# Creep Testing of Platinum Alloys

By F. C. Child, M.Sc., A.I.M.

*An account is given of the Johnson Matthey creep-testing laboratory for determining life-to-rupture under constant stress at temperatures from 400° to 900°C, using miniature creep-testing machines*

The creep-testing laboratory was installed primarily for determination of the creep properties of some of the rarer metals and their alloys. It was desirable therefore to use as small a test specimen as possible and, furthermore, since it was considered that life-to-rupture tests would provide a suitable basis for selection of promising alloys, it was decided to employ Denison miniature creep-testing machines, as these take specimens having a diameter of 0.1785 inch over a gauge length of 1 inch.

The installation described is the result of several years' experience of testing at temperatures of 800° to 900°C during which many modifications were made.

A small room, the temperature of which is thermostatically controlled, houses the eight creep-testing machines which comprise the present installation, the whole of the electrical gear being mounted in the single unit shown in Fig. 1. In the upper half of the control unit are the furnace control panels, time indicating clocks and a 6-inch

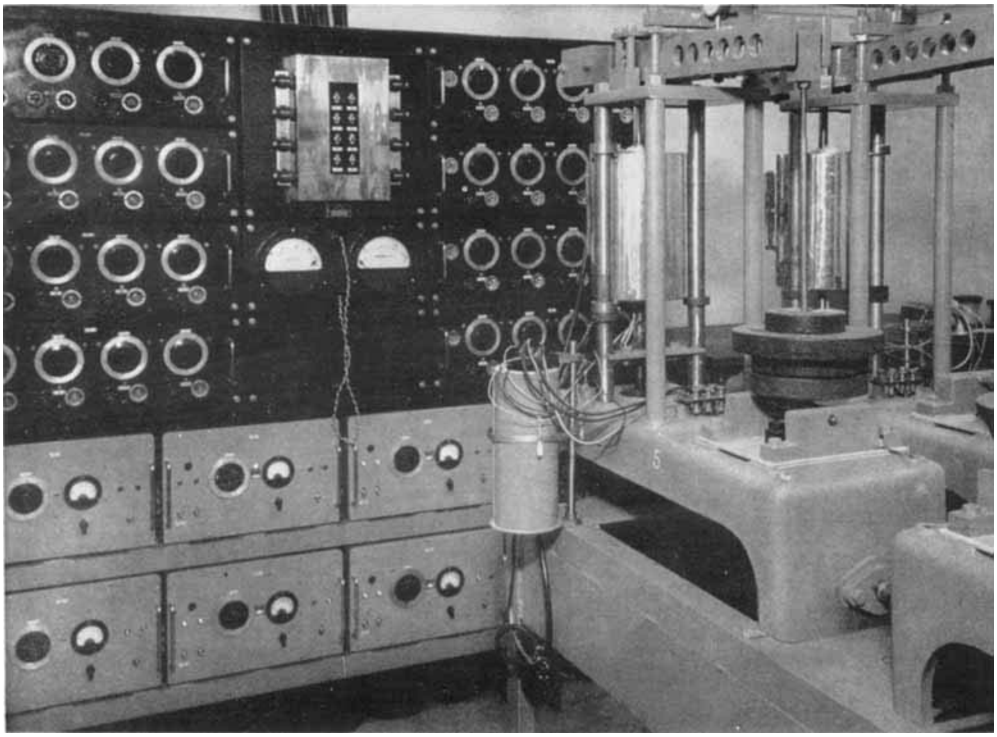

*Fig. 1—The central control panel in the creep-testing laboratory*

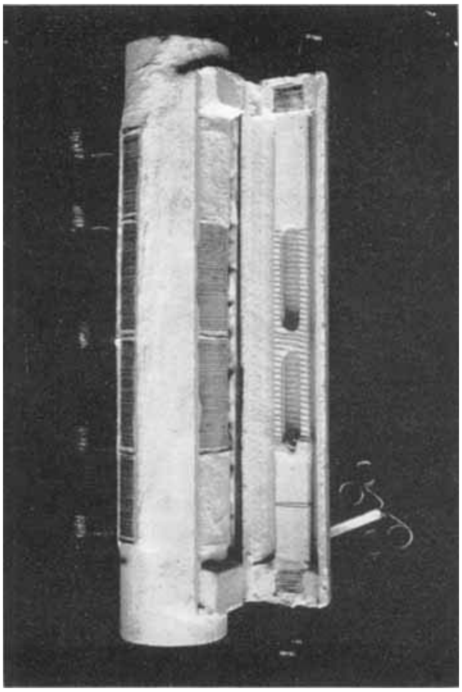

*Fig. 2—A furnace unit showing the rhodium-platinum winding and the platinum resistance thermometer*

scale voltmeter and ammeter; the lower half accommodates the temperature controllers. The whole assembly is constructed on the unit principle to allow for rapid removal for servicing.

