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Retrieval of uncracked single crystals from high pressure in piston-cylinder apparatus

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ABSTRACT

Single crystals of San Carlos olivine have been pressurized to 2.0–3.0 GPa, 1200 °C and retrieved with few or no cracks from piston-cylinder apparatus. Graphite plugs with the same dimensions as the crystals (2.28-mm outside diameter, 1.27 mm thick) were placed above and below the crystals inside the capsules and apparently compensate stresses produced during depressurization and quenching. Specimens were depressurized and cooled at rates of 100–200 MPa/min and 100–150 °C/min, respectively. Cooling rate is a major factor controlling crack formation.

INTRODUCTION

Crystalline and glassy materials run in piston-cylinder (solid-medium) apparatus invariably return from high pressure and temperature with cracks oriented normal to the axis of loading and unloading (referred to hereafter as horizontal cracks). For the most part, these cracks are thought to form during depressurization and quenching, although some cracks may form during pressurization as well. In our laboratory, we have been attempting to measure the solubility and diffusivity of C in olivine at high pressure (Tingle and Green, 1987; Tingle et al., unpub. ms., 1988). As well illustrated by the controversial water weakening of quartz and water diffusion in quartz (e.g., Kronenberg et al., 1986), cracked specimens complicate the interpretation of penetration of C or H as lattice diffusion, because cracks may serve as an alternate penetration pathway. Other limitations imposed by the presence of cracks include (1) the inability to remove a specimen intact from its capsule for further examination without impregnation and (2) restrictions on the size of analysis beam (electron, X-ray, deuteron, laser, etc.) that can be utilized. Uncracked single crystals of quartz have been successfully retrieved from pressures up to 1.5 GPa using very slow pressurization and depressurization (Kronenberg et al., 1986) and water-filled canisters (Rovetta et al., 1987; Gerretson et al., 1985). This note relates techniques developed to return single crystals of olivine from 2.0–3.0 GPa and 1000–1600 °C with very few or no cracks at all using conventional 1.27-cm piston-cylinder apparatus and relatively rapid pressurization and quenching.

EXPERIMENTAL TECHNIQUES

Specimens (2.28-mm outside diameter) were cored from oriented single crystals of San Carlos olivine, sliced, and polished into wafers or cylinders. Crystals oriented with (010) normal to the axis of loading were not used because it was anticipated that such specimens would be prone to formation of cleavage fractures on decompression. Initially, the specimens were 2–4 mm long, but the crack density (actually, the number of horizontal cracks through the center of the crystal per unit length) was observed to decrease exponentially with specimen length from 11 cracks/mm at 3.75 mm to 3 cracks/mm at 2 mm. Decreasing the depressurization rate from 1500 to 50 bars/min showed no measurable effect. Consequently, a length of 1.27 mm (thickness of a petrographic slide) was chosen. Specimens were then loaded with ^{14}C-labeled graphite with and without a fluid source (silver oxalate or oxalic acid monohydrate) into Pt capsules.

The Pt tubing (3-mm outside diameter, 0.05-mm wall thickness) was cut into lengths (8–10 mm) and annealed. One end of the tubing, chucked in a pin vise or collet, was turned to a point on a lathe using a hemispherical tool (like a 0.125-in. (0.318-cm) ball bearing soldered onto a steel rod) at 300–400 rpm. The points were arc-welded shut, producing a smooth bulbous weld that was stamped flat with a drill blank. The other end of the capsule was subsequently welded shut using the "ash-can" technique of Sneeringer and Watson (1985).

An important modification to the specimen assembly was the addition of graphite plugs (same dimensions as the crystals) and metal foil (Au or Mo) above and below the crystal. The metal foil was originally contrived to inhibit Fe loss from the single crystals, and graphite was added to provide an oxygen buffer for the added fluids. The first experiment with graphite plugs produced a specimen with two horizontal cracks over its 2-mm length. Apparently, the graphite's elastic behavior compensates stresses produced during depressurization and cooling. Hence, pressurization and depressurization over several hours (Kronenberg et al., 1986) does not seem to be required.

The sealed capsules were fitted into HBR-grade boron nitride (fired at 1100 °C for 24 h) machined with a flat-bottomed hole 3.18-mm outside diameter (Fig. 1). The cupped-end of the capsule was filled with alumina powder after the boron nitride was inserted into the graphite furnace; a crushable alumina tube for the thermocouple was fitted on top. Pyrex glass tubing and pressed cylinders of NaCl surrounded the furnace.

Temperature was monitored by W_5Re_{95}-$W_{26}Re_{74}$ thermocouples whose emf was not corrected for the effect of pressure. The piston was advanced with an Enerpac hand pump; one full stroke was equivalent to 300–500 MPa.

