27th International Precious Metals Conference
June 14-17, 2003, Puerto Rico

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High Temperature Tensile Properties of Platinum Materials

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This paper continues a series of publications by the authors on the high temperature properties of the platinum group metals.

To the knowledge of the authors, the short-term tensile properties of platinum and its alloys have been measured only up to 700°C (see “Edelmetall-Taschenbuch” 2nd Edition, Degussa AG, Hüthig-Verlag, Heidelberg, 1995, ISBN 3-7785-2448-8).

The main difference between the current investigations and earlier work lies, however, not only in the achievement of higher temperatures but also in the use of quantitative techniques of stress and strain measurement.

Papers presented at the IPMI Conferences:

Iridium: An Exceptional Structural Material for High Temperatures
Philadelphia, USA, October 1995

Stress-Rupture Strength and Creep Behaviour of Platinum Alloys
San Francisco, USA, June 1997

Dispersion Hardened Platinum and Platinum Alloys for Very High Temperature Applications
Toronto, Canada, June 1998

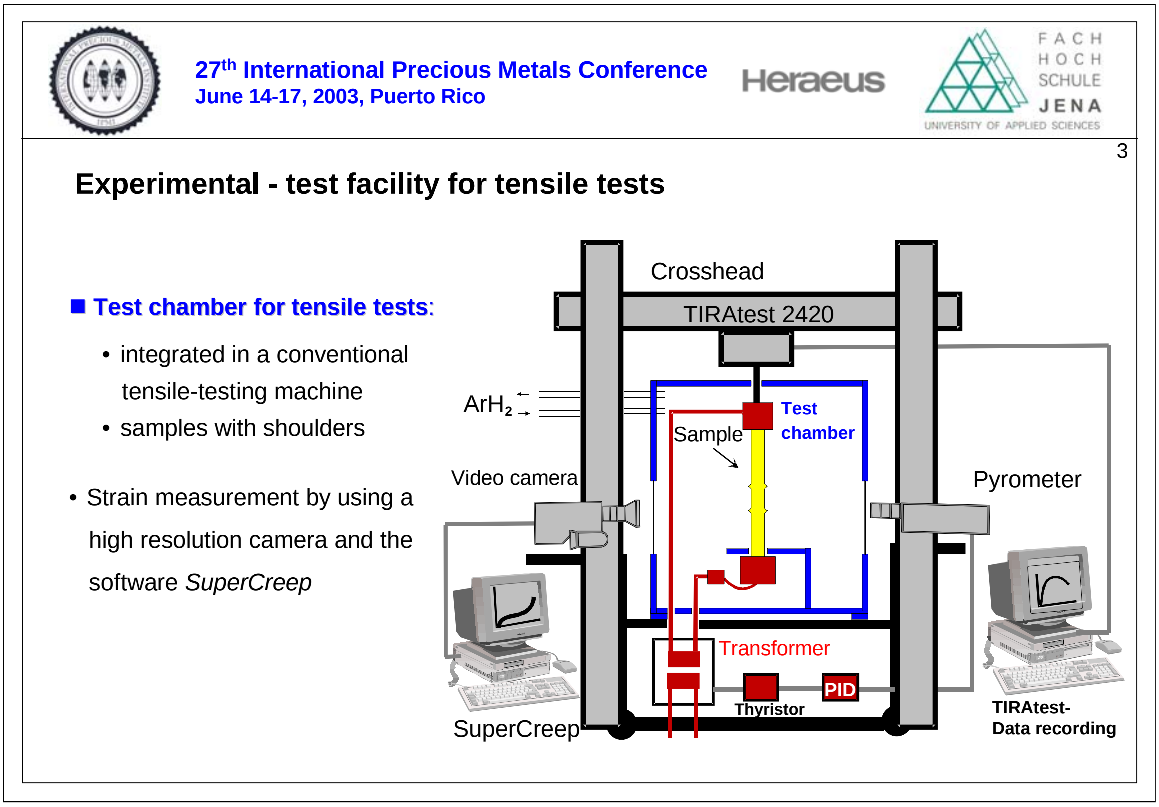
FEM-Modeling the Creep Behavior of Platinum Alloys for the Glass Industry
Acapulco, Mexico, June 1999

Platinum Materials for the Glass Industry
Williamsburg, USA, June 2000

Practical Experience with New Oxide Dispersion Hardened Platinum Materials
Tucson, USA, June 2001

It has become our regular practice to present new results at the IPMI conferences. This illustration gives a summary of the papers we have presented to the Institute over the last eight years.

It is intended to publish the results of our new work in more detail in journals but, as in the past, we are pleased to be able to present the work first to the IPMI International Conference.



