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A Thyristor-Controlled Molybdenum Furnace for Work up to 1800°C

KARL-AXEL WILHELMI, WALTER BURGER and
MATS NYGREN

*Institute of Inorganic and Physical Chemistry, University of Stockholm, Box 6801,
S-113 86 Stockholm, Sweden*

An inexpensive molybdenum wire furnace for long period experiments up to 1800°C and ambient atmosphere inside the heating tube is described. The heating/cooling rates of the furnace are controlled and a constant working temperature is maintained, without any use of a transformer, by employing a thyristor SCR-type power control unit equipped with a potentiometer and a gear reduction set.

Electrical resistance furnaces for temperatures above 1350°C (the maximum working temperature for Kanthal A-1) frequently employ platinum, platinum alloys, or iridium metal as heating element. However, these kinds of furnace are rather expensive. This paper describes an inexpensive furnace employing a molybdenum wire wound on an Al_2O_3 tube with an upper temperature limit of 1800°C. In cases where the experimental conditions do not require a temperature stability of more than ±10°C, no special power control unit is needed. Measurements of electrical conductivity and thermoelectrical power, however, require a constant working temperature within 1°C or better. By using the power control unit described below, this requirement can be achieved.

THE FURNACE

The principle features of the furnace are shown in Fig. 1. The furnace consists of three parts: a supporting casing (1), a heating tube (2), and a lid (4).

The molybdenum heating wire, 0.8 mm in diameter, is wound on a gas-tight tube ("Morgan-Triangle" RR with O.D. 2.1 cm and I.D. 1.5 cm) and fixed to the tube by means of pure Al_2O_3 cement. The heating coil is divided into three sections with separate current terminals fastened on to the casing (not shown in the figure). The heat input near the ends of the coil is increased by spacing the resistance wire more closely at these points. The tube has a length

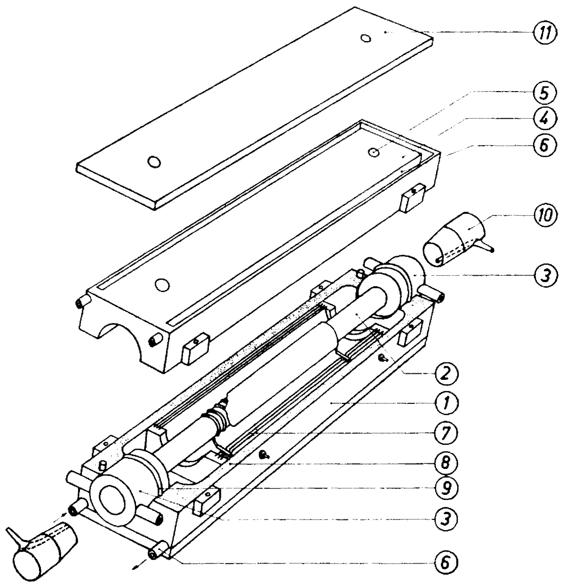

Fig. 1. Schematic diagram of the single-zone molybdenum furnace. 1, supporting casing; 2, heating tube and Mo wire; 3, brass supports; 4, lid; 5, gas inlet; 6, cooling channel with water connection; 7, lower reflector shields; 8, rubber gasket; 9, "O" ring; 10, glass joint; 11, lid cover.

of 80 cm and the heating coil is about 36 cm long. The ends of the tube are water-cooled *via* the two brass supports cemented (3) on the tube.

The casing and its lid are water-cooled and made of rolled aluminium (cross section 10 cm × 10 cm). Three split reflector shields (7) reduce the power consumption which is approx. 3 kVA for 1800°C in a furnace of the size described in this paper. The inner reflector shield is made of molybdenum, the second of nickel and the third of stainless steel.

Gas-tightness is obtained by using a rubber gasket (8) between the casing and its lid and "O"-rings (9) on the brass supports. This permits considerable thermal expansion of the tube. Nitrogen has been used for the inert atmosphere protecting the molybdenum wire. A simple membrane valve (not shown in the figure), connected to the gas inlet (5) on the lid and with a closed outlet, shows when a small overpressure is established and serves simultaneously as a tightness check and, moreover, excludes atmospheric oxygen and encompasses the expanding nitrogen during the heating procedure. Transparent ground-glass joints (10) provided with gas nipples, permit the use of an arbitrary specimen atmosphere.

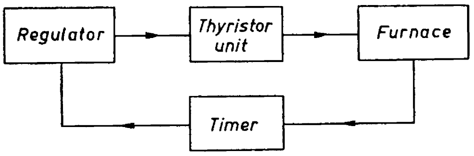

Fig. 2. Block diagram of the temperature regulator and timing device.

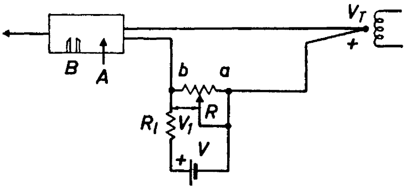

Fig. 3. Method of operation of the timer, see text.

TEMPERATURE REGULATOR AND TIMING DEVICE

Fig. 2 shows a block diagram of the set-up. The thyristor power control unit (Philips PR 7301, max. load 5 kVA) is employed as a correcting unit in an automatic control loop.

