AUTOCLAVE FOR CORROSION AND ELECTROCHEMICAL INVESTIGATIONS AT HIGH TEMPERATURES AND PRESSURES

P. S. Petrov, G. C. Raskin, and G. M. Florianovich

UDC 620.199

The authors propose an autoclave for electrochemical measurements in which the solution can be replaced during the test without lowering the pressure. The apparatus operates as follows. It contains several glass cells filled with solution which can rotate in a horizontal plane. Using a special mechanism the electrode can be moved from one cell to another.

The comparison electrode is placed inside the autoclave. Unlike the autoclave with external comparison electrodes, which are at room temperature [4] this model, used also by other workers [1-3], is simple in design and enables the auxiliary electrode and the comparison electrode to be transferred into a new cell together with the main electrode.

The autoclave (Fig. 1) consists of 4 liter thick-walled stainless steel cup 1 with cover 2. The cover is sealed by bolts 3 and a Teflon packing. The autoclave is heated in muffle electric furnace 4.

The cover has a mechanism for rotating 5, and lifting 6, three electrodes 7, gas connection 8, and pocket for thermocouple 9. The rotating mechanism consists of a rod passing through the lid center and

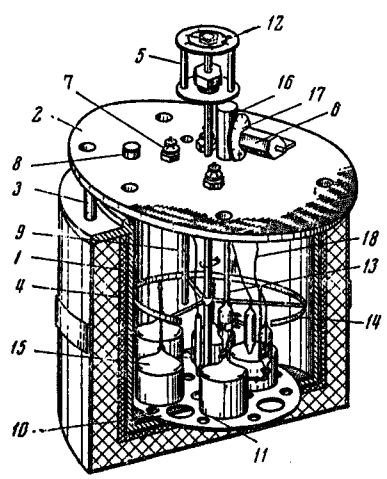

Fig. 1

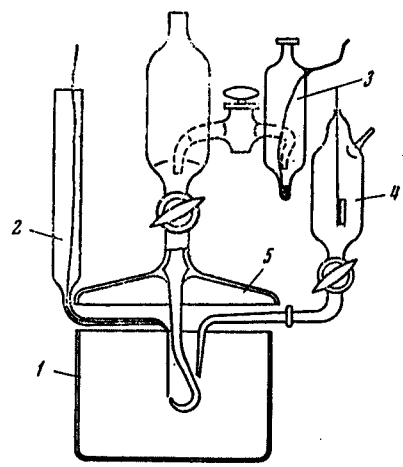

Fig. 2

Fig. 1. Autoclave for corrosion-electrochemical tests.

Fig. 2. Electrochemical cell for investigations at high temperatures and pressures ("Pyrex" glass): 1) cup-cell; 2) working electrode; 3) comparison electrode; 4) vessel for auxiliary electrode; 5) cover.

L. Ya. Karpov Scientific-Research Physicochemical Institute. Translated from Zashchita Metallov, Vol. 7, No. 4, pp. 436-438, July-August, 1971. Original article submitted August 5, 1970.

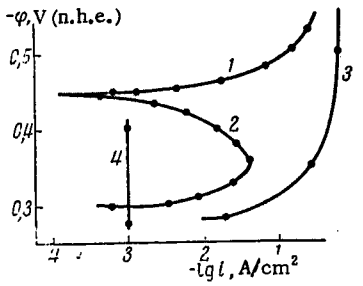

Fig. 3. Cathodic (a) and anodic (2) polarization curves measured on chromium in 1 N sulfuric acid solution at 150°C. Curves 3 and 4 are determined analytically representing the dissolution of chromium at 150°C as a function of the potential: 3) 1 N H_2SO_4; 4) 1 N H_2SO_4 + 0.592 M H_2O_2.

almost reaching the autoclave bottom, it can freely rotate in the Teflon bushing. The bottom end of the rod carries holder disc 10 with 6 holes taking 40-ml cell cups 11; the top end of the rod carries scale 12 with markings indicating the accurate position of cells. The rod can be rotated in any direction.

The lifting mechanism of the rod is mounted eccentrically on the cover and its height is 2/3 of the autoclave height. The bottom end of the rod carries box 13 with three electrodes and ring 14 with suspended cell lids 15. The opposite rod end is connected, through a worm transmission, to drive 16 which raises or lowers the electrodes to the required height (to 30 mm). The drive carries scale 17 indicating the position of electrodes with an accuracy of 0.5 mm.

Tapered electrical connections are inserted into the autoclave lid and insulated against it by Teflon bushings also acting as sealing elements. Inside the autoclave the cables are connected by platinum wire 18 to electrodes. The glass cell is shown in Fig. 2. To separate the electrode under investigation from the auxiliary electrode (platinum electrode) and the comparison electrode (silver chloride electrode) special vessels with fitted lids are used having a bellows and a Luggin capillary (Fig. 2). In replacing the solution the electrodes are moved in a vertical direction together with these vessels and electrolytic switches. All cell elements are mounted on the lifting rod box by stainless steel spring clamps.

To prevent the evaporation of the solution from cells 50-100 ml of water is poured into the autoclave.

For tests in an inert gas atmosphere special capillary tubes are used to deaerate the solutions in all parts of a cell which is followed by a repeated filling of the autoclave by pressurized inert gas and by its removal.

The proposed apparatus has a number of advantages and is much simpler than the double autoclaves used in corrosion investigations [5]. It enables the polarization measurements to be made in solutions of different compositions during a single test, electrochemical and analytical investigations to be combined, the changes in the dissolution rate of metal with time at various controlled potentials to be recorded, etc., during a single test. The use of Teflon as an insulating and sealing material makes possible (at pressure to 40 atm) the investigations to be carried out at temperatures of up to 200°C.

The possibilities of this method are illustrated by Fig. 3. The polarization curves 1 and 2 were measured in the first cell. The points of curves 3 and 4 were obtained in analyzing the solution for chromium ions in five other cells.

The results of the analytical determination in pure sulfuric acid (curve 3) agree with the data obtained earlier for lower temperatures, and attributed to the assumed chemical mechanism of chromium dissolution [6].

The evaluation of the dissolution rate of chromium in the presence of hydrogen peroxide (curve 4) shows that the retarding effect of the chemical dissolution of chromium in the presence of oxidizers, observed under laboratory conditions [7], also takes place at temperatures exceeding 100°C.

The authors are indebted to Ya. M. Kolotyrkin and V. A. Sokol'skii for their assistance.

LITERATURE CITED

1. A. I. Krasil'shchikov, Zh. Fiz. Khim., 26, 216 (1952).
2. G. F. Savchenkov and L. A. Uvarov, Zashchita Metal., 1, 636 (1965).
3. V. P. Kondrat'ev and S. V. Borbachev, Zh. Fiz. Khim., 35, No. 3, 671 (1961).
4. V. V. Gerasimov, A. I. Gromova, and A. A. Sabinin, Zavodsk. Lab., 24, No. 11, 1420 (1958).
5. D. F. MacLennan, Corrosion, 4, 181-184 (1961).
6. Ya. M. Kolotyrkin and G. M. Florianovich, Zashchita Metal., 1, 7 (1965).
7. Ya. M. Kolotyrkin, V. M. Novakovskii, and G. M. Florianovich, Zashchita Metal., 4, 619 (1968).