The basic design of the test facility is shown in this figure. The test chamber is mounted in a standard tensile testing machine which is used to apply stress to a specimen in order to achieve deformation. The rate of deformation in the present tests was maintained constant at 10 mm / min.

The specimen is heated directly by applying an electric current over its length.

The temperature is measured in the central portion of the specimen by an IR pyrometer. The measurement signal is fed to a PID controller which regulates the heating current and thus the temperature via a thyristor controller and transformer.

The strain of the specimen is determined by a CCD-camera as described below.

The chamber can be filled with inert or reducing gas for testing materials sensitive to oxidation.

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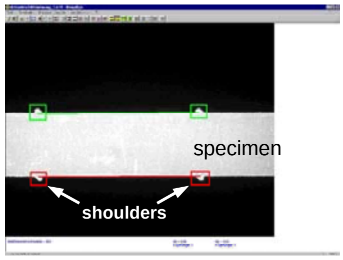
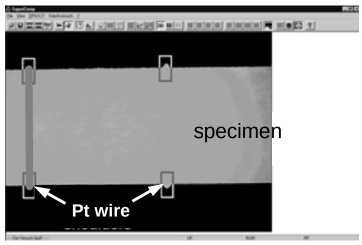
Experimental - specimen dimensions

Strain measurement by digital image analysis *SuperCreep*

- **Specimens I:**
  - Strips 120 × 4 × 0.8 mm³
  - 4 shoulders, laser cut



- **Specimens II:**
  - Wires, 120 mm long
  - Cross section approx. 1-5 mm²
  - Pt wires wound round a specimen as markers



The essential feature of the newly developed testing technique is the continuous measurement of strain at the highest temperatures. This is achieved using the specially developed digital image analysis system "SuperCreep".

The resistively heated specimens are approximately 120 mm long. Although the ends of the specimens are cold, leading to a temperature gradient over the length of the specimen, the central portion of the specimen has a very uniform temperature. This is the maximum temperature of the specimen.

Measurement of strain is achieved by means of markers on the specimen. These are located on both sides and separated by a distance of 10 mm. In the case of sheet and strip, the specimens are laser machined with small shoulders, whereas for wire and rod specimens the markers are provided by wrapping a fine wire round the specimen.

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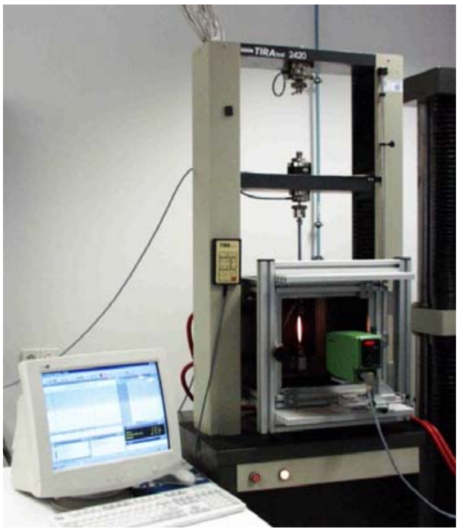
Experimental - high temperature tensile test system



The photographs show the test chamber mounted in the tensile testing machine.

The resistance heated specimen is clearly visible.

The IR pyrometer can be seen in the foreground.

In the left hand photograph a window can be seen behind the specimen. The CCD-camera for strain measurement is located behind this window.

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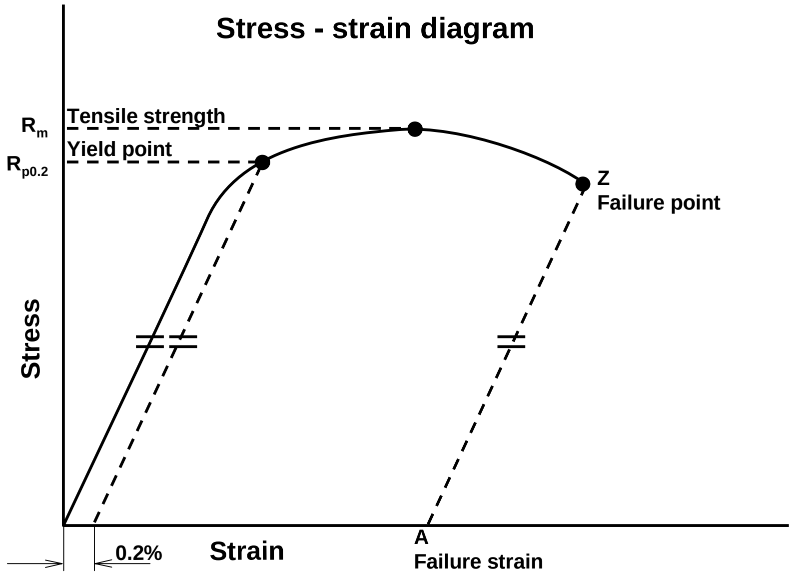
Stress - strain diagram



Our previous contributions have reported on the investigations of the stress rupture strength and the creep properties of the PGMs. We will, therefore, summarize briefly the main features of the tensile test, indicating the differences to the stress rupture and creep tests.