Power is supplied from a Breco constant voltage transformer capable of maintaining a voltage of 240 volts ±1 per cent for mains voltage fluctuations of 180 to 260 volts.

## Furnace Design

The furnaces are wound on tubes approximately 8½ inches long×1¼ inch bore fabricated from alumina cement. The winding of 10 per cent rhodium-platinum wire, 0.01 inch diameter, is made uniformly at 32 turns/inch, but is divided into four sections, two end sections each of 36 turns, and two centre windings of 48 turns. The division of the centre winding is necessary to avoid having high and low voltages side by side. Small alumina pads approximately ½ inch from either end of the winding support the resistance thermometer frame about ⅛ inch from the winding.

The resistance thermometer consists of 4 feet of 0.008 inch diameter Thermopure platinum wire wound non-inductively on an alumina cement frame 7½ inches long× ½ inch wide×⅛ inch thick. Slots are made in the frame over the winding length to expose the greatest possible area of wire to direct radiation from the furnace winding. The resistance thermometer winding is wound to cover the two centre furnace windings—i.e. that portion of the furnace in which the specimen is situated. A thin layer of alumina cement along the edges of the thermometer frame secures the winding. Thermopure platinum leads, 0.02 inch diameter, are welded to the ends of the winding, and compensating leads of the same material are run to the junction of the winding and lead. A spiral of platinum wire at each end of the thermometer frame acts as an earth shield to prevent leakage of voltage from the furnace winding.

The furnace winding is covered with alumina cement carefully worked between the windings, a necessity if volatilisation of the winding is to be reduced to a minimum, except for the centre portion immediately facing the thermometer. The winding over this part is filled, but not covered, with cement to allow of maximum radiation. Finally an alumina cover to the resistance thermometer is cemented in place.

Fig. 2 shows the furnace unit with the thermometer and its cover opened from the winding; a strip of cement has also been omitted from the furnace winding.

The completed unit is mounted in a polished aluminium case approximately 9 inches long×6¼ inches diameter with recessed Sindanyo end plates to position the tubes. Kieselguhr is used as the insulating packing.

A power consumption of approximately 375 watts is required to maintain the specimens at 900°C.

The power inputs to each of the three sections of the furnace winding are individually controlled by three Variac transformers, these being supplied in parallel by a fourth Variac which therefore provides the means of adjusting the power to the furnace without alteration to the proportioning of power between the three zones. A resistance in the primary of the main Variac and shorted by the temperature controller provides the means of temperature control. From 10-20 per cent of the power input is switched for control purposes.

Three-pin sockets and swirches are provided to enable the voltage and current output of each Variac to be determined when necessary.

## Temperature Control

The temperature controllers are slight modifications of the original Prosser design which has been fully described elsewhere (1). The fine adjustment potentiometer in the controller was designed to overcome the variations in contact resistance found in the original proprietary model. It consists of a silver-palladium wire spiral wound on a grooved Tufnol drum. The ends of the spiral are connected to copper cylinders, clad with a gold alloy, mounted concentrically with the Tufnol drum, the whole assembly being mounted in bearings which provide for an adjustable friction grip. Three gold alloy contacts connect with the two drum contacts and with the wire respectively. The contact sliding on the wire is moved along the drum by means of a screw, gear-driven from the drum. This maintains the contact in step with the turns of wire and " jumping turns " is impossible.

The contacts and wire of the instrument, being immune to the formation of tarnish films, do not give rise to contact resistance troubles, and their wiping action obviates troubles from dust, etc., while the direct rotation of the drum ensures freedom from " backlash ".

