



## High Temperature Mechanical Properties of the Platinum Group Metals

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W. C. Heraeus

## Preface

To celebrate Heraeus' 150 years in the forefront of work on the Platinum Group Metals we are pleased to present two recent papers that have been published in Platinum Metals Review. From the earliest days of our company's history we have been developing materials for applications at the highest temperatures. These papers are intended as a contribution to demonstrate that we are maintaining our leading technological role in the research and further development of these essential materials.

Our association with the city of Jena also dates back to the years of our company's founder, Wilhelm Carl Heraeus. Otto Schott, the 19<sup>th</sup> century pioneer of modern optical glassmaking, used platinum crucibles from Heraeus for his trials and production. It is, therefore, a special privilege for us to have been able to re-establish links with this technological centre in recent years and to collaborate with the two prestigious universities in Jena, the Friedrich Schiller University and the Fachhochschule – University of Applied Sciences.

# Stress-Rupture Strength and Creep Behaviour at Extremely High Temperatures

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*There is a constantly increasing need for metallic materials with melting points over 1700°C for use at very high temperatures. In contrast to the refractory metals: tantalum, niobium, tungsten, molybdenum and rhenium, which also have very high melting points, the metals of the platinum group, particularly platinum, rhodium and iridium, are characterised by outstanding chemical stability, oxidation resistance and resistance to many molten oxides. The platinum group metals are therefore ideal materials for using at high temperatures while undergoing simultaneous chemical attack and mechanical loading. However, for optimum effective employment of these metals, it is necessary to know their strength and deformation behaviour at extremely high temperatures. Data have therefore been collected from comparative investigations of platinum, platinum alloys, dispersion hardened platinum materials, rhodium and iridium, and the compilations are presented here.*

The metals of the platinum group are essential high temperature materials for the development of high technology products, used, for example, in small rocket engines and other areas of space technology, and in the manufacture of high quality glass and glass fibres. Indeed, many high temperature problems in space technology can be overcome by the use of platinum group metals. The nozzles of rockets which carry satellites from the launch vehicle to their service orbit or are used for positional corrections may be manufactured from platinum alloys. Capsules for radioactive power sources which provide space probes with electrical energy are made from iridium or platinum alloys (1, 2). In the glass industry, platinum and platinum alloys are used in glass melting equipment, such as in crucibles, tank linings, stirrers, spout bowls, plungers, orifice rings and thermocouple thimbles (3) as well as in bushings for glass fibre production (4). One indispensable application of platinum alloys at elevated temperatures is in catalyst gauzes for the manufacture of fertilisers, cyanides and plastics (5).

Data on the mechanical properties of the platinum group metals at high temperatures are essential for their use in the above areas of application (6). Data are required to ensure that constant material properties are achieved by the production, in order to develop alloys with improved properties, and for the selection of appropriate materials and the optimum design of components.

Because of the creep processes which start to operate at high temperatures, the most important practical material property is the stress-rupture strength. Measurements were therefore taken at temperatures up to 2300°C. The good stress-rupture properties of the platinum group metals, the strengthening effects of certain alloying elements and the possibility of using dispersion hardening in these materials have long been recognised (7–17). However, few investigations of these metals at temperatures over 1500°C could be found in the literature. Platinum is currently used in the glass industry at temperatures up to about 1600°C. To achieve even better quality in glass and to develop new special grades of glass, components made of platinum or platinum alloys are required which can

operate at even higher temperatures. For temperatures at which platinum with its melting point of 1769°C can no longer be used, rhodium (melting point 1963°C) and iridium (melting point 2446°C) have to be employed, for example, in crucibles for pulling laser crystals from oxide melts and in components for space technology. Until now, these components had to be designed without knowledge of the high temperature mechanical properties of their constituent materials.

## Experimental Techniques

In the stress-rupture test, a load is applied to a sample of the material and the time to rupture of the sample is determined. The values of nominal stress are plotted against the time to rupture on a double-logarithmic scale to obtain the stress-rupture curve; this normally approximates to a straight line. As equipment for stress-rupture testing at extremely high temperatures was not commercially available, a special facility for testing high-melting metals was developed and built where:

- the temperature range of operation was 1000°C to 3000°C;
- operation could take place under a protective gas environment if required, for example for iridium;
- creep curves could be determined and
- direct microscopic observations of the crystalline structure of the samples during the stress-rupture test were possible (18, 19).

Using this test facility, comparative investigations were conducted on various platinum, rhodium and iridium materials. The samples of platinum materials for the tests were in the form of strips, 0.5 x 3.0 x 120 mm, supplied from normal production of sheet from Heraeus. For tests on rhodium and iridium the materials were electron-beam melted, then rolled and cut into wire samples of size 1.0 x 1.0 x 120 mm. Comprehensive metallographic and scanning electron microscope (SEM) investigations of the microstructure, the surface structure and the fracture faces were carried out on the samples after testing, to obtain a better understanding of their behaviour during the test. The results of these investigations are summarised here with some examples.

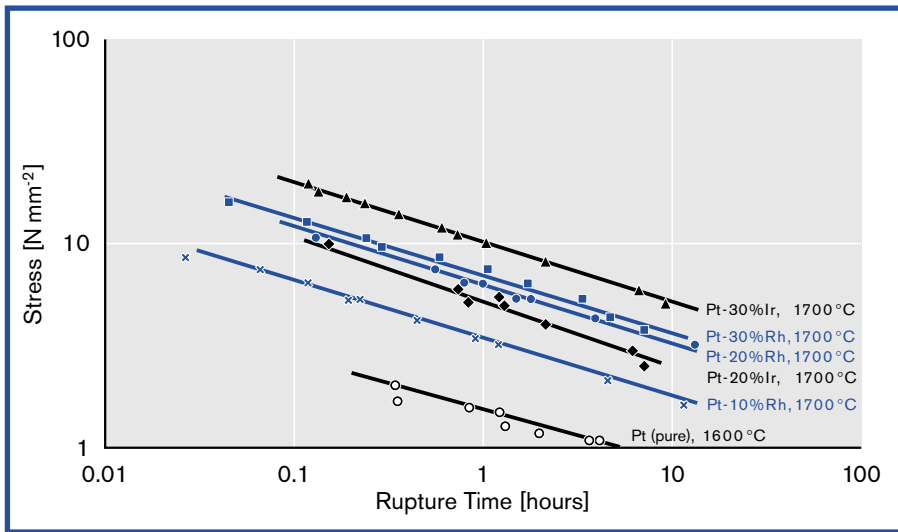


Fig. 1 Stress-rupture curves of platinum alloys at 1700°C and pure platinum at 1600°C

**High Temperature Properties of Platinum and Platinum Alloys**

Stress-rupture curves at 1700°C for platinum alloys containing 10 to 30 per cent of rhodium and iridium and at 1600°C for pure platinum are shown in Figure 1 (20). The stress-rupture curve of pure platinum was included for comparison, because at 1700°C platinum can neither be tested in the stress-rupture apparatus nor used as a structural material. The individual values obtained showed an excellent degree of reproducibility and lie almost ideally on straight lines in the stress-rupture diagrams. By interpolation from the stress-rupture curve, it is possible to define the stress-rupture strength for a particular time, for example, for 10 hours, to facilitate comparison of different materials. In Figure 1 it can be seen that solid-solution strengthening of platinum leads to an enormous increase in the stress-rupture strength. As would be expected, the effect increases with increasing amounts of the alloying addition. The alloys investigated have a higher stress-rupture strength at 1700°C than pure platinum at 1600°C.

However, alloys with alloying additions of more than about 30 per cent are not generally used as structural materials because of problems that arise in their processing. Values for the stress-rupture strengths of alloys with 20 per cent rhodium and 20 per cent iridium do not differ very greatly, although the platinum-rhodium (Pt-Rh) alloy has a slight strength advantage. Increasing the alloying addition

by a further 10 per cent leads to only a moderate increase in stress-rupture strength for the alloy platinum-30 per cent rhodium (Pt-30%Rh), whereas the platinum-30 per cent iridium (Pt-30%Ir) alloy shows a substantial strength increase, see, respectively, Figures 2(a) and 2(b).

At a test temperature of 1700°C, for example, the increase in strength for Pt-20%Rh on addition of a 10 per cent increase in rhodium is only ~ 0.3 N mm<sup>-2</sup> compared with ~ 2.5 N mm<sup>-2</sup> for the Pt-Ir alloys, that is, the strength of these alloys is doubled. Similar relationships have been found in studies of high temperature elastic properties of these alloys (21).