In stress rupture and creep testing the specimen is subjected to a constant load and time-dependent properties are determined (time to rupture and creep rate). In the tensile tests discussed here, the specimen is subjected to a constant rate of deformation and the applied stress is measured.

Initially a rapid increase in stress is observed. This represents the elastic range where the strain is reversible and the gradient is described by Young's modulus.

The onset of permanent irreversible deformation is the yield point. For practical purposes this is defined as the stress to achieve 0.2% permanent plastic deformation (this is also frequently designated the “0.2% proof stress”, $R_{p0.2}$).

Plastic deformation causes work hardening of the material and thus a further increase in stress up to the maximum “tensile strength” (R_m).

The reduction in the cross section of the specimen by necking and, at high temperatures, the formation of pores leads to a steady reduction in the applied stress until rupture occurs. The percentage elongation to rupture is the failure strain or tensile elongation (A).

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Materials examined

Pt

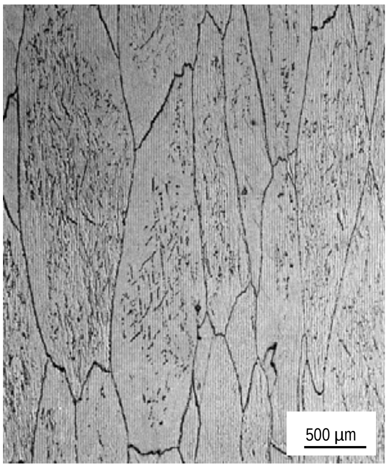
Pt-10%Rh	**Pt-10%Ir**	**Pt-5%Au**
Pt-20%Rh	**Pt-20%Ir**	
Pt-30%Rh		

Pt DPH
Pt-5%Au DPH
Pt-10%Rh DPH
} **Novel oxide dispersion hardened platinum materials (Pt DPH materials)**

The materials examined in the present work were commercially pure platinum and alloys of platinum with rhodium, iridium and gold. These materials were all produced by conventional melting and casting and were then rolled to sheet 0.8 mm thick.

The three dispersion hardened platinum materials (Pt DPH, Pt-5%Au DPH and Pt-10%Rh DPH) have been developed by Heraeus and the University of Applied Sciences in Jena and are manufactured commercially by Heraeus. Their high temperature tensile properties were determined for the first time in the course of this research programme and will therefore be discussed in more detail.

Optical micrograph showing the structure of Pt DPH



The platinum DPH materials are unique because they are produced by the internal oxidation of Pt doped with additions of e.g. Zr, Y, Ce in the bulk form. Other ODS materials are produced by essentially powder metallurgical routes, because the diffusion of oxygen in platinum usually only allows oxidation to depths of a few 10 µm.

After internal oxidation, Pt DPH shows oxides at the grain boundaries and in the grains. Subsequent deformation and annealing cause the distortion of the original grain boundaries and the formation of new grains by recrystallization. The newly formed grains are not directly related to the original grain boundaries visible here.

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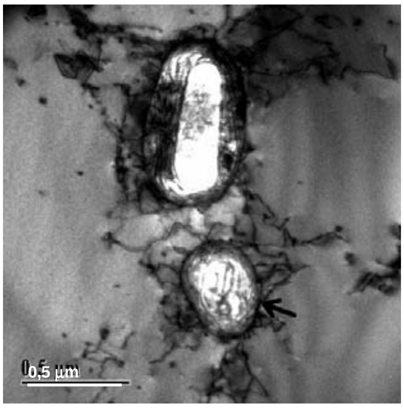
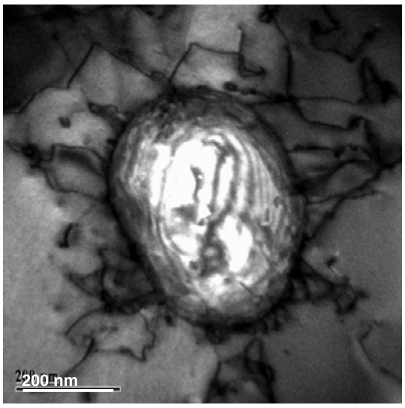
TEM bright-field image of Pt DPH after tensile loading at 1600°C





Transmission electron microscopy (TEM) shows that Pt DPH also contains sub-micron oxide particles which are not visible in the optical micrographs and are typically in the size range 100 – 500 nm. (Our previous report of even finer particles was incorrect. These were the result of an artefact from sample preparation).