The creep machines are the standard Denison miniature machines. The longer furnace which was found advisable for

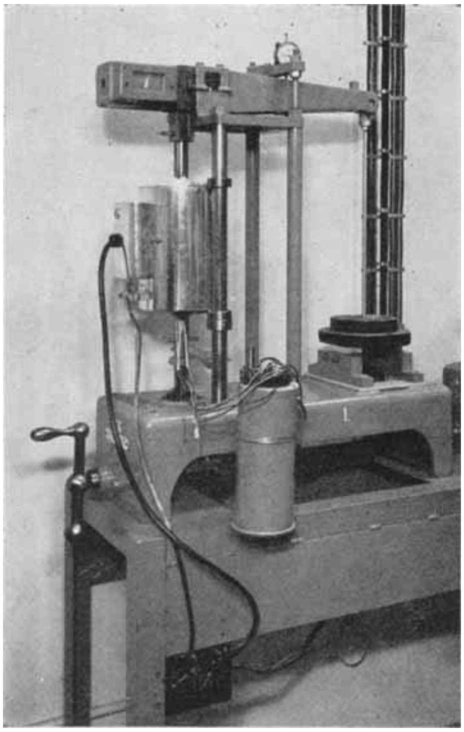

*Fig. 3—A single creep unit with a test in progress*

obtaining uniformity of temperature at 900°C necessitated a minor alteration to the furnace fixings. These are now attached to the machine by two small lugs which bolt on to two loose fitting collars on one pillar thus allowing the furnace to swivel on the pillar. Micro-switches mounted under the weight holder of each machine control the time clocks on the main panel. Fig. 3 shows a single unit with a test in progress.

The specimen temperature is determined by three platinum: rhodium-platinum thermocouples secured to the top, centre and bottom of the test piece and insulated by twin-bore alumina tubing. Originally asbestos pads were used to shield the thermocouples from direct radiation, but experience showed that contamination was occurring and this practice has now been abandoned.

Ice flasks attached to each machine contain the cold junctions of the thermo-

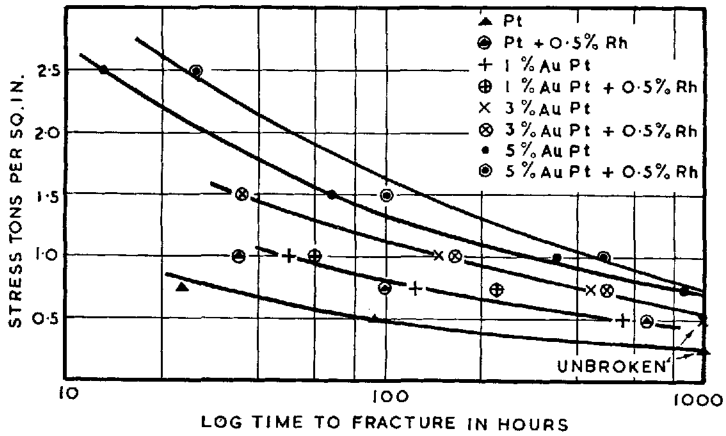


*Fig. 4—Stress against log time curves for gold - platinum and gold-rhodium-platinum alloys*

couples, and flex leads are taken from the cold junctions to a 25 point Post Office type selector switch. This switch is remote controlled from the potentiometer with which all temperature measurements are made.

## Experimental Results

The apparatus had been found capable of giving satisfactory results over the temperature range of 400° to 1000°C. At 900°C the gradient in the specimen can be maintained at better than 1°C while temperatures can be controlled to ±1°C over periods of up to 1,000 hours. Since the limits of accuracy of the thermocouples in such circumstances are probably of the same order, attempts at closer control than this would be pointless.

During its development, and for some considerable time afterwards, the creep laboratory was fully occupied with work on rare metals other than platinum and its alloys, and only recently has the opportunity arisen for some work on these. The only complete investigation to date is on the effect of 0.5 per cent rhodium on the time to fracture of pure platinum, and platinum containing 1, 3 and 5 per cent gold at 900°C.

The results, shown graphically in Fig. 4, show that:

(*a*) the addition of 0.5 per cent rhodium to platinum increases the life at a given stress by four or five times, without affecting the ductility

(*b*) additions of up to 5 per cent gold to platinum progressively increase the creep endurance but failure of the alloys is accompanied by intercrystalline cracking

(*c*) the addition of 0.5 per cent rhodium to the gold platinum alloys increases the creep resistance slightly, but does not reduce the tendency to intercrystalline cracking

Little attention has been given so far to the creep characteristics of the platinum metals and their alloys, but with their increasing use in chemical engineering at elevated temperatures the need for such data is growing.

## Review of Published Data

A review of the available information together with some original work has been made by Dr. G. Reinacher (2).