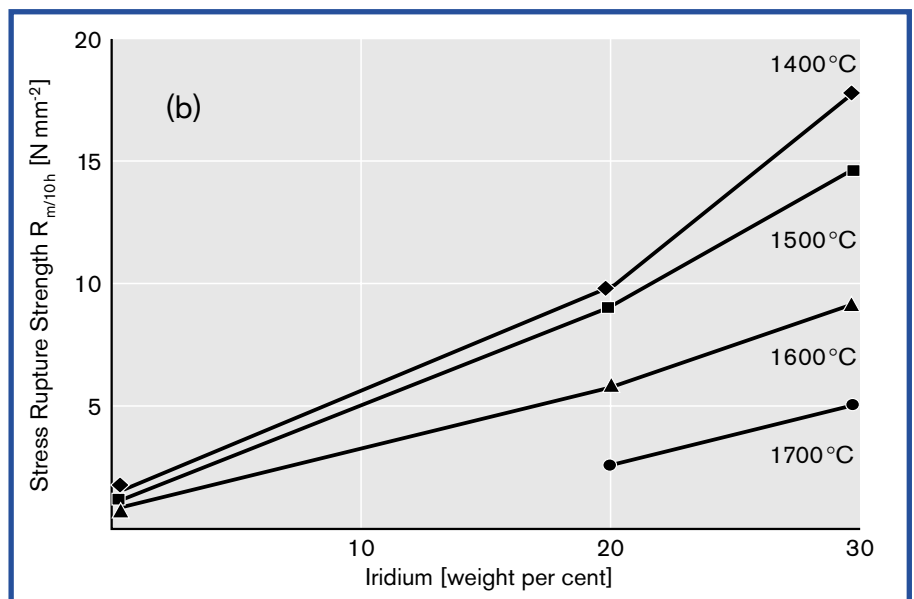
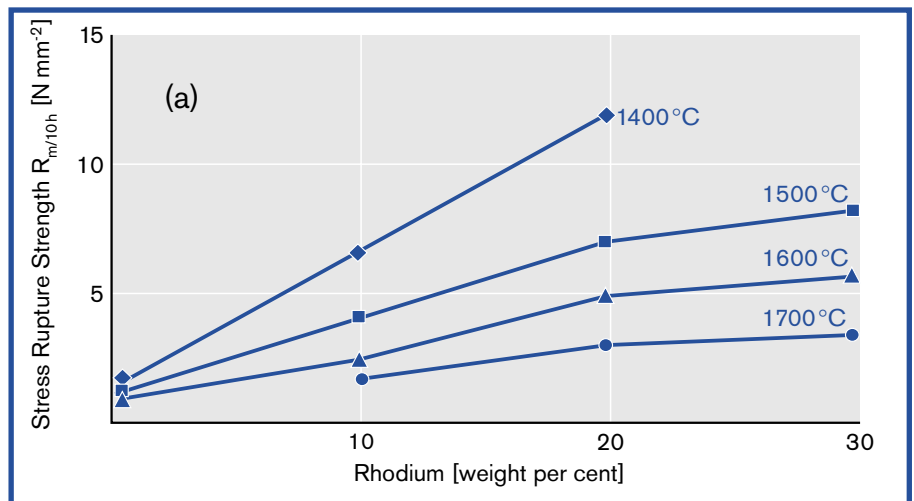


Fig. 2 Stress-rupture strengths of platinum alloys as a function of the alloying content at various temperatures: (a) 10-hour stress-rupture strength versus rhodium content (b) 10-hour stress-rupture strength versus iridium content

## Stress-Rupture Strength and Creep Behaviour at Extremely High Temperatures

In practice alloys with more than 20 per cent rhodium are rarely used because the increase in strength does not compensate for increased problems in fabrication. Care must also be taken in using Pt-Ir alloys with high iridium contents because they can become embrittled in service at intermediate temperatures due to the precipitation of primary iridium in the platinum matrix.

Figure 3 shows the 10-hour stress-rupture strengths as a function of temperature for pure platinum and the alloys over the temperature range 1400 to 1700°C. At all temperatures the Pt-30%Ir alloy has the highest strength. The stress-rupture strength of pure platinum even at 1400°C is lower than that of any of the alloys investigated at 1700°C. For all the alloys the relative decrease in stress-rupture strength resulting from a temperature increase from 1400 to 1700°C is very similar. However, the strength of pure platinum, although low at all temperatures, decreases more slowly as the temperature is increased.

Using alloys at high temperatures under conditions of mechanical loading leads to a change in their dimensions by creep, that is by diffusion-controlled deformation processes that are time and rate dependent. Figure 4 shows creep curves of the platinum alloys investigated. The stress was selected for each sample so that a stress-rupture life of approximately 10 hours was achieved. The change in the overall length of the samples was measured as a function of the testing time. The elongation values therefore relate to the nominal gauge length of 100 mm.

However, because samples were direct resistance heated, only the central part of each gauge length reaches the test temperature and it is cooler near the grips. The true elongation in the central portion of the sample is therefore considerably greater than the values shown. In the secondary, steady-state range of creep, all alloys show similar creep rates in the range 2 to 5  $\mu\text{m s}^{-1}$ .

The Pt-Ir alloys give higher values for rupture elongation than the Pt-Rh alloys. The Pt-30%Ir alloy, which has the highest stress-rupture strength, also shows the greatest elongation to fracture. It also demonstrates excellent ductility at very high temperatures, despite the very high

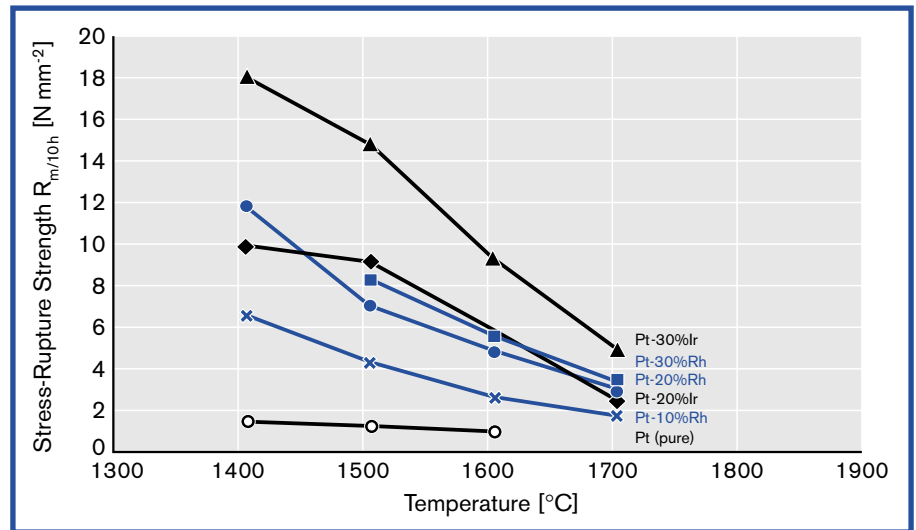


Fig. 3 10-hour stress-rupture strengths of pure platinum and platinum alloys at various temperatures

level of solid solution strengthening and the tendency of this alloy to embrittlement at lower temperatures. The high fracture elongation of the Pt-30%Ir samples in the stress-rupture tests was accompanied by a very considerable degree of necking. After short test times slip bands could be seen on the surface of the material in the zone close to the fracture, some of which can be seen in Figure 5(a).

At longer test times, intergranular cracks, creep pores and superficial structures resulting from the selective evaporation of iridium were observed, see Figure 5(b). A certain amount of loss of iridium by evaporation in the form of volatile oxides had no measurable influence on the high temperature mechanical properties at the test durations studied.

### High Temperature Properties of Dispersion Hardened Platinum Materials

In a number of applications solid solution strengthening is insufficient, for example, when maximum mechanical stability is required at very high temperatures. The use of solid solution additions may also be limited for other reasons, such as, they have insufficient oxidation resistance or there is embrittlement of the alloy after exposure. In these cases the dispersion hardening of platinum materials can be used to its full advantage.

One example of this is the melting and processing equipment used for the manufacture of quality high grade optical glass, when it is not possible to use the Pt-Rh

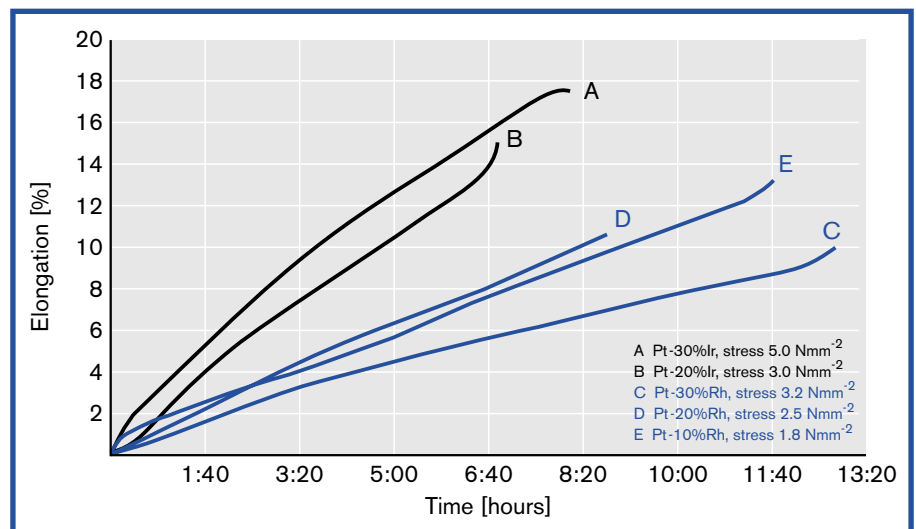


Fig. 4 Creep curves of platinum alloys at test temperature of 1700°C. The rupture time is approximately 10 hours