The oxide particles interact strongly with individual dislocations and dislocation networks. They are believed to be responsible for the unusual mechanical properties, high strength and ductility, which have been previously demonstrated in stress-rupture and creep tests.

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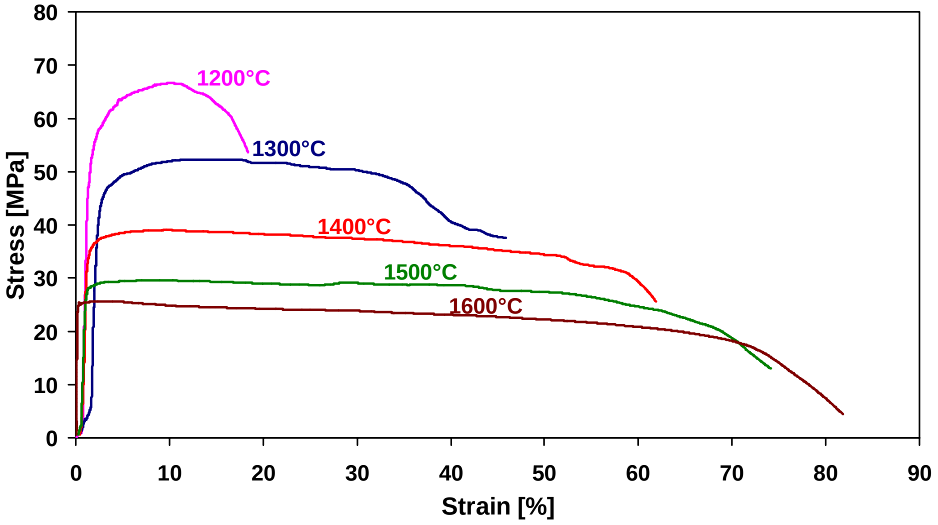
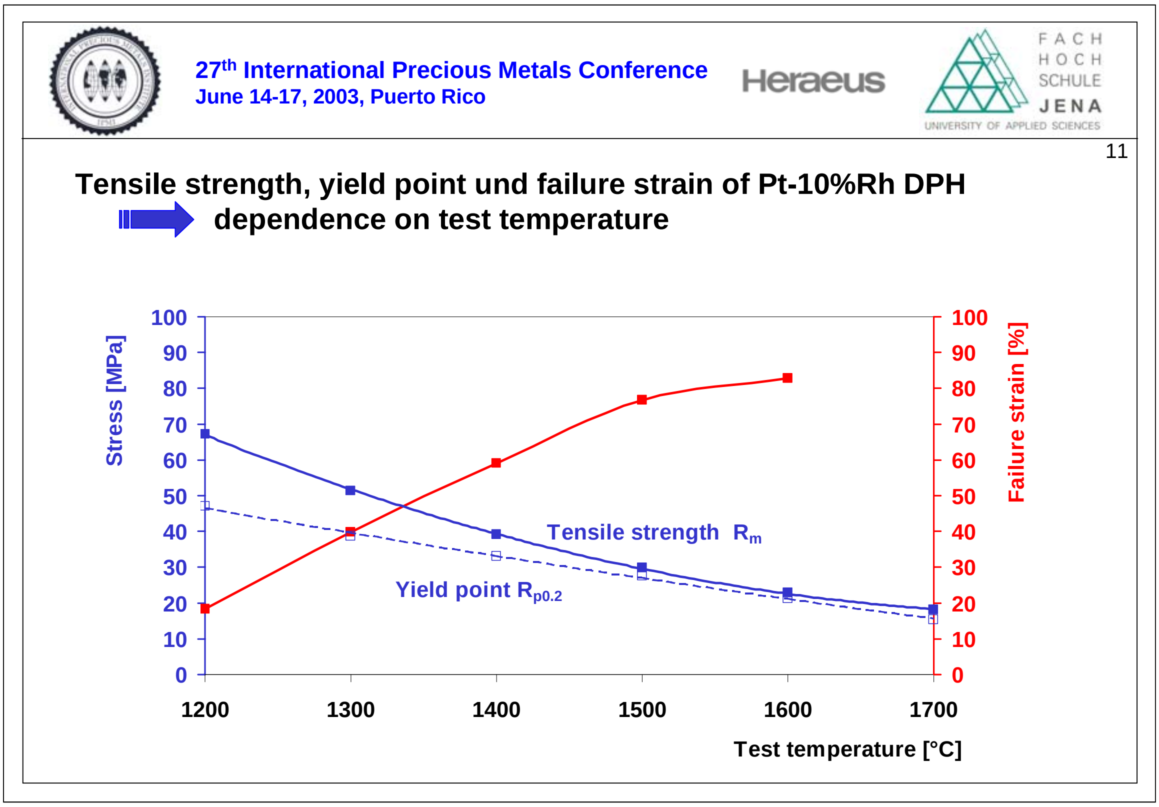
Stress-strain diagrams of Pt-10%Rh DPH



This diagram shows a family of tensile stress-strain curves determined at various temperatures on Pt-10%Rh DPH. An increase in temperature leads to a decrease in yield point and tensile strength. At the highest temperatures there is only a negligible difference between these two values, which indicates very limited work hardening.

