Development of High-pressure Optical Cells and the Progress of Basic Research in Experimental Geochemistry

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Abstract: Two types of high-pressure optical cells have been installed in a modern hydrothermal laboratory in the newly established Institute of Deep-sea Science and Engineering (IDSSE), Chinese Academy of Sciences, in Sanya, Hainan. One type is with diamond windows (Bassett et al., 1993) for temperatures from -190 to 1000°C and pressures up to 3 GPa, and the other with fused silica windows (Chou et al., 2005; Chou et al., 2008) for temperatures from -190 to 600°C and pressures up to 160 MPa. Because of the transparency of these windows, the major advantages of these high-pressure optical cells include the allowance of (1) in situ observations and spectroscopic analyses of the samples, and (2) continuous recording during experiments for later reviews and kinetic studies.

Detail descriptions of the hydrothermal diamond-anvil cell (HDAC) and its geological applications were given previously (e.g., Chou, 2003; Fang and Smith, 2009; Schmidt and Chou, 2012). The major applications include measurements of mineral solubilities, detections of phase transitions, and formulations of structures of hydrothermal solutions based on either Raman spectroscopic analyses or synchrotron X-ray absorption fine structure studies (see examples cited in Schmidt and Chou, 2012). The newly modified HDAC (type VT; Li et al., 2016) has been used for routine experiments at temperatures up to 1000°C and pressures up to 3.5 GPa with no or only minor repairs needed between runs; the modifications greatly improved the efficiency of HDAC.

Two types of optical cells using fused silica windows have been installed; one is the high-pressure optical cell (HPOC; Chou et al., 2005) and the other is the fused silica capillary capsules (FSCC; Chou et al., 2008). The outer diameters of the commonly used fused silica tubes are 0.30-0.67 mm and the inner diameters are from 0.05 to 0.3 mm. For the HPOC, one end of the fused silica capillary tube is sealed with a hydrogen flame, and the other open end is connected to a pressure line, so that fluid of known composition can be loaded and the pressure of the sample can be measured with a pressure transducer. For the FSCC, both ends of the fused silica capillary tube are sealed with a hydrogen flame. The FSCC (normally less than 2 cm in length) can be fitted in a specially designed heating-cooling stage for in situ observations and Raman spectroscopic analyses at temperatures between -190 and 500 or 600°C; the stage designed by Linkam can control sample temperatures up to 500°C and that by INSTEC up to 600°C (Chou, 2012). The FSCC can also be heated in a cold-sealed pressure vessel with or without external Ar pressure for durations up to few months. The quenched samples can then be analyzed by a non-destructive method (i.e., Raman spectroscopy) or a destructive method (i.e., gas chromatography). The detail descriptions and geological applications were given in Chou et al. (2005; 2008), and Chou (2012).

In China, through my direct or indirect inputs, these optical cells have been installed in several hydrothermal laboratories in universities (e.g., Zhongshan Univ., Nanjing Univ., Zhejiang Univ. of Technology, China Univ. of Geosciences at Wuhan, Univ. of Petroleum (east China) at Dongying, China Univ. of Science and Technology at Hefei) and research institutes (e.g., Institute of Geochemistry, Chinese Academy of Sciences at Guiyang and Guangzhou, Chinese Academy of Geological Sciences at Beijing, Petroleum Exploration and Production Research Institute of SINOPEC at Wuxi; Research Institute of Petroleum Exploration & Development, PetroChina at Beijing). I also introduced these optical cells to other countries; HDAC to Hans Keppler (Bayerisches Geoinstitut, Bayreuth, Germany) and Christian Schmidt (Deutsches GeoFoschungsZentrum (GFZ), Potsdam, Germany), and HPOC and FSCC to Jean Dubessy (Lorraine Univ. at Nancy, France), Peter Tremaine (University of Guelph, Canada), Julien Bourdet (Commonwealth Scientific and Industrial Research Organization, Australia), and among others. These optical cells have been applied in the study of geological fluids, especially the recognition of the existence of the trisulfur radical ion (S_3^-) in hydrothermal solutions at temperatures above 150°C. Because S_3^- ion is non-quenchable, its existence can only be recognized in situ at elevated pressure-temperature conditions. This new recognition has significantly improved our ability for the construction of ore formation models because this ion has important effects in the transport of many metals, including Au, Pt, Pd, Mo, and Re, and their related ore formation processes (Pokrovski and Dubrovinsky, 2011; Pokrovski and Dubessy, 2015; Pokrovski et al., 2015).

By using these optical cells, my research team at IDSSE has been doing the following basic research: (1) the vapor pressure measurements of H_2O and aqueous solutions containing salt(s); (2) establishment of a Raman spectroscopic data bank for geological fluids in the system COHNS at various pressures and temperatures; (3) establishment of equations of state of H_2O and aqueous solutions containing salt(s); and (4) development of a hydrogen fugacity sensor, based on FSCC and quantitative Raman spectroscopic analysis method, for redox control and

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measurements in hydrothermal experiments at temperatures below 400°C. The development of these high-pressure optical cells since 1990 has made significant contributions for the progress of basic research in experimental geochemistry.

Key words: hydrothermal laboratory, optical cells, diamond windows, fused silica windows, in situ Raman spectroscopy, basic research in experimental geochemistry

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