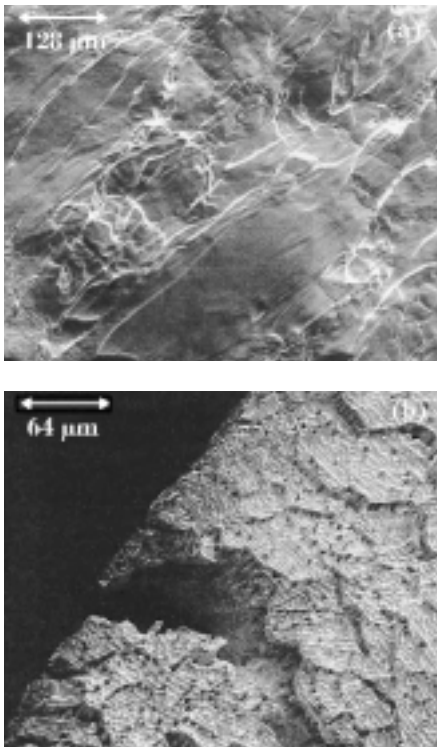


Fig. 5 Scanning electron micrographs of the surface near the fracture zone of Pt-30%Ir samples after the stress-rupture test:

- (a) rupture time approximately 1 hour  
 (b) rupture time approximately 10 hours

and Pt-Ir alloys discussed above. If trace quantities of the alloying elements dissolved in the glass melts it would be severely detrimental to the optical properties of the glass, causing discoloration of the glass and a reduction in the optical transmission. Less noble alloying elements cannot be considered for the solid solution strengthening of platinum in these applications because they would be detrimental to the corrosion resistance of platinum in the molten glass and the corrosion products would lead to even greater contamination.

Platinum used for melting equipment for optical glass must therefore be strengthened by other means. A strengthening effect can be achieved by the introduction of finely dispersed stable particles into the platinum matrix; this is known for other materials, such as the alloys of nickel, copper and aluminium. In order to ensure that the dispersion hardening is effective up to the highest possible temperatures, the dispersed phase must have a very high thermodynamic stability, a higher melting temperature than the matrix and must be insoluble in the matrix even at the highest temperatures. For this reason

oxides are preferable to carbides and silicides. Furthermore, oxygen is harmless to platinum, in contrast to both carbon and silicon. Oxides of zirconium and yttrium have proved particularly effective for the oxide dispersion hardening of platinum. Provided that the oxide particles are very small ( $< 1 \mu\text{m}$ ) and the inter-particle spacing is also small ( $< 10 \mu\text{m}$ ), the dispersion of particles will hinder the movement of dislocations in the matrix. During cold-working of the semi-products, high dislocation densities are created. These lead to a significant hardening of the material. The presence of particles at grain boundaries also hinders the movement of the grain boundaries at high temperatures and thus restricts grain coarsening. In this way, it is possible to achieve a stable fine-grained microstructure in the platinum which is maintained during long-term service at high temperatures.

The use of dispersion hardening is not limited only to pure platinum. It is also possible to increase the strength of solid-solution strengthened platinum alloys further, especially at very high temperatures. The development of dispersion hardened platinum and platinum alloys has permitted a substantial increase in the range of possible applications of platinum materials at high temperatures.

During the present work, platinum materials which had been dispersion hardened with zirconium-yttrium oxide particles were investigated (22). These materials are designated DPH here. High temperature properties were determined on dispersion hardened platinum (Pt DPH) and on the dispersion hardened alloys Pt-10%Rh DPH and Pt-5%Au DPH. The materials Pt DPH and Pt-10% Rh DPH are already widely used in the glass industry and many other fields where long term reliability is essential. They can be readily processed, and their good forming properties and excellent weldability, in particular, are important features in the manufacture of equipment and components. The micrograph in Figure 6(a) shows finely distributed particles both in the matrix and at the grain boundaries of Pt-10%Rh DPH. The lower-magnification micrograph in Fig. 6(b) shows elongated grains resulting from cold rolling of the sheet. After the stress-rupture test the microstructure has recrystallised, see Figure 6(c). However, despite the high test temperature of

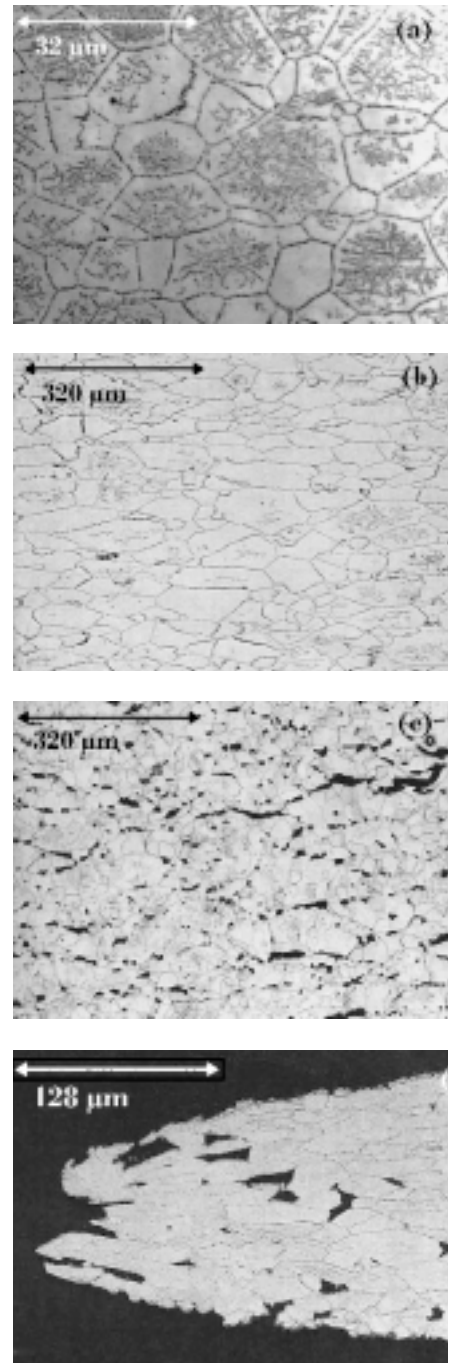


Fig. 6 Microstructure of Pt-10%Rh DPH:

- (a) initial state  
 (b) initial state after cold rolling  
 (c) after stress-rupture test at  $1600^{\circ}\text{C}$ ;  
 tested for 8.87 hours; some way from fracture  
 (d) after stress-rupture test at  $1600^{\circ}\text{C}$ ;  
 tested for 8.87 hours; fracture with deformation

$1600^{\circ}\text{C}$  it has remained very fine grained. The severe deformation of the material, which largely occurs at high temperatures by grain boundary sliding, has resulted in numerous creep pores at the grain boundaries. This microstructure is much more favourable than that of the conven-

## Stress-Rupture Strength and Creep Behaviour at Extremely High Temperatures

tional alloy Pt-10%Rh in which similar conditions lead to a coarse microstructure with fewer, much larger pores at the grain boundaries. The dispersion hardened material has necked down substantially during the stress-rupture test, see Figure 6(d), which shows its good ductility. The severe deformation in the necked zone has led to a marked elongation of the grains. It is possible to control the stress-rupture strength of the dispersion hardened platinum materials by adjusting the content of the dispersed phase and modifying the production process. As would be expected, maximum strength is associated with a reduced level of ductility.