The elongation to fracture increases continuously with increasing temperature in the range tested. This covers the typical range of temperatures for applications of dispersion hardened platinum materials.



The influence of temperature on the most important tensile properties ($R_{p0.2}$, R_m, A) is shown in this figure for the dispersion hardened alloy Pt-10%Rh DPH.

With increasing temperature we observe a steady decrease in the strength properties and a convergence of yield point and tensile strength.

For this material the ductility increases to remarkably high levels (approx. 80%).

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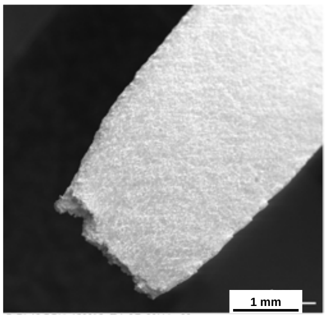
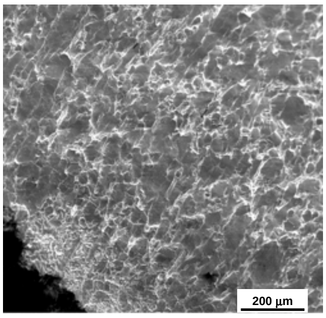
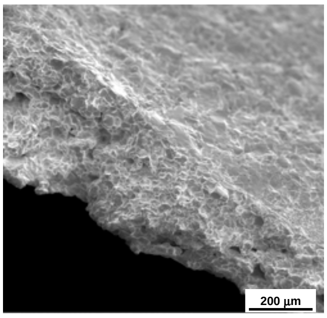
SEM images showing a specimen of Pt-10%Rh DPH after tensile test at 1600°C



Overview



Surface



Fracture surface

Examination of specimens of Pt-10%Rh DPH in the scanning electron microscope (SEM) after tensile testing at 1600°C shows considerable necking (left) and a fine grain structure which has produced an uneven surface as a result of the plastic deformation of the individual grains (middle).

The fracture face (right) shows a small number of pores and fracture along the individual grain boundaries. These are typical features of ductile behaviour at very high temperatures.

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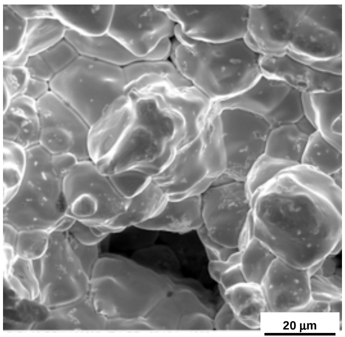
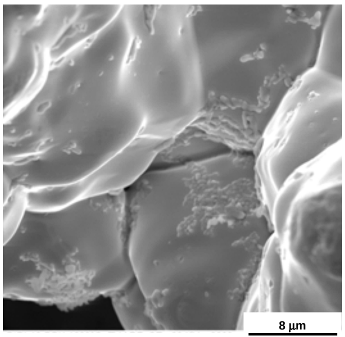
SEM images showing a specimen of Pt-10%Rh DPH after tensile test at 1600°C (higher magnification)



Fracture surface



Fracture surface

The features described in the previous illustration can be seen more clearly here at higher magnification.

There is very limited crack formation at the grain boundaries.

Occasional creep pores are found.

Small oxide particles are present on the intergranular fracture surfaces.

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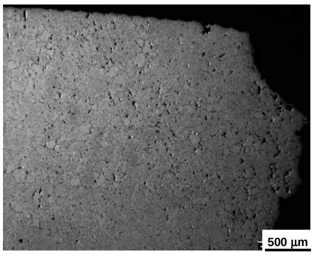
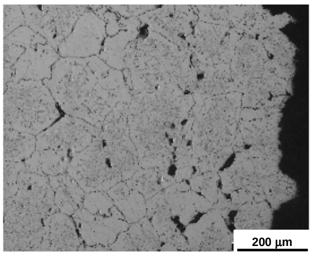
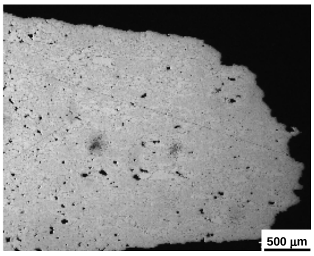
Optical micrographs showing structure of Pt-10%Rh DPH after tensile test (longitudinal sections, points of rupture)



1200°C



1200°C



1600°C

These optical micrographs show the structure close to the point of rupture for specimens of Pt-10%Rh DPH after tensile tests at 1200°C and 1600°C.

