AN AUTOCLAVE FOR ELECTROCHEMICAL STUDIES AT HIGH TEMPERATURES AND PRESSURES

V.V. Gerasimov, A.I. Gromova and A.A. Sabinin

There are at present no methods of measuring electrochemical potentials and of recording polarization curves that are satisfactory for use at 300-350°C and 100-200 atmos. Autoclaves in which specimens can be polarized have been described [1]. The problem of measuring potentials has not yet been solved, though. The main difficulty is that it is not possible to measure the potential of a reference electrode placed in the autoclave under pressure relative to some standard electrode under normal conditions. An electrolyte bridge that will support the pressure and temperature in the autoclave is required.

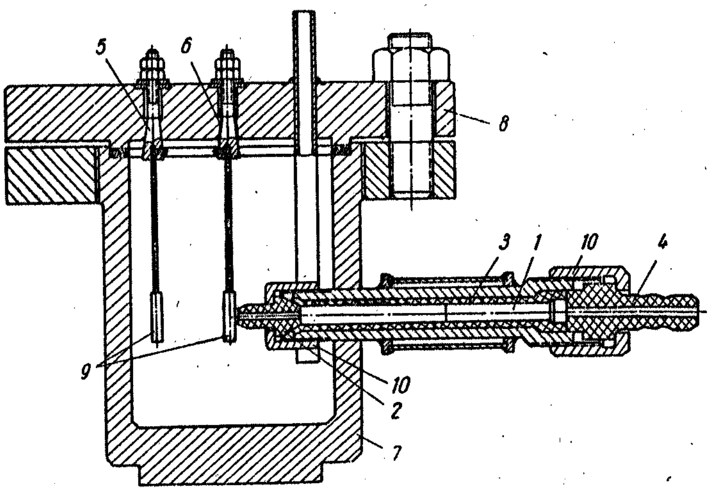


Fig. 1. The autoclave for electrochemical studies: 7) body; 8) lid; 9) specimen. See text for other parts.

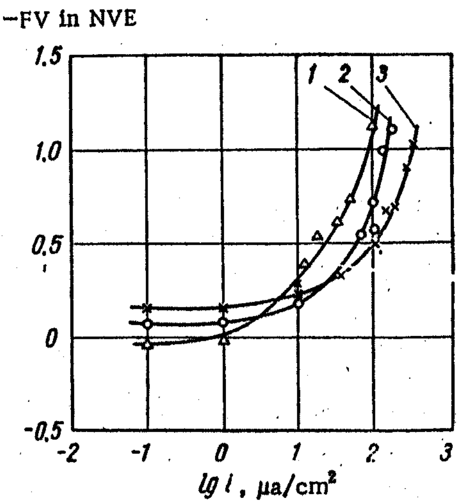


Fig. 2. Cathode polarization curves for 1Kh18N9T steel in distilled water: 1-3) 200, 300 and 350°C, respectively, for which temperatures the pressures are 15.8, 87 and 168 atmos.

Figure 1 shows an autoclave* for the purpose, which is fitted with such a bridge.

The wooden rod 1 is soaked in the test solution; it ensures that the vessel is sealed, and provides the circuit. The solution cannot come into contact with the autoclave because the nipple 2, the tube 3 and the coupler 4 are made of insulators (fluorinated plastics, micalex). The fluorinated plastic should not be worked above 200°C, so the bridge is cooled with running water. The calomel half-cell is joined to the coupler 4 via a glass bridge.

Bonnemay [2] has shown that the potential difference at the boundary between otherwise identical solutions at different temperatures is less than 10^{-6} v/°C. The error caused by this effect can therefore be neglected.

The polarizing current is fed to the specimen via the leads 5, which are insulated from the lid by the mica 6. The autoclave's capacity is 0.5 liter. The body and lid are made of 1Kh18N9T steel. The lid carried a thermocouple jacket and a pipe for coupling to a gauge.

Electrode process kinetics and potential-time curves can be examined, galvanic currents can be measured, and reference electrodes can be calibrated.

*V.A. Gavrilin worked on the design.

Figure 2 shows cathode polarization curves for 1Kh18N9T steel in distilled water. The cathode process is controlled by the rate at which oxygen ionizes, so the limiting diffusion current increases continuously between 200 and 350°C.

The autoclave can be used at temperatures up to 350°C, and at pressures up to 200 atmos.

LITERATURE CITED

[1] O. Fuchs, Z. Electrochem. 47, 2, 101 (1944); J.E. Draley and W.E. Ruther, Corrosion 12, 10, 20 (1956).

[2] M. Bonnemay, Proc. Meeting International Committee of Electrochemical Thermodynamics and Kinetics, 1954 (London, 1955) pp. 68-76; Referat. Zhur. Khim. 2, 3956, page 69 (1958).

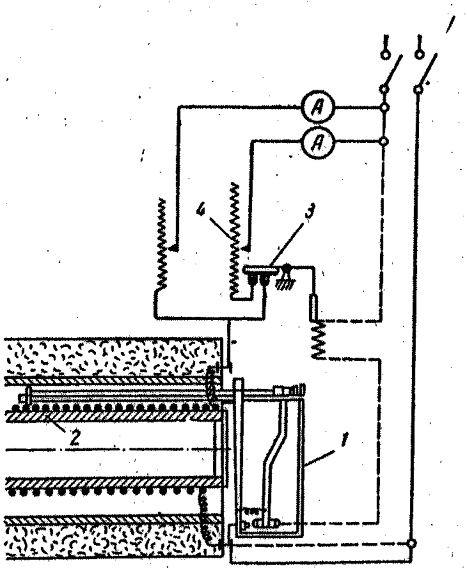
A LAG-FREE TEMPERATURE REGULATOR

V.I. Smirnov

(Kalinin Polytechnical Institute, Leningrad)

Zaitsev's dilatometric regulator [1] is widely used in resistance furnaces, but it has the disadvantage of using a large (10 x 25 x 400 mm) tube of austenitic (heat-resisting) steel as the sensing element.

We have designed and made a removable regulator 1 (see the figure), in which the sensing element is a nichrome strip 0.3 x 1.5 mm (other strip sizes, or wires, can be used); this strip is fixed to a quartz rod 0.5-1.0 m long at the inner end (the size of the furnace determines the size of the rod). The strip or wire runs along the quartz rod, and is fixed at its other end to the short arm of a lever with arms in the ratio 1:10.



Furnace circuit and dilatometric regulator system.

The long arm carries a contact, which operates, on the left, the mercury relay 3, which breaks the supply circuit 4, when the furnace 2 overheats; and on the right, the contact operates a warning bell when the furnace becomes too cool. If the overheating exceeds the control limits, the sprung contact operates a limit relay that switches the supply off.

The use of nichrome wire or strip reduces the lag and increases the accuracy, because the thermal capacity of the sensing element is reduced; the design is also simplified, and the regulator made cheaper.

The regulator can control temperatures to ±0.5°C in the range 500-1000°C. The accuracy is ±1°C at lower temperatures. The design has been tested with line voltages varying from 110 to 125 v.

LITERATURE CITED

[1] Ia.S. Ginzburg, Kotloturbostroenie, 4, 29-30 (1947).