However, our experience with customers has shown that for most applications

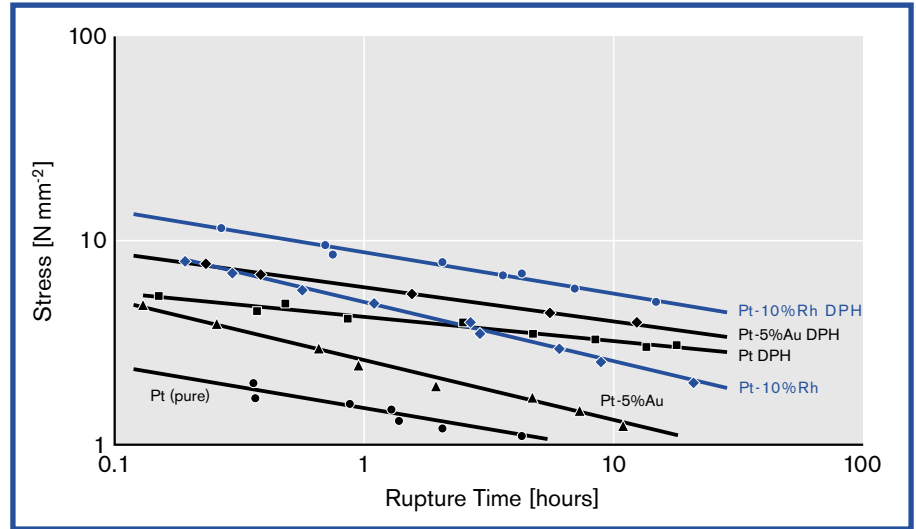


Fig. 7 Stress-rupture curves of platinum materials at 1600°C

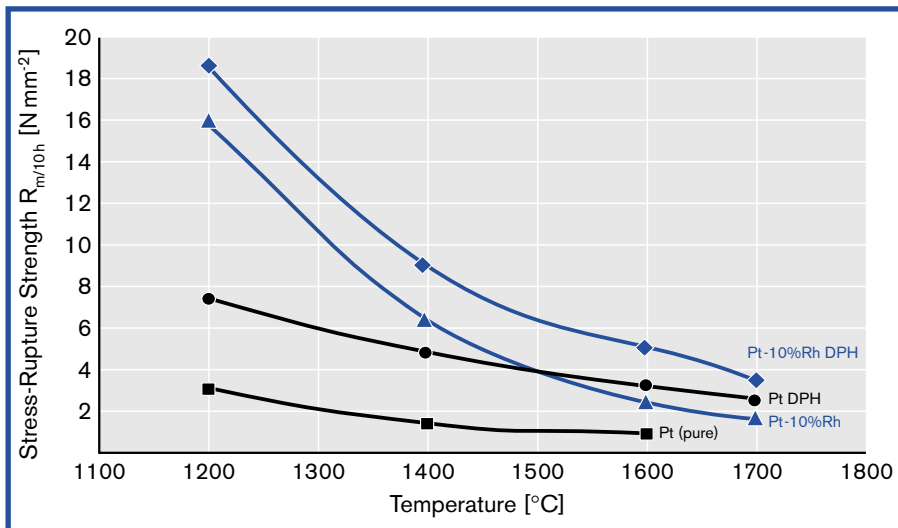


Fig. 8 10-hour stress-rupture strengths of platinum alloys at various temperatures

material with maximum ductility is preferred to material with the highest level of strength. The stress-rupture curves in Figure 7 and the following diagrams refer to DPH materials which are optimised with regard to their ductility.

It is particularly interesting that all DPH materials have significantly greater stress-rupture strengths than the three conventional alloys. At the longer test times Pt DPH has a higher stress-rupture strength than the Pt-10%Rh alloy. It is significant that the stress-rupture curves of the dispersion hardened materials show a more gradual decrease in strength with increasing rupture time than those of the conventional materials. This means that the dispersion hardened materials show

an even greater strength advantage when they are used for components with long service lives. The relatively short test times, which are dictated by the available testing capacity, are completely adequate to compare different materials. However, the design of components for use, for example, in the glass industry requires values determined over longer test times and such tests are currently in progress.

The main application for the Pt-5%Au DPH alloy is in the production of crucibles, dishes and other items for the analytical laboratory, where its stable fine-grained structure is of particular advantage along with its well known anti-wetting characteristics. This material has not yet achieved significant use in structural components

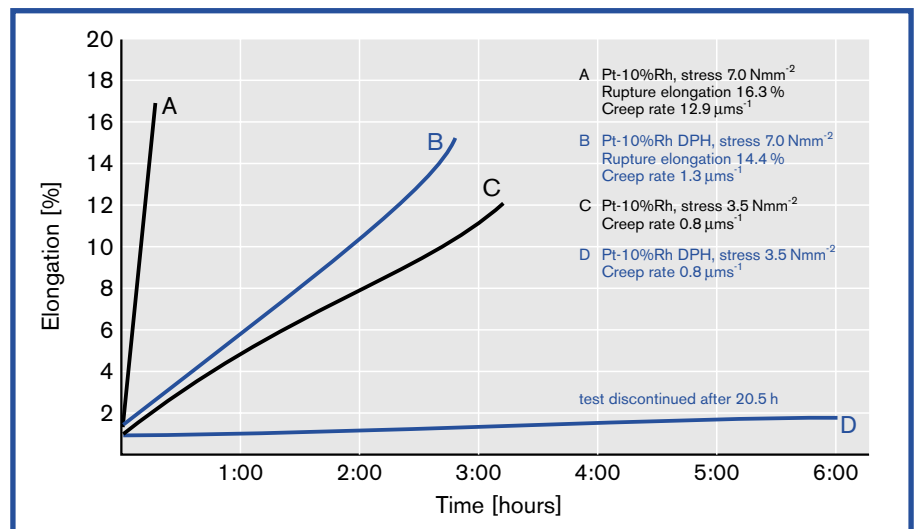


Fig. 9 Creep curves of Pt-10%Rh and of dispersion hardened Pt-10%Rh DPH at 1600°C

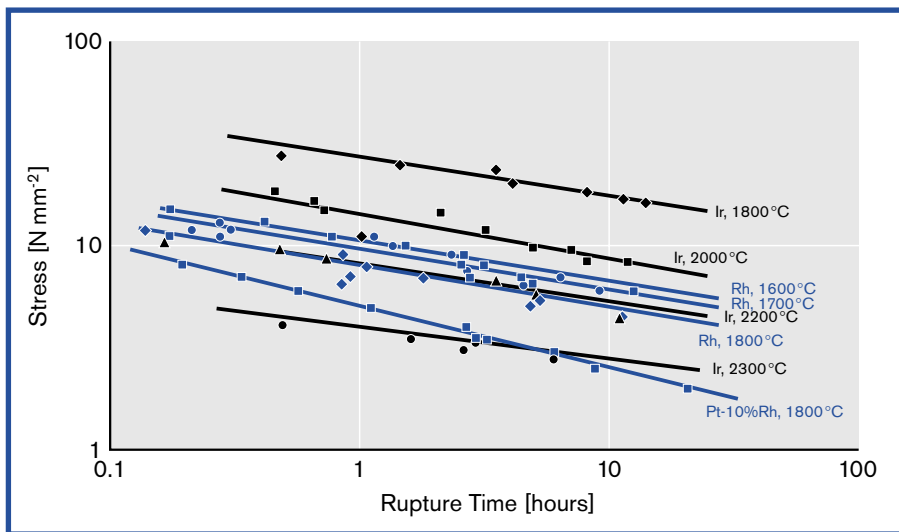


Fig. 10 Stress-rupture curves of rhodium and iridium at various temperatures compared with Pt-10%Rh at 1600°C

for glass processing. However, as can be seen in Figure 7, the alloy also has interesting high temperature mechanical properties and its strength exceeds that of conventional Pt-10%Rh.

In Figure 8 is shown the 10-hour stress-rupture strength as a function of temperature for the dispersion hardened materials Pt DPH and Pt-10%Rh DPH in comparison to the conventional materials. The advantage of the dispersion hardened materials, especially at the highest temperatures, is quite clear. The temperature limit for the use of platinum materials, for example in the glass industry, is currently ~1600°C. However, Figure 8 shows that Pt-10%Rh DPH has a higher level of strength than the conventional alloy even at considerably higher temperatures.

Examples of creep curves for Pt-10%Rh DPH, compared to the conventional alloy, are shown in Figure 9 for a test temperature of 1600°C and two test stresses. The higher strength of the dispersion hardened material produces a much longer time to rupture at the same stress and a much lower creep rate in the linear range of secondary creep. For example, the creep rate of Pt-10%Rh at 1600°C and a test stress of 7.0 N mm<sup>-2</sup> is reduced by a factor of ten (from 12.9 μm s<sup>-1</sup> to 1.3 μm s<sup>-1</sup>) by dispersion hardening. Structural components which are manufactured from the dispersion hardened material therefore show greater stiffness and resistance to deformation, so it is therefore possible to increase their service

life. In addition to the strength increase resulting from dispersion hardening it is also important to consider the ductility of the materials. It can be seen in Figure 9, for example, that the fracture elongation of Pt-10%Rh has been little reduced by being dispersion hardened (from 16.3 to 14.4% at 1600°C and 7.0 N mm<sup>-2</sup>). This means that it is not necessary to sacrifice ductility to obtain the strengthening effect of dispersion hardening.

#### High Temperature Properties of Rhodium and Iridium

In Figure 10 are shown stress-rupture curves for pure rhodium (1600 to 1800°C) and pure iridium (1800 to 2300°C) (23). The measurements on iridium were carried out under a protective atmosphere of argon to prevent the evaporation of volatile oxides which would lead to a reduction in cross-section. For comparison, the stress-rupture curve of Pt-10%Rh at 1600°C is included. It can be clearly seen that rhodium and, in particular, iridium still have substantial stress-rupture strengths which can be made use of at the very high temperatures where platinum materials cannot be considered.

The stress-rupture curves for rhodium at temperatures up to 1800°C and for iridium up to 2200°C lie considerably higher than the stress-rupture curve for Pt-10%Rh at 1600°C which is generally considered to be the maximum temperature for its application.