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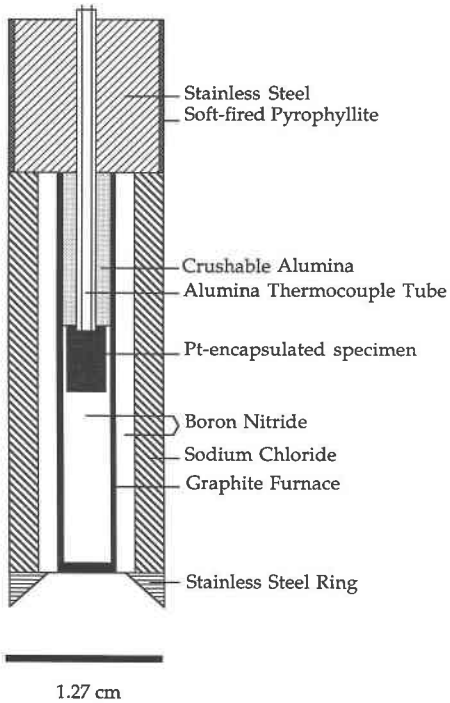

Fig. 1. Scale drawing of sample assembly.

Specimens were taken to run conditions by (1) pressurizing to 100–200 MPa, (2) heating to 600–800 °C, (3) pressurizing to 1000 MPa, (4) heating to 100 °C, (5) pressurizing to run pressure (2000–3000 MPa) or slightly greater, and (6) heating to the desired run temperature. The pressure was adjusted to the desired run pressure by bleeding off 100–200 MPa (hot piston-out technique). Specimens were depressurized by (1) cooling at 100 °C/min to 1000 °C, (2) depressurization at 100–200 MPa/min to 200–300 MPa or less, (3) cooling at 100–200 °C/min to 100 °C or less, and (4) final release of pressure, if necessary.

A slow steady temperature decrease is essential to retrieving uncracked crystals, because the thermal-expansion behavior of olivine seems to be a major contributor to horizontal crack formation (−3.8 vol% from 1000 to 100 °C, as opposed to 0.5 vol% on decompression from 1000 to 100 MPa). The significance of temperature-decrease rate was realized when single crystals at 150 °C shattered after being tossed into room temperature acetone for cleaning. Also, specimens invariably displayed horizontal cracks when the power was shut off to the furnace after depressurization at 1000 °C and slow cooling to 400–600 °C.

Results and discussion

Three specimens and their pressure-temperature histories are shown (Figs. 2, 3). In two samples containing CO_2-H_2O fluid (Figs. 3b, 3c), a prominent vertical crack formed. These cracks (and vertical cracks observed in other specimens) show no preferred crystallographic orientation. Most specimens containing fluid display vertical cracks, and it seems reasonable that fluid would enhance crack formation. No cracks were observed in the fluid-absent specimen, unless they fortuitously lay in the plane of the section. A couple of horizontal (unloading) cracks were produced in ASC-IX3 (Fig. 3b); these appear to have nucleated at the corner and propagated along (100) subgrain boundaries. Hence, application of the techniques described here should not be expected to always produce success because of the variety of crystal defects extant in natural starting materials, particularly San Carlos olivine.

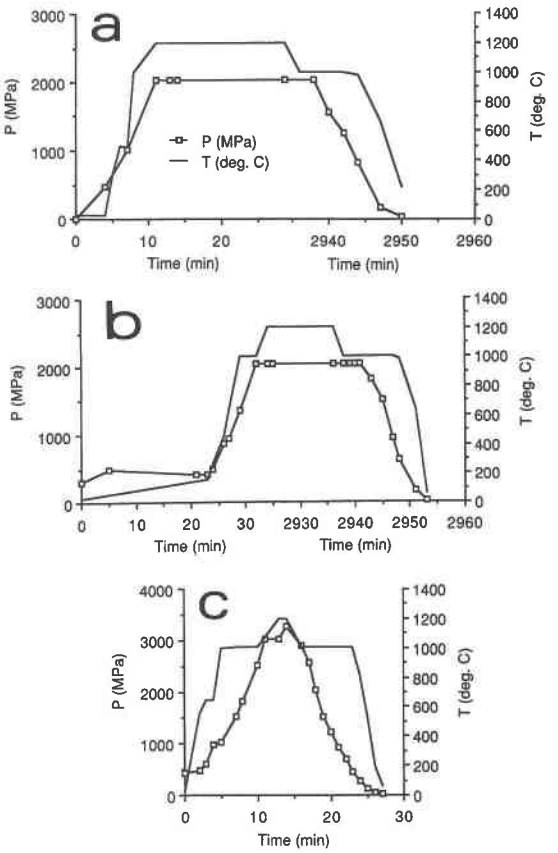

Fig. 2. Pressure-temperature histories for (a) ASC-IX2, (b) ASC-IX3, and (c) ASC-IX5. Specimen ASC-IX5 (c) was a zero-time diffusion experiment; the other two specimens, (a) and (b), were annealed for 2 d at 1200 °C and 2.0 GPa. The pressure- and temperature-decrease rates are approximately −200 MPa/min and −150 °C/min, respectively. The 20-min delay after initial pressurization of ASC-IX3 was due to repair of a leaky water-cooling line.

