

High Temperature Mechanical Properties of the Platinum Group Metals

ELASTIC PROPERTIES OF PLATINUM, RHODIUM AND IRIIDIUM AND THEIR ALLOYS AT HIGH TEMPERATURES

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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.

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°C to 1650°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

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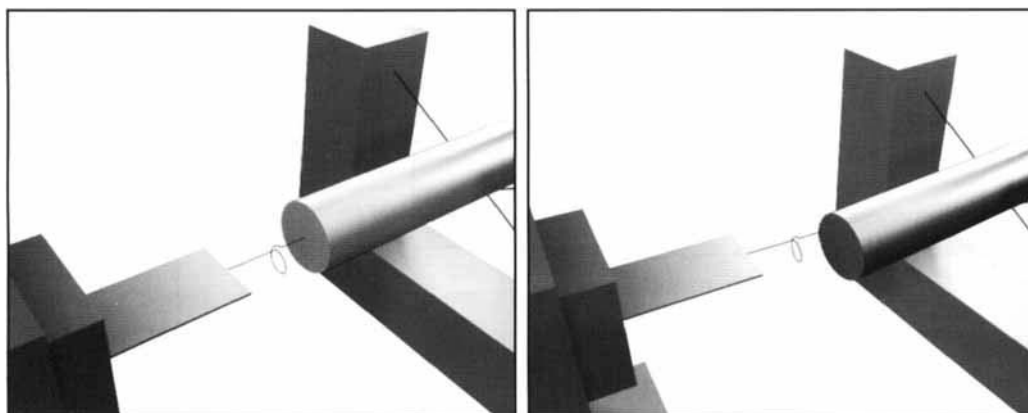


Fig. 1 The elastic properties of metal and alloy samples determined at various temperatures in a high temperature furnace. The beam is supported on alumina knife-edges. Oscillations are generated with the aid of a network analyser, transformed into mechanical oscillations by piezo sensors and transmitted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

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 (ν):

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

where \vec{u} = displacement vector, ρ = density

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:

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:

$$[F_{Ln}^2(1+\nu) - 1]^2 \epsilon_n J_0(ba) J_1(ka) + ba J_1(ba) [ka J_0(ka) - F_{Ln}^2(1+\nu) J_1(ka)] = 0 \quad (iv)$$

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

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

(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 ϵ_n , we obtain:

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

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 ν 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 (ν_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 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-

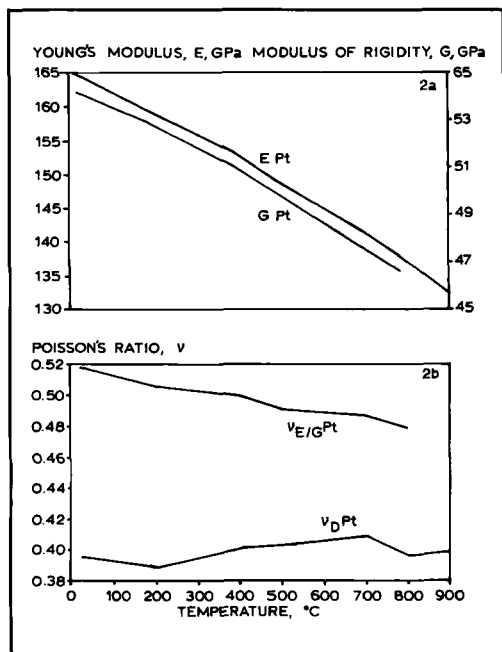


Fig. 2 Temperature dependences of: (a) Young's modulus, E , and the modulus of rigidity, G , for platinum; (b) Poisson's ratio, ν , for platinum. The value ν_D was determined from the dispersion of the characteristic longitudinal frequencies; while $\nu_{E/G}$ was determined from the relationship $\nu_{E/G}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

T, °C	E, GPa	ν_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		

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 ν 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, ν , was determined as ν_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

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

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.