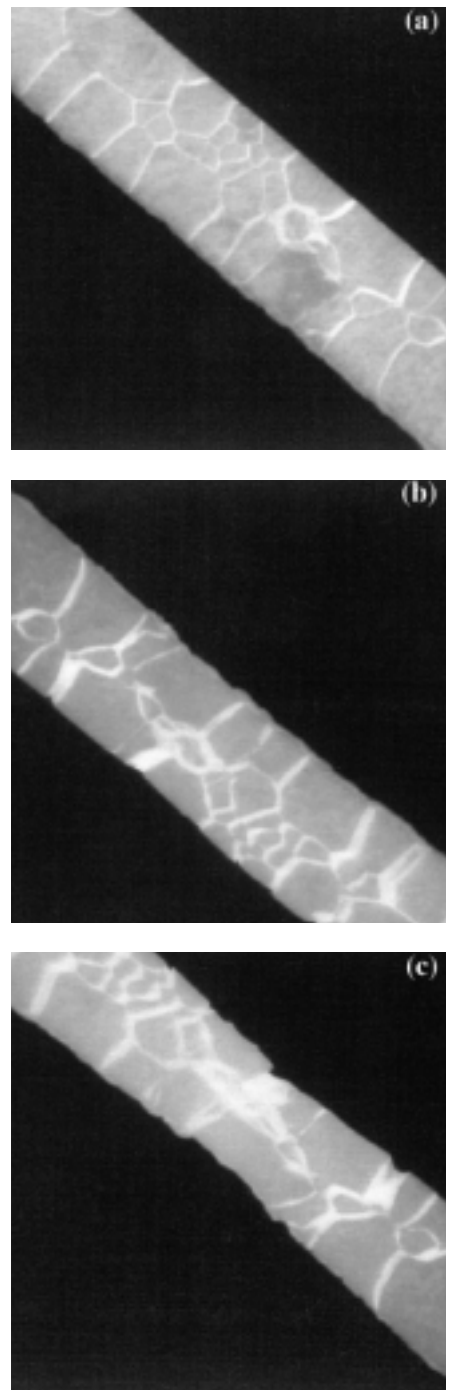


Fig. 11 High temperature images of an iridium sample 1 x 1 mm in cross-section during stress-rupture testing at 2000°C; stress: 19 N mm<sup>-2</sup>: (a) at the start (b) after 0.7 hour (c) after 0.8 hour (10 seconds before rupture)

The photographs in Figure 11 show the surface of an iridium sample after various test times during stress-rupture testing at 2000°C. The grain boundaries are clearly visible and they become broader with increasing test time. Despite the high test temperature the material shows no signifi-

cant grain coarsening. Shortly before rupture the sample shows first indications of necking and the cracking initiates at a broadened grain boundary.

### Further Investigations

Important uses of platinum materials are in complex components which may contain numerous welds. The welds tend to be areas of weakness in the structure, particularly when dispersion hardened materials are used. In many applications, for example, in glass making and processing, components are exposed to significant corroding effects. The properties of platinum materials in the welded state and after exposure to corrosive glass melts are currently being investigated and will be the subject of a subsequent report.

### Acknowledgement

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# Elastic Properties of Platinum, Rhodium and Iridium and their Alloys at High Temperatures

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Platinum group metals (pgms) and in particular platinum alloys are indispensable in many fields of industrial application because of their outstanding physical and chemical properties. Components made from these materials are frequently subjected to extremely complex mechanical loading at high temperatures, often being simultaneously exposed to corrosive attack. A major aspect in the design of components to be used, for example, in the glass industry, in aerospace technology and in single crystal growing is to ensure optimum service life while using the least possible quantity of noble metal. In addition to data on the stress-rupture strength and creep properties (1), the design engineer requires values for the elastic properties of these materials up to very high temperatures.

However, very little data on the temperature dependence of the elastic constants of the platinum metals and their alloys is found in the literature. Apart from the published investigations (2–3), a current monograph gives the elastic properties of platinum alloys at room temperature (4). The elastic moduli of pure pgms as a function of temperature are given in the same publication (4) with reference to work carried out by Reinacher in the 1960s (5–7), and published more recently (8). Comprehensive work on the temperature dependence of the elastic moduli of metals and alloys was published by Köster in the 1940s (9–11). However, in view of the state of technical development at that time, these results can only be regarded as a guide.

## Experimental Procedure

The resonance method used to determine the elastic properties is a non-destructive,

dynamic technique characterised by its high precision. It is applicable to all materials which can be stimulated to mechanical oscillation, see Figure 1. This state-of-the-art process is suitable for determining elastic constants of materials with isotropic, cubic or transverse-isotropic mechanical behaviour in a temperature range from  $-30^{\circ}\text{C}$  to  $1650^{\circ}\text{C}$  (12–14).

In order to derive these properties with a high degree of precision from the characteristic frequencies (of oscillation) on specimens using the resonance method, it is necessary to know the mathematical relationships between these quantities as exactly as possible. The frequency equations derived from the basic theory of oscillating beams, which are commonly used for such evaluations, do not give the required accuracy. The necessary relationships can therefore only be derived on the basis of the known three dimensional Equation of motion from the linear theory of elasticity. Under the condition that the body is ideally elastic, homogeneous and isotropic, we derive for Young's modulus (E) and Poisson's ratio ( $\nu$ ):

$$\rho \frac{\partial^2 \vec{u}}{\partial t^2} = \frac{E}{2(1+\nu)} \left[ \Delta \vec{u} + \frac{1}{1-2\nu} \text{grad div } \vec{u} \right]$$

where  $\vec{u}$  = displacement vector,  $\rho$  = density (i)

The solutions of this system of differential equations must also fulfil the boundary conditions, that is: zero stress over the complete surface in the practical experimental arrangement. If the partial spectra of only the torsional and longitudinal oscillations are evaluated, we obtain the frequency Equations:

*The platinum group metals are well suited for use at extremely high temperatures under mechanical loads and simultaneous corrosive attack. They have high melting points, excellent chemical stability and are highly resistant to oxidation. When using these materials in the design of components it is necessary to have data available on their elastic properties as a function of temperature. In this paper, investigations are presented into the temperature dependence of Young's modulus, the modulus of rigidity and Poisson's ratio for platinum, platinum alloys, rhodium and iridium. Measurements were carried out at the Friedrich Schiller University, Jena, using a resonance technique. Influences from both the microstructure and the alloying elements on the elastic properties and their temperature dependence were found.*

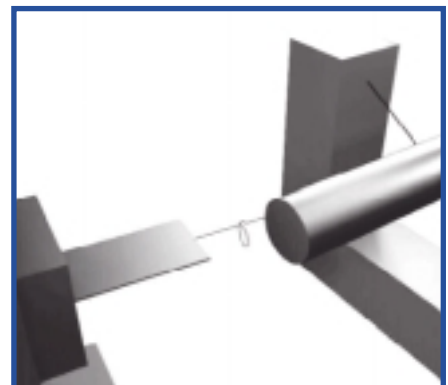
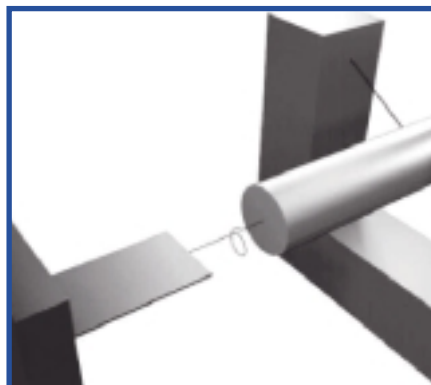


Fig. 1 The elastic properties of metal and alloy samples determined at various temperatures in a high temperature furnace. The beam is supported on alumina knife-edges. Oscillations are generated with the aid of a network analyser, transformed into mechanical oscillations by piezo sensors and transmitted to the beam via alumina fibre couplers

Frequency of torsional oscillations

$$f_{Tn} = \frac{n}{2l} \sqrt{\frac{G}{\rho}} F_{Tn} \quad (ii)$$

with  $F_{Tn} = 1$  for circular cylindrical beams,  $G$  = modulus of rigidity,  $l$  = beam length,  $n$  = order

Frequency of longitudinal oscillations

$$f_{Ln} = \frac{n}{2l} \sqrt{\frac{E}{\rho}} F_{Ln} \quad (iii)$$

where the factor  $F_{Ln}$  for circular cylindrical beams is derived from the Equation:

$$\left[ F_{Ln}^2(1+\nu) - 1 \right]^2 \varepsilon_n J_0(ha) J_1(ka) + ha J_1(ha) \left[ ka J_0(ka) - F_{Ln}^2(1+\nu) J_1(ka) \right] = 0$$

where  $(ha)^2 = \varepsilon_n \left[ F_{Ln}^2 \frac{(1-2\nu)(1+\nu)}{(1-\nu)} - 1 \right]$

$$(ka)^2 = \varepsilon_n \left[ F_{Ln}^2 2(1+\nu) - 1 \right] \text{ and } \varepsilon_n = \left( \frac{n\pi a}{l} \right)^2$$

( $J_0, J_1$  are Bessel functions of the first kind,  $a$  = radius)  
If  $F_{Ln}^2$  from Equation (iv) is developed into a power series in  $\varepsilon_n$ , we obtain:

$$F_{Ln}^2 = 1 + \varepsilon_n k_1 + \varepsilon_n^2 k_2 + \dots \quad (v)$$

with  $k_1 = -\frac{1}{2}\nu^2$  and

$$k_2 = -\frac{\nu^2}{48(1-\nu^2)} [7 - 4\nu - 32\nu^2 + 4\nu^3 + 24\nu^4]$$

Equation (v) thus obtained shows clearly the dependence of the factor  $F_{Ln}$  on  $\nu$  and  $na/l$  which is caused by the coupling of the longitudinal and transverse oscillations (dispersion). However, it also shows that the accuracy of the basic theory ( $F_{Ln} = 1$ ) is insufficient and that the more precise modelling permits the determination of Young's modulus and Poisson's ratio ( $\nu_D$ ) from a measured partial spectrum of the longitudinal characteristic frequencies alone. The modulus of rigidity can be determined from the measured partial spectrum of the torsional oscillations according to Equation (ii).