The control signal for the thyristor is produced by a "Getroduct" regulator. The output of this instrument is a low-voltage D.C. current which varies between 0 and 5 mA according to the position of the indicating pointer within the proportional region. This signal thus controls the output of the thyristor unit between the limits of 2 and 98 % of a nominal 220 V AC.

The temperature sensitive element connected to the regulator is a Pt—Pt/10 % Rh thermocouple. It is placed close to the windings and away from the hot zone in order to permit a temperature regulation above the upper limit of the thermocouple range.

The timer permits a gradual heating or cooling at a variable rate and hinders the development of too large currents during the first period of the heating process due to the low resistance of the windings when cold. The timer unit operates by connecting a variable voltage in series with the thermocouple (see Fig. 3). This is effected by means of a motor-driven variable resistance. Before starting, this voltage (V_1) is adjusted to a value slightly in excess of that delivered by the thermocouple at the desired temperature. When the motor is switched on, the voltage indicated by the regulator decreases at a steady rate until the indicating pointer (A) moves into the regulation region (B). The thyristor unit will then deliver current to the furnace so as to keep the sum of V_1 and V_T constant. This means that the furnace temperature will increase until V_1 reaches zero. The motor is then automatically switched off. The reverse procedure is followed when the furnace is switched off. A complete circuit diagram for the timer unit is shown in Fig. 4.

TESTS APPLIED TO THE FURNACE AND THE POWER CONTROL UNIT

The set-up has been tested during a period of three weeks with regard to stability and temperature distribution as well as heating and cooling rates. Temperature measurements above 1600°C have been performed by observing a specimen with an optical pyrometer—the long tube provides satisfactory black body conditions. In the temperature region below 1600°C, a Pt—Pt/10 %

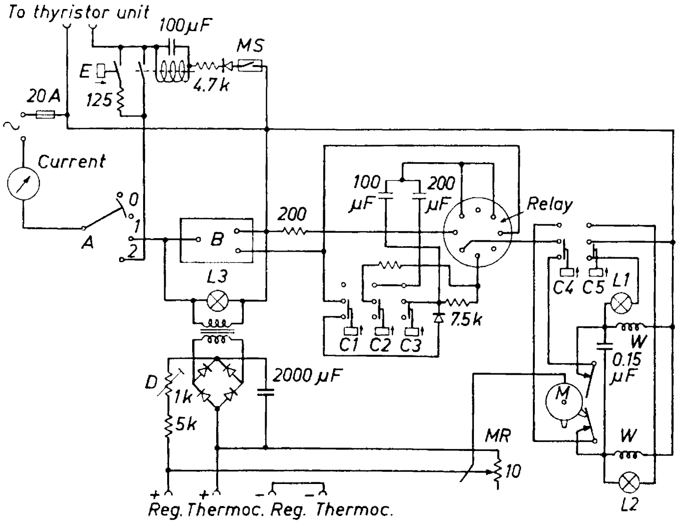

Fig. 4. Timer, circuit diagram.

A	Switch. Position 0: off; position 1: auxiliary voltage (V_1 Fig. 3) connected; position 2: thyristor voltage connected.
B	Bimetallic regulator.
C	Push-button switch in which C_1, C_2 and C_3 determine the switching-on and switching-off times of the relay and the motor *M* and C_4 and C_5 determine the direction of rotation of the motor.
E	Manual starting switch.
L_1-L_3	Control lamps.
MR	Motor driven rheostat.
MS	Micro-switch operated by failure of the water-cooling system.
W	Motor windings.

Rh thermocouple has been used. The thermocouple voltage has been measured by a potentiometer (Leads-Northrup type K-4) with an accuracy of 1 μV and, in the case of measurements extending over more than 10 h, by means of a recorder (Philips PR 3500) with an accuracy considerably better than 25 μV.

The temperature *versus* time measurements have shown that the fluctuations of temperature in the furnace are very small. The temperature is stable to within ±0.5°C during several hours and furthermore the deviation from the average temperature during a period of 60 h has been found to be less than 2°C at 1500°C.

The heating and cooling rates can be varied from 20°C/min to 0.02°C/min. Since the Al_2O_3 tube has a relatively poor resistance to thermal shocks, a heating or cooling rate of 20°C/min is not recommended for frequent use. A rate of 3—5°C/min is to be preferred and should result ultimately in a longer furnace life.

The uniformity of temperature throughout the hot zone depends very much on the design of the heating coil. Since the heating wire is divided into three sections, this permits a very fine adjustment of the current in each section. The best design of the heating coil for a given temperature region and purpose has to be found by trial and error. For the heating tube described above, it has thus been possible to obtain an axial temperature uniformity of ± 1°C over a length of 6 cm as well as two flat temperature zones with a maximum ΔT of 100°C. The latter is achieved by connecting a rheostat parallel to one of the sections. If a greater ΔT is required, this furnace possesses the advantage of simplicity in exchanging heating tubes, *i.e.* temperature profiles.

This set-up has been used for several purposes, *e.g.* quenching experiments, transport reactions in the temperature interval 1200—1600°C, preparative work and electrical conductivity measurements (below 1650°C). The quenching experiments were performed with the furnace in a vertical position. The lower end was provided with a thin mylar film to prevent the occurrence of chimney effects. This permitted a rapid quenching of ampoules, suspended by a fine platinum wire, from 1750°C into ice/water without disturbing the atmospheric conditions in the furnace.

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