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

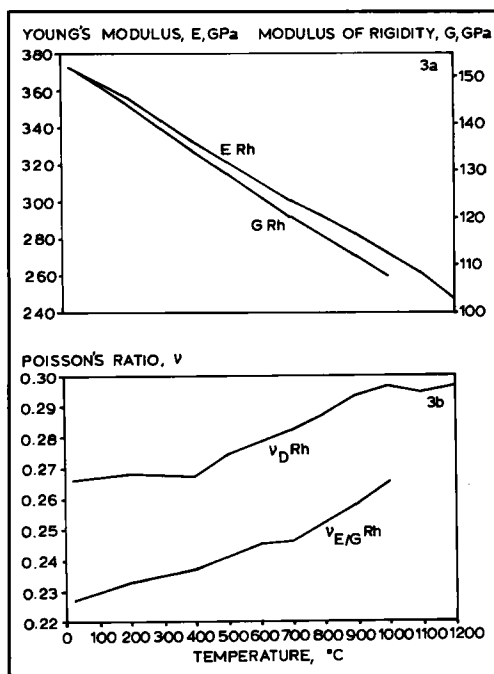


Fig. 3 Temperature dependence of: (a) the elastic properties E and G for forged rhodium (b) Poisson's ratio for forged rhodium

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

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

T, °C	E, GPa	ν_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		

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 ν_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 highest modulus of rigidity of all metals. The elastic properties E , G , ν_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 ν_D and $\nu_{E/G}$

T, °C	E, GPa	ν_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		

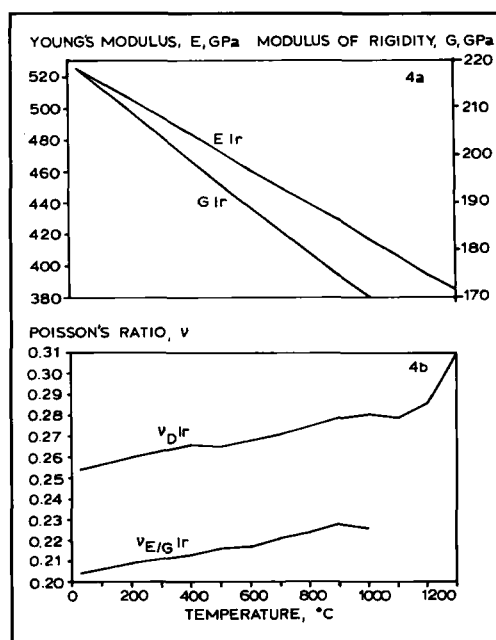


Fig. 4 Temperature dependence of:
(a) the elastic properties E and G for as-cast iridium
(b) Poisson's ratio for as-cast iridium

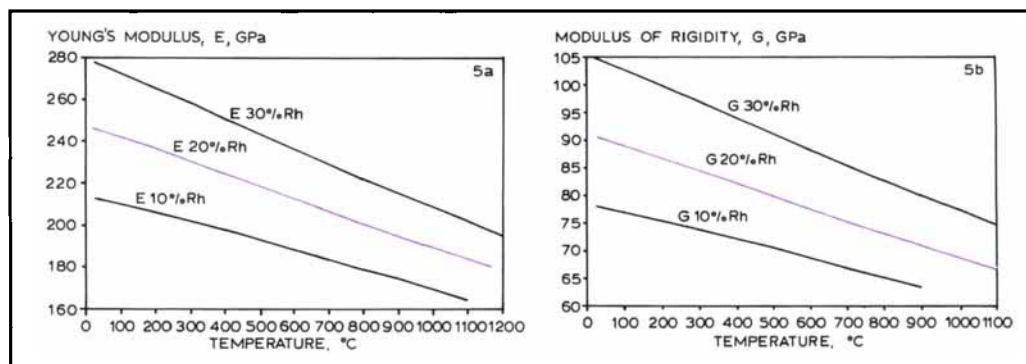
increase with increasing test temperature. The difference between the two values was about 18 per cent. This indicates marked anisotropy associated with the primary as-cast microstructure. A comparison of these results with previous investigations (2) shows that deformation by hot rolling leads to somewhat higher values for Young's modulus ($E_{RT} = 532$ GPa, $E_{1000^\circ\text{C}} = 424$ GPa) and the modulus of rigidity ($G_{RT} = 223$ GPa, $G_{1000^\circ\text{C}} = 173$ 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 ν_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 of Platinum-Rhodium Alloys

The elastic properties E , G , ν_D and $\nu_{E/G}$ determined for alloys Pt-10%Rh, Pt-20%Rh and Pt-30%Rh as a function of temperature for speci-