The temperature dependence of the elastic constants was determined in a high temperature furnace. The cylindrical sample beam is supported on alumina knife-edges, on the right of each diagram in Figure 1. The oscillations were generated using a network analyser, transformed into mechanical oscillations via piezo sensors (on the left of each diagram) and transmitted to the beam via fine alumina fibre couplers. The oscillations of the sample are detected via a further alumina

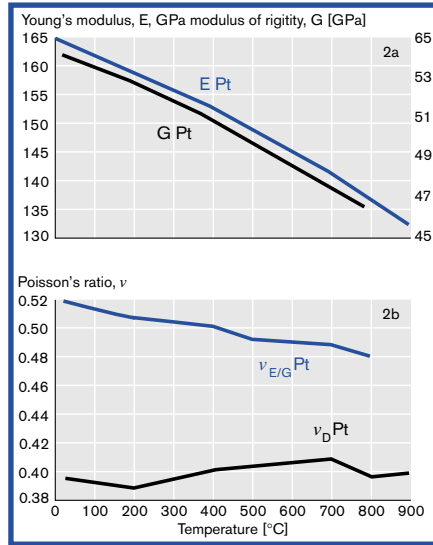


Fig. 2 Temperature dependences of: (a) Young's modulus, E, and the modulus of rigidity, G, for platinum; (b) Poisson's ratio,  $\nu$ , for platinum. The value  $\nu_D$  was determined from the dispersion of the characteristic longitudinal frequencies; while  $\nu_{E/G}$  was determined from the relationship  $\nu_{E/G} = E/(2G) - 1$

coupler attached to a second piezo sensor (not shown) and transmitted back to the network analyser for processing. The alumina fibre coupler is placed at the centre of the circular end surface of the sample if longitudinal oscillations are to be analysed (left-hand diagram) or at the circumference of the end surface for torsional oscillations (right-hand diagram). The resonant frequencies and the half-peak width of the amplification function (determining damping) can be recorded. The sample beam requires time to achieve a stable temperature between measurements to avoid errors.

The elastic constants, Young's modulus E, the modulus of rigidity G and Poisson's ratio  $\nu$  were measured on platinum, iridium and rhodium and on alloys of platinum with 10, 20 and 30 weight per cent of iridium and rhodium at both room temperature and elevated temperatures, by the resonance method. Poisson's ratio,  $\nu$ , was determined as  $\nu_D$  from the dispersion of the characteristic longitudinal frequencies and also as  $\nu_{E/G}$  from the relationship  $\nu_{E/G} = E/(2G) - 1$ . If the two values are the same the sample is isotropic or quasi-isotropic.

All the materials could be measured at temperatures where the loss factor of internal friction (damping),  $d$ , was not greater than  $10^{-2}$ . At higher values of

loss factor it was not possible to determine the resonance point reliably from the amplification function\*.

Elastic Properties of Platinum

Measurements with reproducible results were possible up to 800°C and in the case of repetition up to 900°C. Both Young's modulus and the modulus of rigidity of platinum show a steady decrease with increasing temperature, see Figure 2. This is partly in contrast to earlier determinations (2, 20) which showed a steady decrease in Young's modulus from 174 GPa at room temperature to 168 GPa at 400°C during a first measurement, followed by a decrease to 146 GPa at 500°C and then a steady decrease to 135 GPa at 700°C. This effect was found to be irreversible. Repeat measurements showed a Young's modulus of 155 GPa at room temperature which decreased continuously to 127 GPa at 800°C. The current measured values given in Table I were determined on as-cast platinum rods, and show relatively good agreement with the repeat determinations and with values measured at temperatures  $\geq 500^\circ\text{C}$  (2). The irreversible decrease in Young's modulus found in the earlier work was apparently due to a deformation structure in the material which was removed by recrystallisation during the measurement.

Temperature Dependence of the Elastic Properties E, G and  $\nu$  for Platinum

T [°C]	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$
25	164.6	0.396	54.2	0.518
200	159.3	0.389	52.9	0.506
400	153.3	0.401	51.1	0.500
500	149.1	0.403	50.0	0.491
600	145.6	0.406	48.9	0.489
700	141.9	0.409	47.7	0.487
800	137.8	0.396	46.6	0.479
900	132.7	0.399		

Table I

It is interesting that the values of Young's modulus determined at room temperature on the specimen with the apparently deformed structure correspond reasonably well with the values in the literature (4, 9, 16), whereas the values determined on platinum in the recrystallised state (155 GPa) and the as-cast state (165 GPa) are lower. Furthermore, Young's modulus was found to be dependent on the purity of

\* See <http://www.uni-jena.de/matwil/mechanik/literatur.html> for further information.

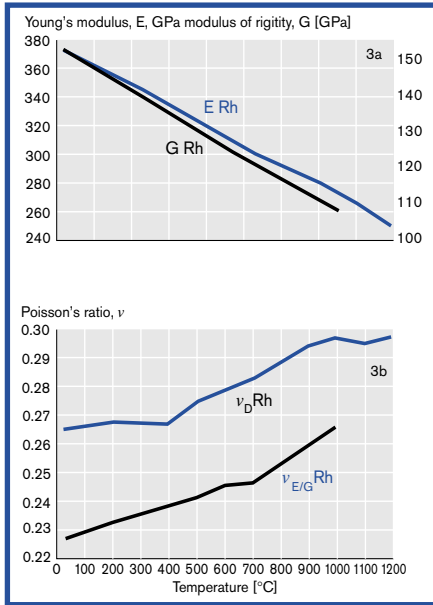


Fig. 3 Temperature dependence of: (a) the elastic properties E and G for forged rhodium (b) Poisson's ratio for forged rhodium

the platinum. On undeformed specimens, the following values were determined: 169 GPa with 99.99% Pt, 172 GPa with 99.95% Pt and 177 GPa with 99.9% Pt.

The value for Poisson's ratio determined from the dispersion of the longitudinal characteristic frequencies  $\nu_D$  is approximately constant over the whole temperature range, whereas the value of Poisson's ratio determined from the elastic moduli  $\nu_{E/G}$  decreases slightly with increasing test temperature. The difference between  $\nu_D$  and  $\nu_{E/G}$  indicates some influence from anisotropy which may be related to the primary solidification structure.

**Temperature Dependence of the Elastic Properties E, G and  $\nu$  for Forged Rhodium**

T [°C]	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$
25	372.4	0.266	151.7	0.227
200	355.8	0.268	144.3	0.233
400	332.1	0.267	134.2	0.237
500	321.4	0.274	129.5	0.241
600	310.4	0.278	124.7	0.245
700	299.4	0.282	120.3	0.246
800	291.0	0.287	116.2	0.252
900	281.6	0.293	111.9	0.258
1000	271.5	0.296	107.3	0.265
1100	260.6	0.294		
1200	246.9	0.296		

Table II

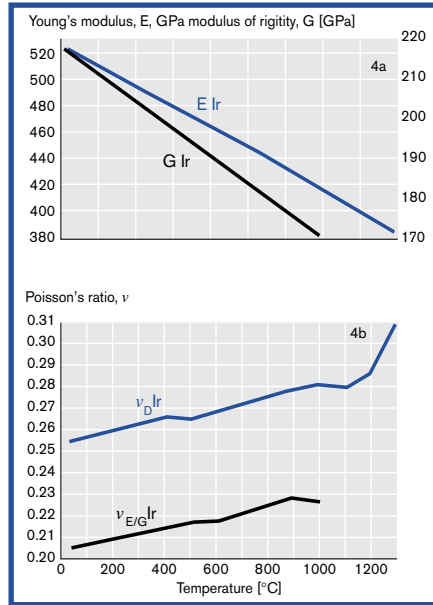


Fig. 4 Temperature dependence of: (a) the elastic properties E and G for as-cast iridium (b) Poisson's ratio for as-cast iridium

**Elastic Properties of Rhodium**

At room temperature, Young's modulus for rhodium (373 GPa to 384 GPa (2)) is considerably higher than that for platinum. With increasing temperature Young's modulus decreases in an approximately linear manner to 280 GPa (at 1000°C) (2) and 248 GPa (at 1200°C). The modulus of rigidity also shows a linear decrease with increasing temperature.

