an internal heated pressure vessel. Reflectance IR spectra were taken between 650-7000cm^{-1} on singly polished glass samples. The maximum amplitude of the signal at around 3650 cm^{-1} resonance normalized to a baseline of reflectivity was plotted against water contents of the samples determined by Karl-Fischer titration (ranging from 0.1 to 5.1 wt%), and a linear correlation was obtained. It appears that the slope of the correlation line slightly depends on the bulk composition. When a reflectance spectrum is taken with a microscope aperture of 20x20 micron and 1024 scans, the intensity of noise around 3650cm^{-1} is 0.02 (1σ), corresponding to approximately 0.2 wt% H_2O.

As the second improvement, we devised a correction method for spectrum interference by host crystal. When a small melt inclusion is analyzed, some signal from host crystal may overlap the spectra of melt inclusion, resulting in underestimation of water content in the melt inclusion. However, fortunately enough, most silicate crystals have distinct reflection peaks in the region 800-1200cm^{-1}. Using those peaks, the contribution of host crystal to the measured reflective spectra can be estimated within a precision of 10% relative.

Olivine-hosted melt inclusions collected from 2011 Shinmoe-dake eruption were studied as a practical application of the new technique.