T, °C	Pt-10%Rh				Pt-20%Rh				Pt-30%Rh			
	E, GPa	ν_D	G, GPa	$\nu_{E/G}$	E, GPa	ν_D	G, GPa	$\nu_{E/G}$	E, GPa	ν_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		



mens 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 ν_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

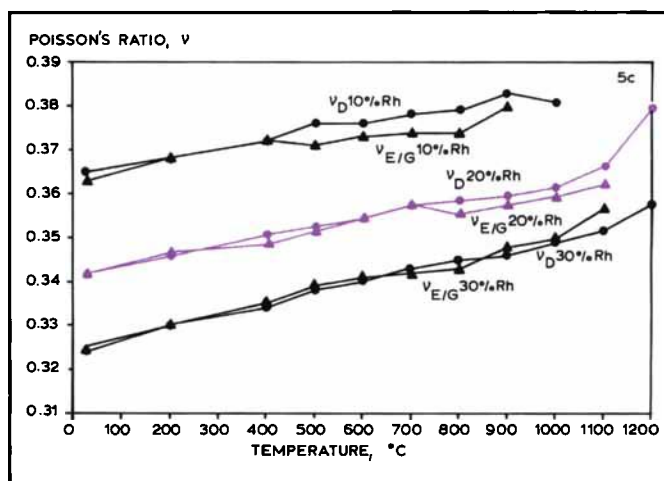


Fig. 5 Dependence of:
(a) Young's modulus on temperature for as-cast Pt-Rh alloys
(b) the modulus of rigidity on temperature for as-cast Pt-Rh alloys
(c) Poisson's ratio on temperature for as-cast Pt-Rh alloys

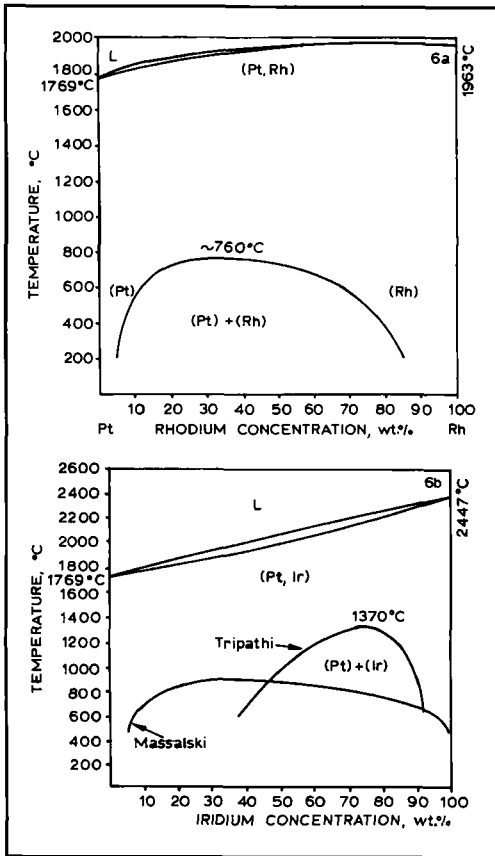


Fig. 6 Phase diagram of the binary systems: (a) Pt-Rh system (17); (b) Pt-Ir system (17, 20)

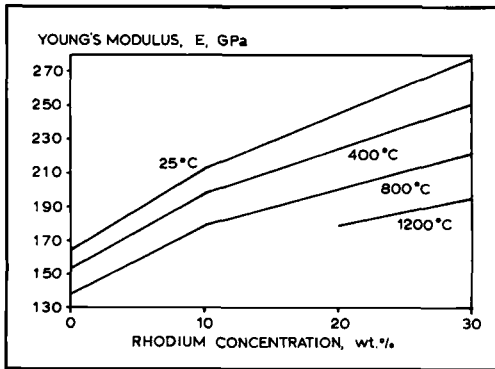


Fig. 7 Dependence of Young's modulus on rhodium content for as-cast Pt-Rh alloys at various temperatures

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 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 , ν_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 ν_D and $\nu_{E/G}$ are somewhat greater for the Pt-Ir alloys