A comparison of the current measurements, also carried out on forged and subsequently machined rhodium rods (Table II and Figure 3), and earlier investigations (2) shows that for Young's modulus, the earlier measurements are reproducible at about 10 GPa higher than current values. The earlier values for Poisson's ratio  $\nu_D$  and  $\nu_{E/G}$  differ by only about 5 per cent (2), while in the current measurements the difference is 12 to 15 per cent. This means that the anisotropy is significantly less for those samples with the higher Young's modulus. This difference is presumably related to the fact that the earlier samples (2) were more severely deformed by forging because a larger ingot size had been used. The values for Young's modulus given in the literature (4, 7) also indicate that the microstructure is relatively severely deformed.

**Elastic Properties of Iridium**

Iridium has the highest Young's modulus of all face-centred cubic metals and the

**Temperature Dependence of the Elastic Properties E, G and  $\nu$  of As-cast Iridium**

T [°C]	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$
25	525.5	0.254	218.2	0.204
200	507.4	0.260	209.9	0.209
400	483.6	0.261	199.4	0.213
500	472.7	0.265	194.3	0.216
600	461.2	0.268	189.5	0.217
700	450.5	0.271	184.5	0.221
800	439.9	0.275	179.7	0.224
900	429.5	0.279	174.9	0.228
1000	417.5	0.281	170.3	0.226
1100	406.1	0.279		
1200	394.4	0.286		
1300	384.2	0.309		

Table III

highest modulus of rigidity of all metals. The elastic properties E, G,  $\nu_D$  and  $\nu_{E/G}$  measured on iridium in the as-cast state are summarised in Table III. Young's modulus and the modulus of rigidity decrease linearly from room temperature with increasing temperature, see Figure 4. At 1000°C the modulus of rigidity was still 170 GPa and Young's modulus 417 GPa. Young's modulus could be measured up to 1300°C (382 GPa). The values for Poisson's ratio  $\nu_D$  and  $\nu_{E/G}$  increase with increasing test temperature. The difference between the two values was about 18 per cent. This indicates marked anisotropy associated with the primary as-cast microstructure. A comparison of these results with previous investigations (2) shows that deformation by hot rolling leads to somewhat higher values for Young's modulus ( $E_{RT} = 532$  GPa,  $E_{1000°C} = 424$  GPa) and the modulus of rigidity ( $G_{RT} = 223$  GPa,  $G_{1000°C} = 173$  GPa).

These prior values correspond relatively well with data from the literature (4, 9, 16). However, although the increase in Poisson's ratio with increasing temperature measured by both sets of investigations corresponds qualitatively fairly closely, more substantial discrepancies are determined between  $\nu_D$  and  $\nu_{E/G}$  (~35 per cent), thus indicating a high degree of anisotropy caused by the deformation microstructure from the hot rolling.

Elastic Properties E, G and  $\nu_D$  and  $\nu_{E/G}$  for As-cast Platinum-Rhodium Alloys at Selected Temperatures

T [°C]	Pt-10%Rh				Pt-20%Rh				Pt-30%Rh			
	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$
25	212.6	0.365	78.0	0.363	245.9	0.342	91.6	0.342	277.7	0.324	104.8	0.325
200	206.3	0.368	75.4	0.368	236.6	0.346	87.8	0.347	265.7	0.330	99.9	0.330
400	197.9	0.372	72.1	0.372	224.7	0.351	83.3	0.349	251.0	0.334	94.0	0.335
500	193.3	0.376	70.5	0.371	218.8	0.353	80.9	0.352	243.9	0.338	91.1	0.339
600	188.7	0.376	68.7	0.373	213.0	0.355	78.6	0.355	236.6	0.340	88.2	0.341
700	183.9	0.378	66.9	0.374	207.2	0.358	76.3	0.358	229.5	0.343	85.5	0.342
800	179.2	0.379	65.2	0.374	201.0	0.359	74.1	0.356	222.1	0.345	82.7	0.343
900	175.0	0.383	63.4	0.380	195.5	0.360	72.0	0.358	215.7	0.346	80.0	0.348
1000	169.7	0.381			189.8	0.362	69.8	0.360	209.3	0.350	77.5	0.350
1100	164.9	0.385			184.6	0.367	67.7	0.363	202.8	0.352	74.7	0.357
1200					179.2	0.380			195.4	0.358		

Table IV

**Elastic Properties of Platinum-Rhodium Alloys**

The elastic properties E, G,  $\nu_D$  and  $\nu_{E/G}$  determined for alloys Pt-10%Rh, Pt-20%Rh and Pt-30%Rh as a function of temperature for specimens in the as-cast condition are presented in Table IV. Young's modulus and the modulus of rigidity decrease linearly with increasing temperature, see Figures 5a and 5b.

The values for Poisson's ratio  $\nu_D$  and  $\nu_{E/G}$  show only slight differences which become negligible at high rhodium concentrations, Figure 5c. In contrast to the large discrepancies found for the pure metals, these small differences may be due to the influence of solid solution formation during the development of the primary cast microstructure. The damping showed maxima in specific regions for the various alloys. This indicates a miscibility gap in the

binary Pt-Rh system similar to that shown in Figure 6 (17).

The higher values in the literature for Young's modulus at room temperature (4, 18) have a high probability of being attributable to prior deformation of the specimens. Figure 7 shows the effect of rhodium content on Young's modulus of specimens in the as-cast condition at various test temperatures. The greatest

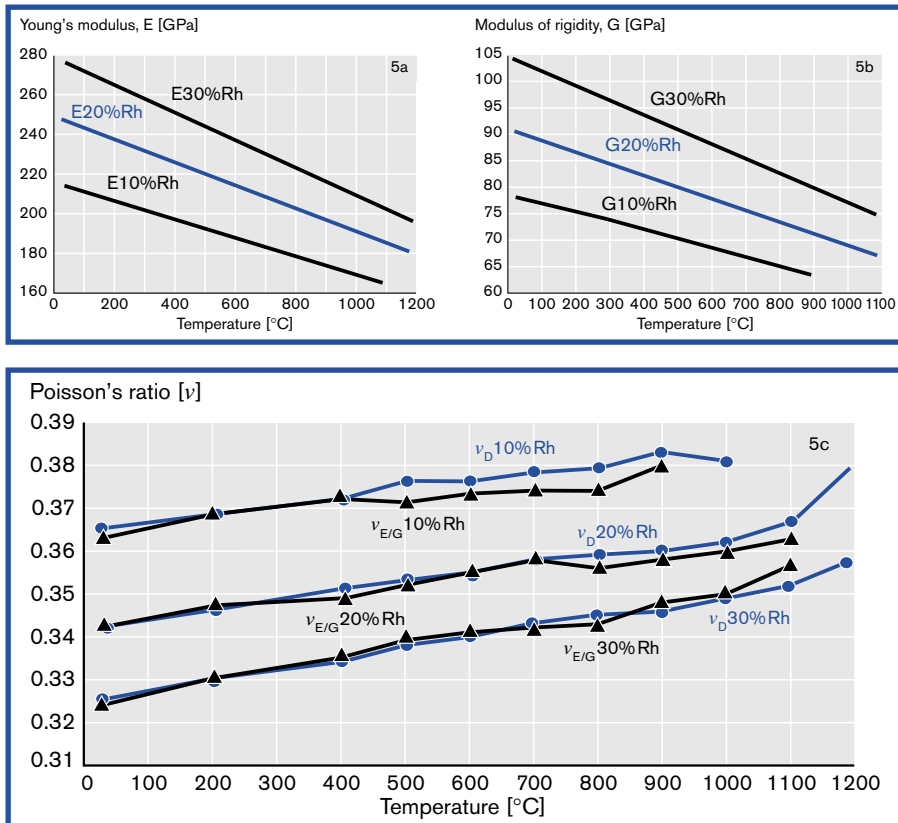


Fig. 5 Dependence of: (a) Young's modulus on temperature for as-cast Pt-Rh alloys. (b) The modulus of rigidity on temperature for as-cast Pt-Rh alloys. (c) Poisson's ratio on temperature for as-cast Pt-Rh alloys