Even at the higher test temperature a very fine recrystallized structure is found.

The necking of the specimen indicates high ductility, especially at 1600°C.

Whereas at 1200°C typical elongated pores have developed along the grain boundaries, particularly at triple intersections, they take on the form of rounded voids at the higher temperature.

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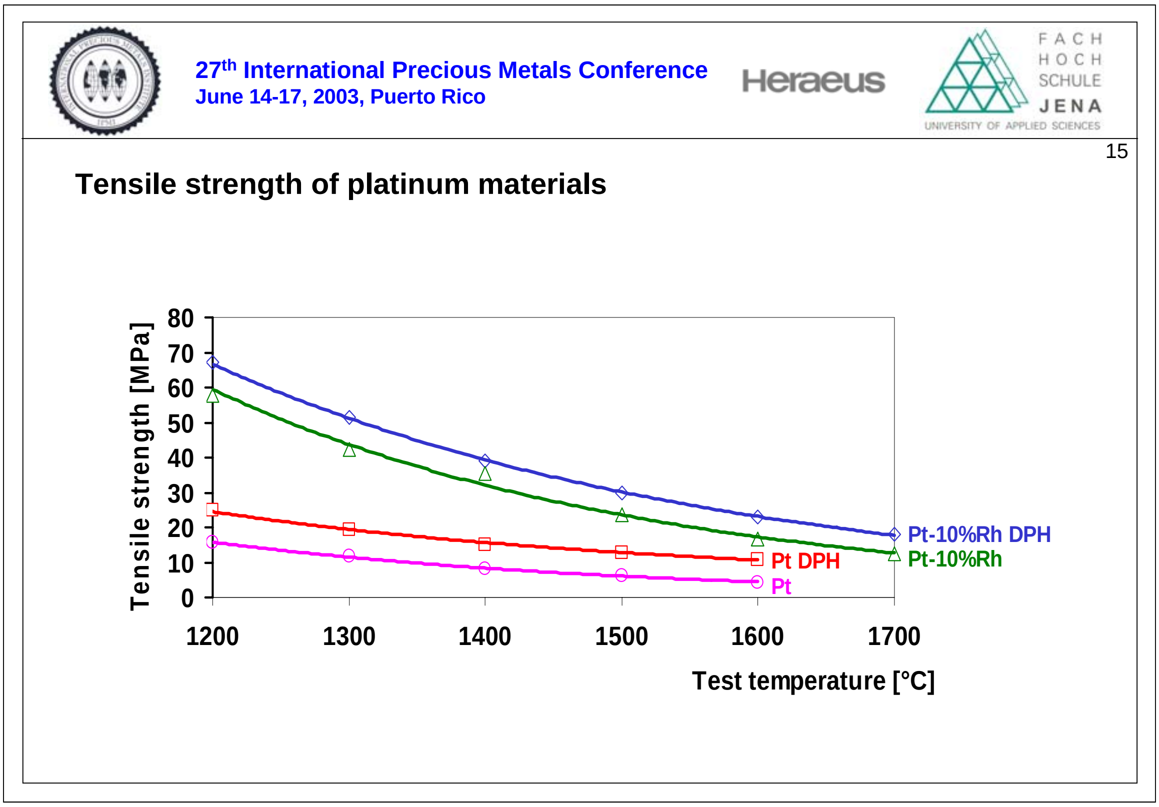
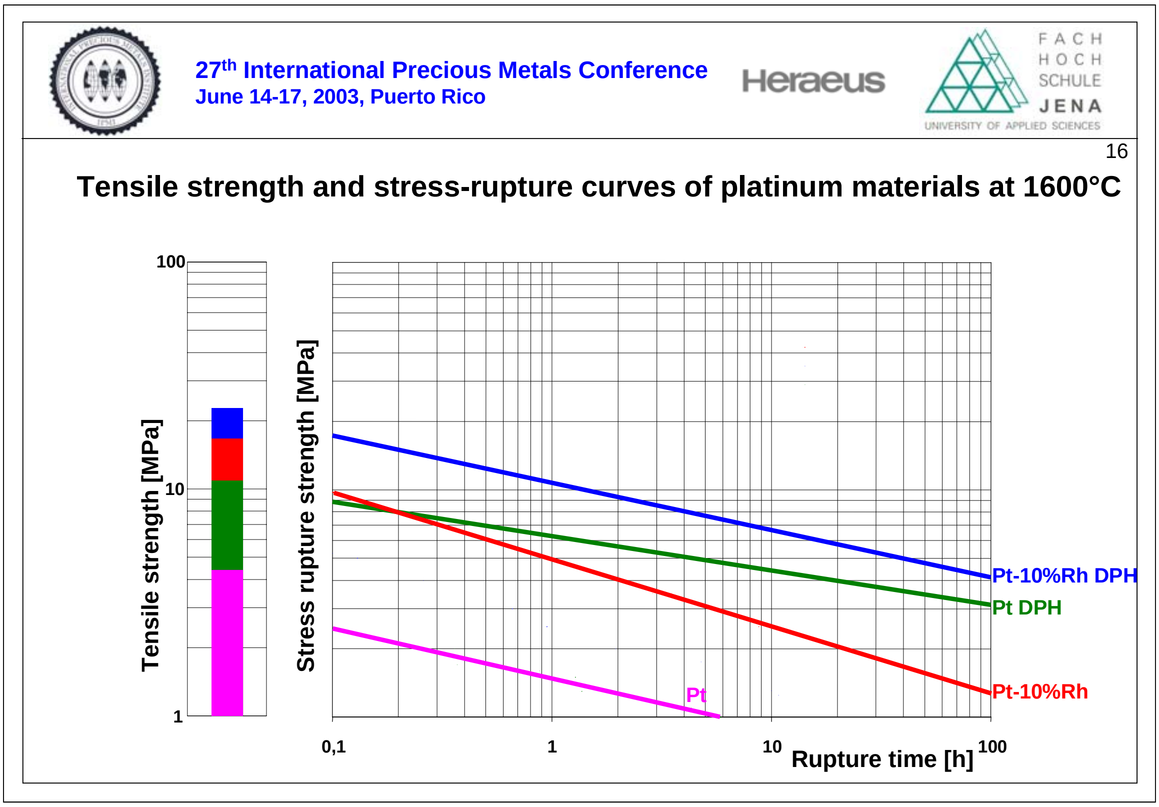