Two papers have been published on long time creep of the platinum metals and their alloys (3, 4) and a third (5) includes some work on compression creep. Four other papers (6, 7, 8, 9) deal mainly with short time tests, the results of which are of little use to the design engineer.

A summary is given below of the papers on long time creep.

Atkinson and Furman (3) determined the creep properties of 99.95 per cent platinum, high purity 10 per cent rhodium-platinum,

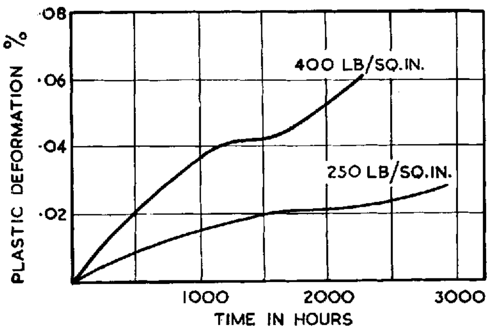


*Fig. 5—Platinum creep curves at 750°C (Atkinson and Furman)*

and two batches of palladium deoxidised respectively with calcium boride and aluminium. The tests were made at 750°C on specimens of 4 inch gauge length 0.29 inch diameter, and the creep curves are shown in Figs. 5, 6 and 7.

Two platinum specimens tested at 250 lb./sq. in. after annealing at 750°C for 17 hours gave practically identical creep rates of 8 to $9 \times 10^{-6}$ per cent per hour at 2,000 hours, while a third tested at 400 lb./sq. in. gave a creep rate of $26 \times 10^{-6}$ per cent per hour.

A 10 per cent rhodium-platinum specimen annealed at 750°C and tested at 400 lb./sq. in. showed approximately 50 per cent higher creep rate than platinum, the grain size being almost half that of the platinum specimens. Further specimens annealed for one hour at 1050°C had grain sizes similar to those of the platinum bars with better creep resistance.

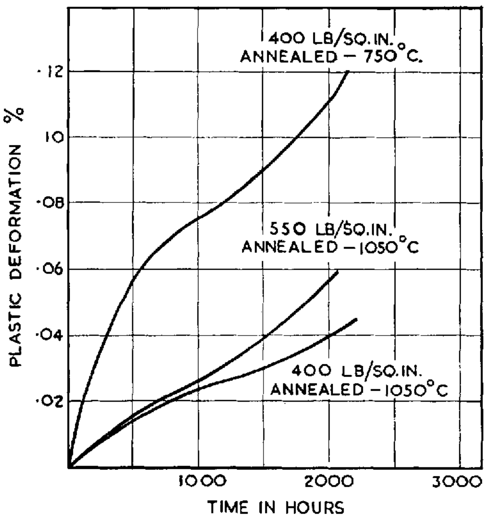


*Fig. 6—Creep curves for 10% rhodium-platinum at 750°C (Atkinson and Furman)*

Palladium bars, whether deoxidised with calcium boride or with aluminium, had high first stages of creep, but after 1,200 hours the rates decreased. Pretreatment for 1,250 hours at 750°C eliminated the high first stage possibly because of internal oxidation.

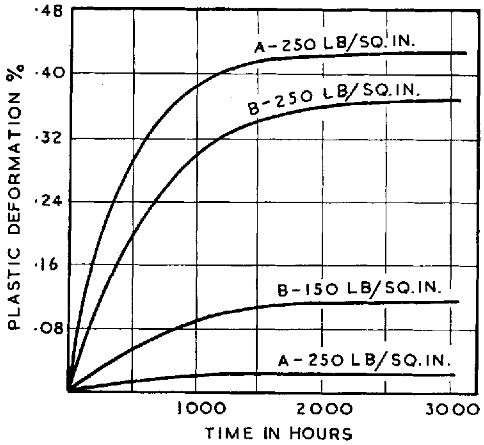


*Fig. 7—Palladium creep curves at 750°C (Atkinson and Furman)*

Stauss (4) has examined the behaviour of 99.99 per cent platinum, 10 per cent iridium-platinum and 10 per cent rhodium-platinum alloys, the tests being made at 1100°C on wires 0.010 inch diameter previously annealed at 1200°C. The results, plotted in Fig. 8 as stress/log time to fracture curves, show that the stress for a life of 3,000 hours is roughly 420 lb./sq. in. and 700 lb./sq. in. for platinum and for 10 per cent iridium-platinum respectively.