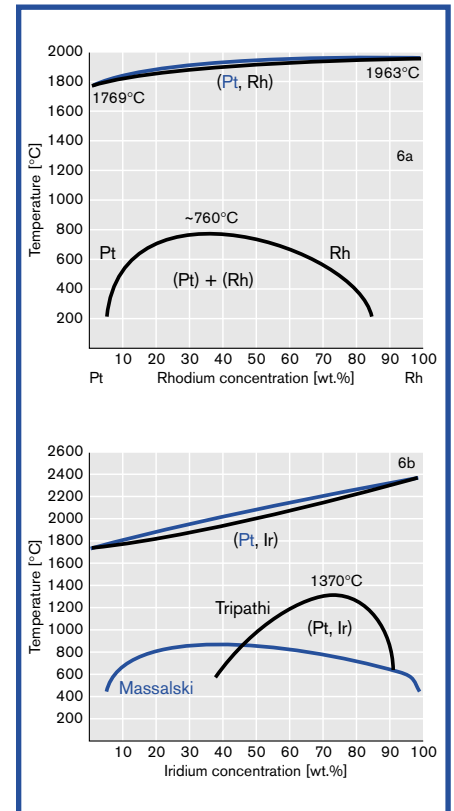


Fig. 6 Phase diagrams of the binary systems: (a) Pt-Rh system (17); (b) Pt-Ir system (17, 20)

Elastic Properties E, G and  $\nu_D$  and  $\nu_{E/G}$  for As-cast Platinum-Iridium Alloys at Selected Temperatures

T [°C]	Pt-10%Ir				Pt-20%Ir				Pt-30%Ir			
	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$	E [GPa]	$\nu_D$	G [GPa]	$\nu_{E/G}$
25	202.3	0.378	73.4	0.378	233.3	0.368	85.5	0.364	263.3	0.346	97.5	0.350
200	196.6	0.382	71.1	0.382	224.8	0.368	82.2	0.347	253.6	0.351	93.6	0.352
400	188.3	0.382	68.1	0.382	214.3	0.371	78.2	0.370	240.8	0.354	88.6	0.359
500	183.9	0.384	66.4	0.385	209.0	0.373	76.2	0.371	234.7	0.356	86.2	0.361
600	178.8	0.381	64.8	0.381	201.6	0.379	73.9	0.364	228.5	0.358	83.9	0.362
700	173.6	0.382	62.8	0.381	196.2	0.378	71.9	0.364	222.5	0.361	81.5	0.365
800	170.7	0.389	58.1		192.3	0.384	70.1	0.372	216.1	0.359	79.3	0.363
900	166.4	0.391			186.9	0.378	68.2	0.370	210.2	0.363	76.9	0.367
1000	162.2	0.396			182.5	0.386	66.2	0.378	204.5	0.368	74.7	0.369
1100	157.1	0.400			176.9	0.387	64.1	0.380	198.5	0.368	72.5	0.369
1200	150.8	0.393			171.1	0.386			192.2	0.372		
1300					165.0	0.393			185.3	0.374		
1400									176.8	0.375		

Table V

effect on Young's modulus due to rhodium additions is observed for concentrations of up to ~10 weight per cent. The rate of increase is less marked at higher rhodium contents. A similar effect has been found for the stress-rupture strength of Pt-Rh alloys (19).

**Elastic Properties of Platinum-Iridium Alloys**

The elastic properties E, G,  $\nu_D$  and  $\nu_{E/G}$  determined on specimens of as-cast alloys Pt-10%Ir, Pt-20%Ir and Pt-30%Ir are shown in Table V as functions of temperature. Young's modulus and the modulus of rigidity decrease linearly with increasing temperature, see Figure 8. The differences between the values for Poisson's ratio  $\nu_D$  and  $\nu_{E/G}$  are somewhat greater for the Pt-Ir alloys than for the Pt-Rh alloys. At this stage, it is not clear why the difference for Pt-20%Ir is so large.

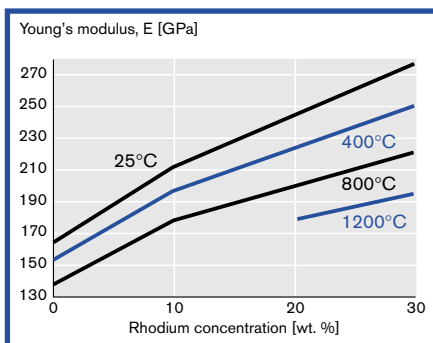


Fig. 7 Dependence of Young's modulus on rhodium content for as-cast Pt-Rh alloys at various temperatures

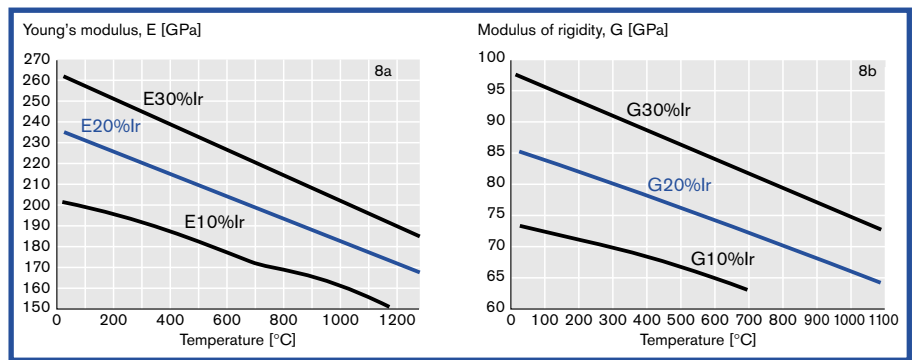


Fig. 8 Dependence of: (a) Young's modulus on temperature for as-cast Pt-Ir alloys; (b) the modulus of rigidity on temperature for as-cast Pt-Ir alloys

The behaviour of the Pt-Ir alloys also indicates a maximum in damping corresponding to the miscibility gap (Figure 6b (17, 20)). This maximum was more clearly distinguished than that found in the Pt-Rh system.

In Figure 9 the influence of the iridium content on Young's modulus at various test temperatures is shown for as-cast specimens. The modulus increases nearly linearly with iridium content up to 30 weight

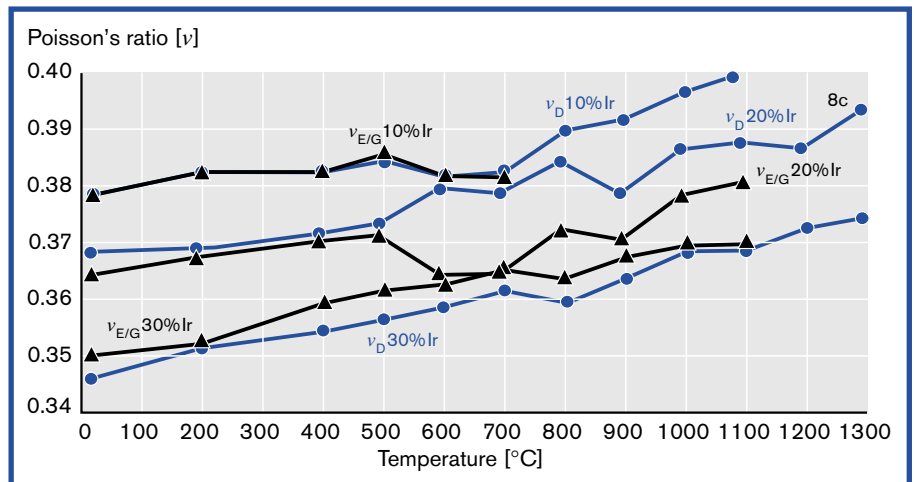


Fig. 8(c) Dependence of Poisson's ratio on temperature for as-cast Pt-Ir alloys

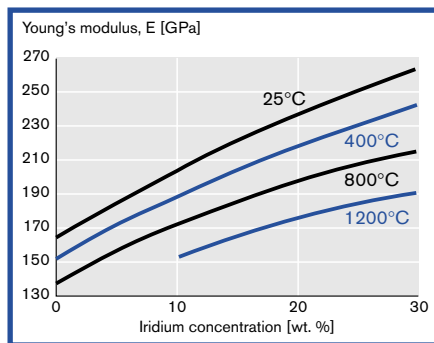


Fig. 9 Dependence of Young's modulus on iridium content for as-cast Pt-Ir alloys at various temperatures

per cent. Comparing values for Young's modulus shows generally good agreement with results of prior investigations (2) and data from the literature (4, 18). The relatively small discrepancies are attributable to different processing conditions.

## Conclusions

The results of investigations carried out using the resonance method show that Young's modulus and the modulus of rigidity of platinum, rhodium and iridium and various platinum alloys in the as-cast condition decrease linearly with increasing test temperature. The gradients of the lines are dependent on the compositions of the alloys.

The microstructural state of the material resulting from prior deformation influences in particular the magnitude of Young's modulus and the anisotropic behaviour of Poisson's ratio. Poisson's ratio is also influenced by the state of the primary as-cast microstructure.

A marked increase in damping was observed in the regions of the miscibility gaps. This suggests that the resonance method could be a sensitive technique for determining miscibility gaps in materials which can be subjected to mechanical oscillations and whose basic damping,  $d$ , is less than  $10^{-3}$  (21). Further microstructural and crystallographic investigations are required to confirm these correlations.

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