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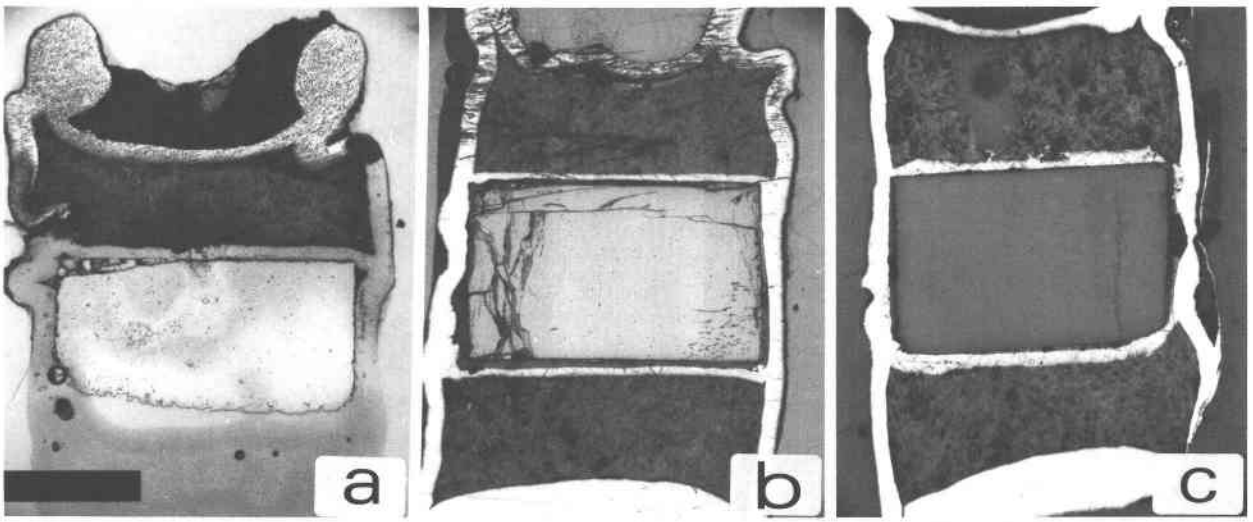

Fig. 3. Photomicrographs of specimens (a) ASC-IX2, (b) ASC-IX3, and (c) ASC-IX5. Scale bar = 1 mm. In all specimens, [001] is parallel to the long axis of the capsule. Specimen ASC-IX2 (a) is poorly polished because the section was ground too thin; one end of the capsule plucked off the section during preparation. No cracks were observed in this specimen, except for the corner. The sharp corners of the polished specimens are fragile and commonly break during pressurization owing to subtle misfits in the sample assembly. Specimen ASC-IX3 (b) displays a couple of horizontal cracks at one end and a prominent vertical fracture network on the left. The lower right corner of the specimen contains several fluid inclusions; the fractures emanating from them probably formed during depressurization due to the differences in compressibility between fluid and crystal. Specimen ASC-IX5 (c) displays only a single vertical fracture.

References cited

Gerretson, J., Paterson, M.S., and Bitmead, J. (1985) Improved procedures for diffusing "water" into quartz at high temperature and pressure (abs.). EOS, 66, 1144.

Kronenberg, A.K., Kirby, S.H., Aines, R.D., and Rossman, G.R. (1986) Solubility and diffusional uptake of hydrogen in quartz at high water pressures: Implications for hydrolytic weakening. Journal of Geophysical Research, 91, 12723–12744.

Rovetta, M.R., Blacic, J.D., Hervig, R.L., and Holloway, J.R. (1987) Aluminum and hydrogen defects in quartz: Enhanced diffusion at high pressures and high f(H2) (abs.). EOS, 68, 627.

Sneeringer, M.A., and Watson, E.B. (1985) Milk cartons and ash cans: Two unconventional welding techniques. American Mineralogist, 70, 200–201.

Tingle, T.N., and Green, H.W. (1987) Carbon solubility in olivine: Implications for upper mantle evolution. Geology, 15, 324–326.

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