In Fig. 9 the results are plotted as log stress against log time to fracture. The pure platinum curve shows a sharp discontinuity at 5 hours which coincided with a change in the type of fracture of the wire from the normal plastic-necked type to the knife-edge

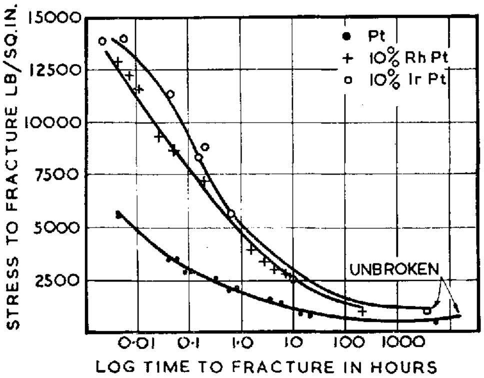


*Fig. 8—Stress against log time to fracture for platinum, 10% rhodium-platinum and 10% iridium-platinum (Stauss)*

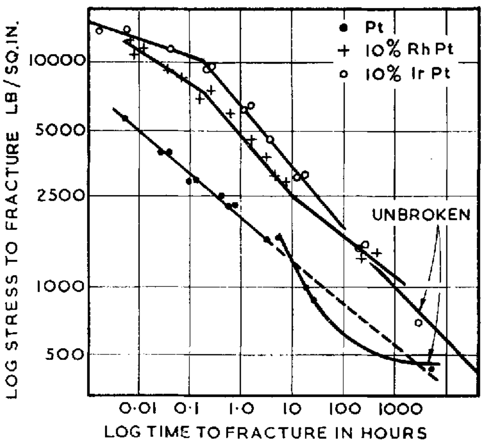


*Fig. 9—Log stress against log time to fracture for platinum, 10% rhodium-platinum and 10% iridium-platinum (Stauss)*

type associated with single crystals. Metallographic examination verified that grain growth had resulted in single crystals across the diameter of the wire.

The 10 per cent iridium-platinum curve shows two breaks. The first, after 8 minutes, accompanied by a change of fracture from the plastic to the brittle intercrystalline type, is ascribed to oxidation, and the second upward inflection at about 170 hours is interpreted as the result of increasing difficulty in the penetration of oxidation. The curve for 10 per cent rhodium-platinum shows similar breaks for probably similar reasons. Neither of these alloys showed grain growth and hence the break present in the platinum curve due to this cause is absent.

The results verify the practical experience that the 10 per cent rhodium-platinum alloy is more satisfactory than the 10 per cent iridium-platinum alloy for prolonged use at elevated temperatures under oxidising conditions. The continuous stress at 1100°C on these alloys must not exceed 1.5 per cent of the normal room temperature tensile strength or 6 per cent of the strength at 1100°C.

## References


1 L. E. Prosser ... ... ... Alternating Current Bridge Thermostat for Creep Testing, *Engineering*, 1939, **148**, 95-96.

2 G. Reinacher ... ... ... The Creep Properties of the Noble Metals, *Metall* 1956, **10**, 13/14, 597-607.

3 R. H. Atkinson and D. E. Furman — Creep Characteristics of some Platinum Metals at 1382° F, *J. Metals*, 1951, **3**, 806-808.

4 H. E. Stauss ... ... ... Time-to-Fracture Tests on Platinum, 10 per cent Iridium-platinum and 10 per cent Rhodium-platinum Alloys, *Trans. A.I.M.E.*, 1943, **152**, 286-290.

5 N. P. Allen and W. E. Carrington — Exploratory Creep Tests on Metals of High Melting Point, *J. Inst. Met.*, 1953-4, **82**, 525-533.

6 R. P. Carreker, Junr. ... ... Plastic Flow of Platinum Wires, *J. Appl. Phys.*, 1950, **21**, 1289-1296.

7 S. Dushman, L. W. Dunbar and H. Huthsteiner. — Creep of Metals, *J. Appl. Phys.*, 1944, **15**, 108-124.

8 R. B. Middleton, L. B. Pfeil and E. C. Rhodes — Pure Platinum, of High Recrystallisation Temperature, Produced by Powder Metallurgy, *J. Inst. Met.*, 1948-49, **75**, 595-608.

9 E. M. Wise and J. I. Eash ... Properties of the Platinum Metals II, *Trans. A.I.M.E.*, 1938, **128**, 282-294.