This diagram shows the variation of tensile strength with test temperature in the range 1200°C to 1700°C for conventional Pt-10%Rh and the dispersion hardened alloy Pt-10%Rh DPH.

Similar curves are shown for technically pure Pt and Pt DPH between 1200°C and 1600°C.

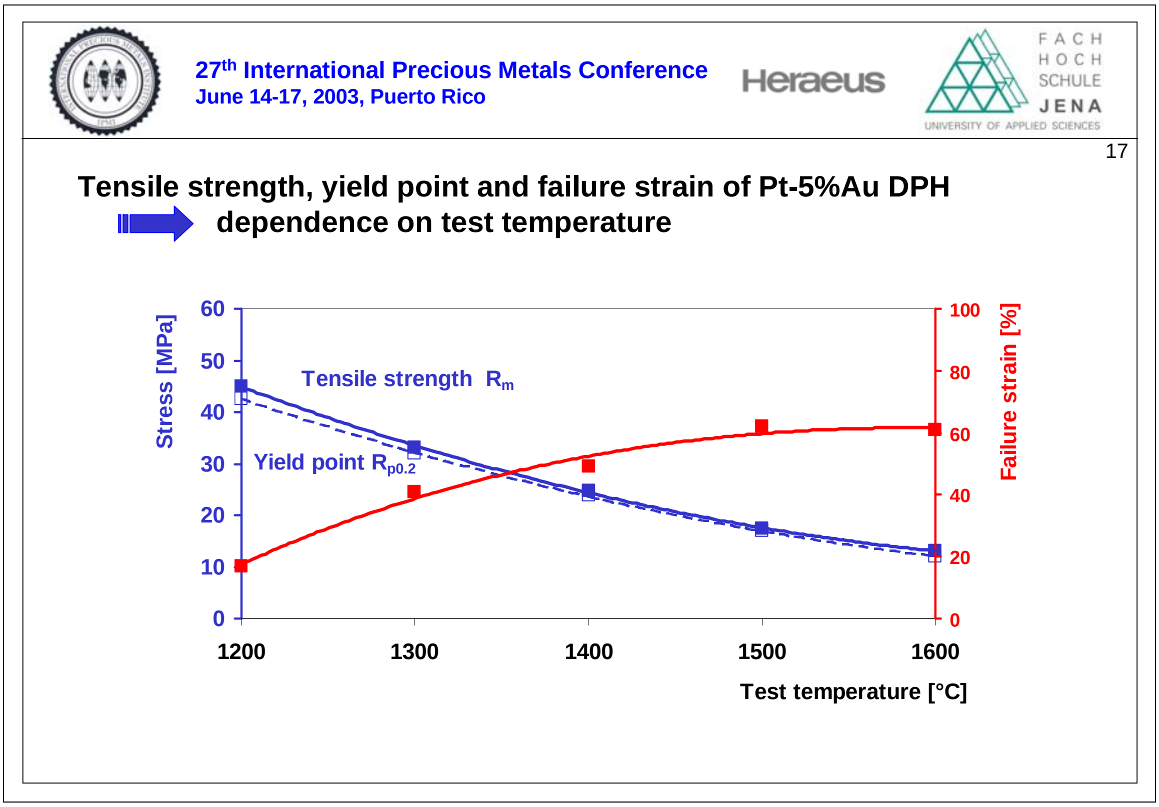
The curves show a steady decrease in tensile strength with increasing temperature but also demonstrate the strength advantage of the DPH materials at all test temperatures.