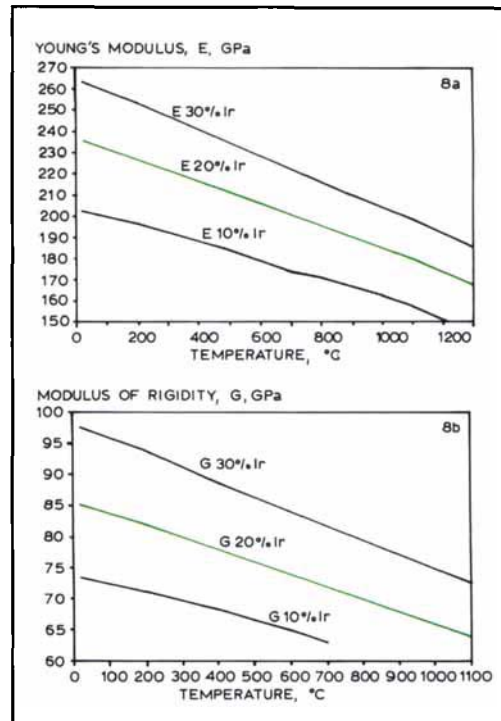


Fig. 8 Dependence of: (a) Young's modulus on temperature for as-cast Pt-Ir alloys; (b) the modulus of rigidity on temperature for as-cast Pt-Ir alloys

Table V Elastic Properties E, G, ν_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	ν_D	G, GPa	$\nu_{E/G}$	E, GPa	ν_D	G, GPa	$\nu_{E/G}$	E, GPa	ν_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	0.367	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			

than for the Pt-Rh alloys. At this stage, it is not clear why the difference for Pt-20%Ir is so large. 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 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

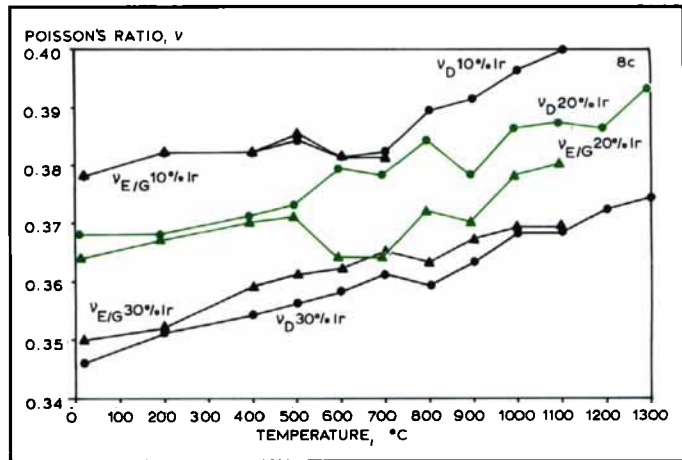


Fig. 8(c) Dependence of Poisson's ratio on temperature for as-cast Pt-Ir alloys

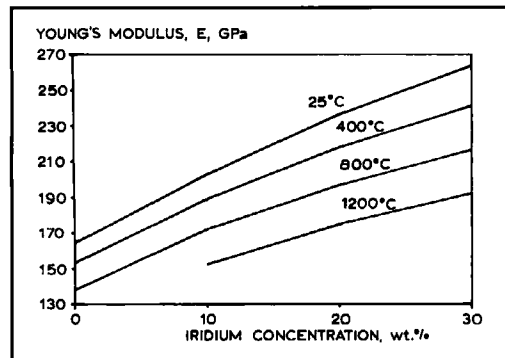


Fig. 9 Dependence of Young's modulus on iridium content for as-cast Pt-Ir alloys at various temperatures

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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Ruthenium-Initiated Star Polymers

Star-shaped polymers are attracting interest as polymeric materials because of their unusual structures. Such structures can be made by living polymerisation processes, one of which involves a linking reaction using living linear polymers and divinyl compounds.

Researchers at Kyoto University in Japan now report a multi-arm star-shaped polymer with a cross-linked microgel core (K.-Y. Baek, M. Kamigaito and M. Sawamoto, *Macromolecules*, 2001, 34, (2), 215–221). Using *in-situ* polymerisation of methyl methacrylate (MMA), a halide initiator and $\text{RuCl}_2(\text{PPh}_3)_3$, in the presence of $\text{Al}(\text{O}i\text{-Pr})_3$ a living poly(MMA) was formed which on reaction with a divinyl compound resulted in star-shaped polymers.

The yield depended on the structures of the initiators, divinyl compounds, monomers and other reaction conditions. The best system gave a polymer of about 20 poly(MMA) arms per molecule.