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A new reaction cell for hydrothermal solution equipment

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Abstract

A new reaction cell consisting of a deformable gold body pressure-sealed to a detachable titanium top has been developed. This cell, used in conjunction with a commercially available pressure vessel and noncontaminating sampling valve block, is effective in hydrothermal experimental investigations.

Introduction

During the past ten years experimental studies involving reactions between rocks of varying chemistry and mineralogy and aqueous fluids have received much attention. These studies, usually performed at elevated temperatures and pressures, are typically designed to obtain a better understanding of directions and magnitude of chemical exchange and mechanisms of alteration mineral formation. The ideal experimental system should be inert, and should in no way participate in the reaction under investigation, and furthermore, should permit the investigator to monitor the progress of the reaction.

These requirements were the guiding philosophy for the Hydrothermal Solution Equipment (HSE) designed by Dickson *et al.* (1963). This equipment consisted of a deformable reaction cell held in place within a stainless-steel pressure vessel surrounded by distilled H_2O. Periodically, aqueous samples could be "squeezed" from the reaction cell by a force exerted on the pressure fluid surrounding the cell by a high pressure hydraulic pump and analyzed. For this procedure, the reaction cell must be deformable without rupturing and therefore was constructed of teflon. Teflon, however, has two disadvantages; (a) it can only be used below 285°C and (b) it acts as an osmotic membrane, allowing gases and dissolved molecular species (H_2O, H_2S, H_4SiO_4, CO_2) to migrate through the cell walls.

Reaction cells constructed entirely of gold have been used to eliminate the problems associated with the teflon (Rytuba and Dickson, 1974). However, gold-cell fabrication is particularly tedious, requiring many hours of difficult welding. Furthermore, at the termination of each experiment, the gold cell must be cut open in order to retrieve the reactants. This operation is at best awkward, and because the cell is cut open it must be rewelded, which consumes time and also decreases the volume of the cell.

Therefore, we have designed a new reaction cell, which in conjunction with a commercially available pressure vessel provides a greatly simplified gold-cell technology.

Reaction cell

The pressure vessel housing the reaction cell (Fig. 1) is commercially available (American Instrument Company, Silver Springs, Maryland), and is constructed of chrome-vanadium steel. However, we have modified the closure piece to allow passage of a

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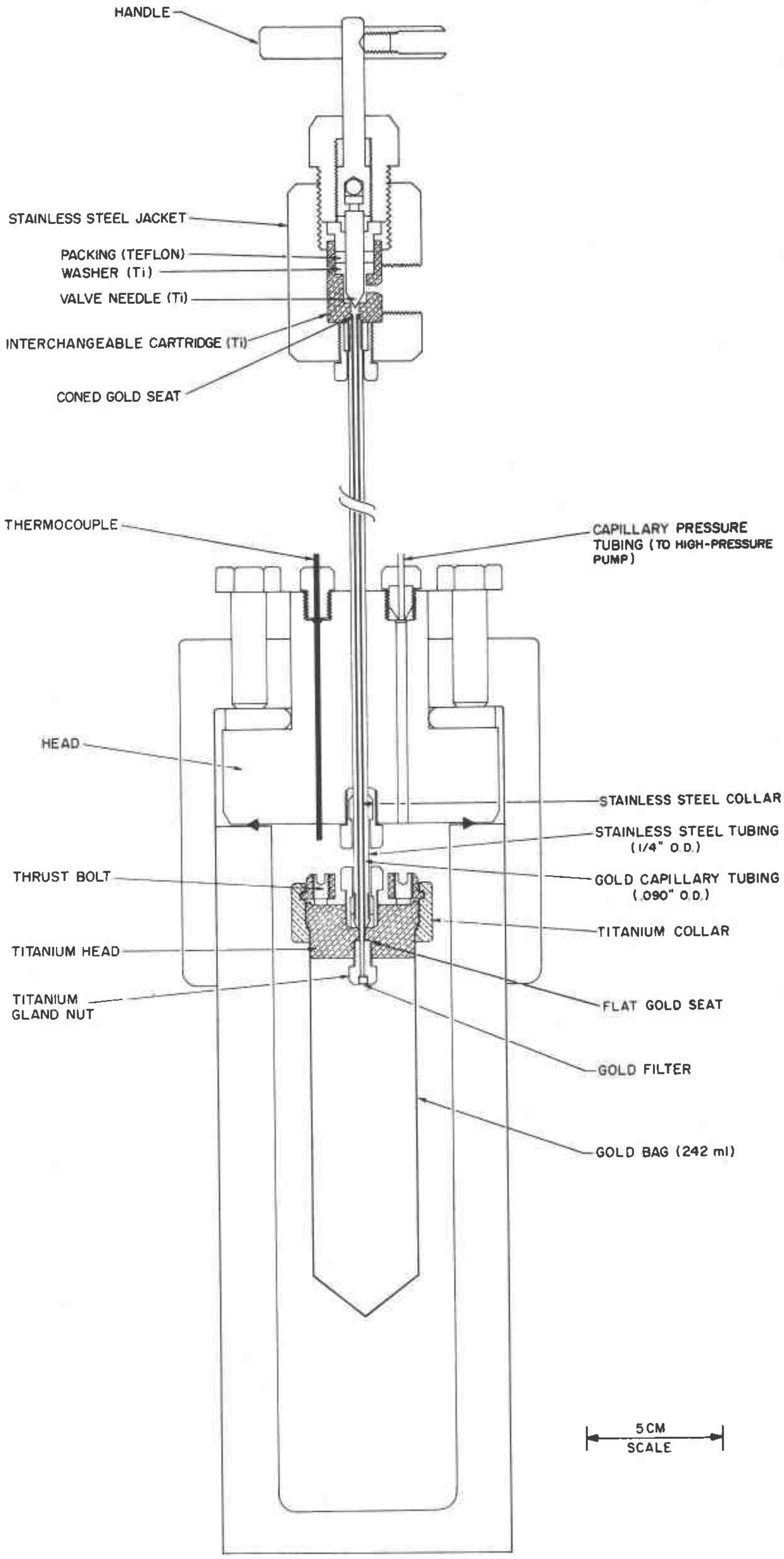

Fig. 1. Schematic illustration of hydrothermal solution equipment and new gold–titanium reaction cell.

thermocouple, capillary pressure tubing, and a 0.25″ O.D. stainless-steel exit tube for sampling. The exit tube is attached at one end to the reaction cell and at the other to a sampling valve block (Dickson and Gordon, patent pending). The inner cavity of this valve is constructed of titanium, which prevents contamination of the aqueous phase during sampling. A conventional cone seal secures the exit tube to the head of the pressure vessel.

Gold capillary tubing (0.090″ O.D. × 0.025″ I.D. × 16″) lines the inside of the exit tube. At the junction of the exit tube and reaction cell (Fig. 1), the gold tubing is flared slightly and is held in place within the reaction cell top by a titanium gland nut. The gland nut contains a sintered gold disk which serves as a filter for particles $\geq 2\mu$. At its upper end, the gold tubing is again flared and "coned" over the stainless-steel exit tube. Thus, the gold tubing, flared both top and bottom, establishes a permanent, contamination-free conduit for the passage of filtered aqueous samples from the reaction cell to the valve.

The reaction cell consists of a lower cylindrical-shaped gold body (1.75″ O.D. × 7″ length and 0.01″ wall thickness) and is purchased prefabricated (Mathey Bishop Company, Malvern, Pennsylvania). A single permanent weld is used to close the gold cylinder at the bottom. The top of the reaction cell is constructed of titanium (99.98 percent Ti).

The titanium head (Fig. 1) fits snugly into the reaction cell and serves as a cap; the gold extends approximately half-way up the flared portion of the head. A pressure seal between the titanium head and the gold–titanium collar is established by 6 thrust bolts threaded into a retainer plate (Fig. 2). These bolts force the titanium head against the collar, thereby making a pressure seal against the gold. Prior to initiation of the pressure seal the flanges of the retainer plate must directly underlie those of the collar.

Titanium was used in the construction of the reaction cell top because it is highly inert to such natural fluids as sea water, brines, and many dilute acids, even at elevated temperatures (Pettibone and Kane, 1963; Bischoff and Seyfried, 1978).

A major advantage of the titanium–gold reaction cell is that at the end of an experiment the top can be quickly and easily detached from the gold body. The removal of the top is accomplished by first removing all of the titanium thrust bolts from the retainer plate. The retainer plate is then oriented so that its flanges overlie the flanges of the titanium collar. Then 3 0.125″ bolts ($\simeq$1.75″ long) are inserted into the holes adjacent to retainer plate flanges. The bolts, which pass through the retainer plate, are then screwed into the 3 holes tapped in the titanium head (Fig. 2). Once the bolt heads contact the retainer plate, only a hand wrench is required to free the top of the reaction cell from the gold body. The removal of the titanium top from the gold permits convenient access to the reactants without having to cut open and subsequently reweld the gold cell.

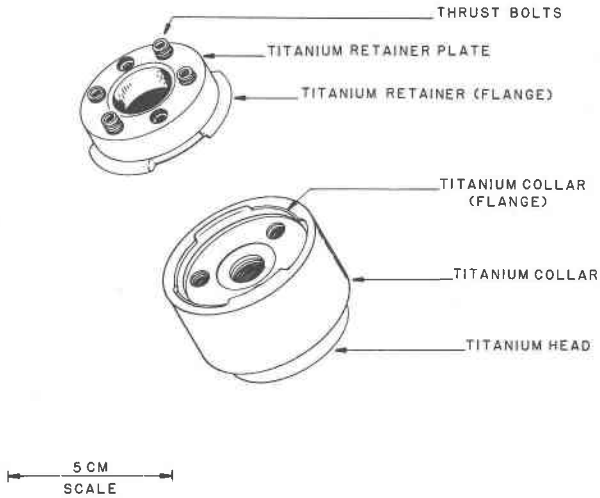

Fig. 2. Titanium closure for gold–titanium reaction cell.

Summary

The titanium–gold reaction cell used with a commercially available pressure vessel provides an effective means for hydrothermal experimental investigations. Periodically, internally filtered aqueous samples can be removed from the reaction cell during the experiment. Because the volume of sample removed is displaced by pressure fluid (H_2O) surrounding the cell, sampling is carried out at constant pressure as well as temperature. Thus the system is applicable to studies in which the chemistry of the aqueous phase requires monitoring with time.

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