As pointed out above, tensile tests are carried out under a constant rate of deformation or, strictly, a constant rate of crosshead movement (10 mm / min). Depending on the ductility of the material on tests, this corresponds to a rate of deformation in the specimen gauge length of 0.5 to 1% per second. A typical tensile test therefore takes between 60 s and 100 s to rupture, i.e. approx. 0.015 to 0.03 h.

The above diagram demonstrates the relationship between the tensile strength illustrated by the column on the left (N.B.: logarithmic scale!) and the time-dependent stress-rupture strength of Pt-10%Rh, Pt-10%Rh DPH, Pt and Pt DPH at 1600°C.

The comparison shows very markedly the increase of the strengthening effect in DPH alloys relative to the conventional materials with increasing test duration.



Tensile test results on Pt-5%Au DPH are summarized in this figure for temperatures between 1200°C and 1600°C.

The strength decreases steadily with increasing temperature and, especially at the higher temperatures, there is very little difference between $R_{p0.2}$ and R_m.

The ductility at 1200°C is already relatively high (approx. 20% failure strain) and increases steadily with temperature to about 60% at 1600°C.

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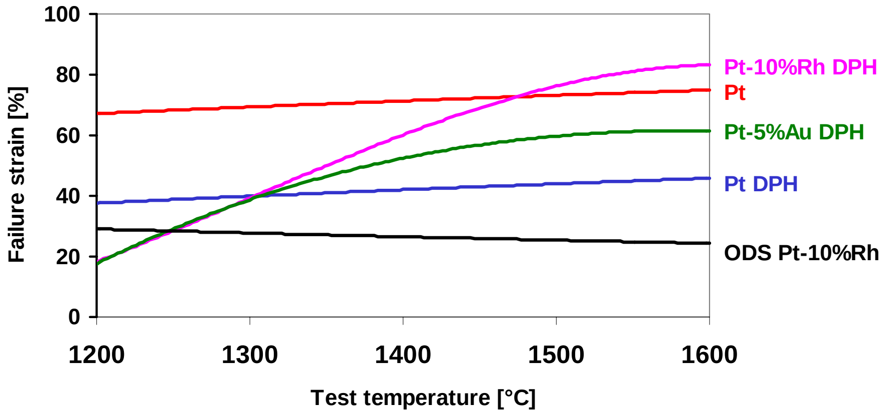
Failure strain of platinum materials



As mentioned above, a remarkable feature of the DPH materials is the very good ductility at high temperatures. However, the influence of temperature on ductility varies considerably from one material to another.

Pt DPH shows elongation values which, although somewhat lower than those for unalloyed Pt, are uniformly high over the whole temperature range investigated.

The alloyed DPH materials Pt-10%Rh DPH and Pt-5%Au DPH reach fracture strains of about 20% at 1200°C and the ductility increases to astonishingly high levels at 1600°C. At the highest temperatures the rupture elongation of Pt-10%Rh DPH even exceeds that of pure Pt.

The behaviour of the DPH materials is in complete contrast to that of other commercially available ODS platinum materials. These show similarly good fracture strain at 1200°C but the ductility decreases continuously with increasing temperature.

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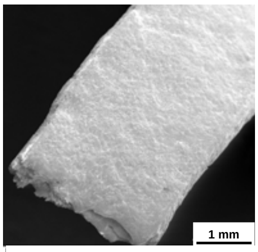
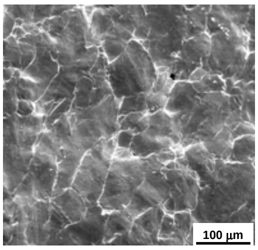
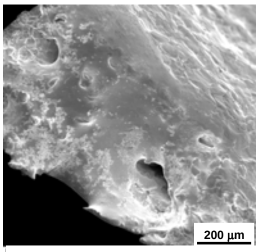
SEM images showing a specimen of Pt DPH after tensile test at 1600°C



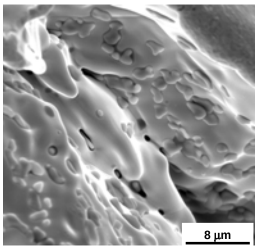
Overview



Surface



Fracture surface



Fracture surface

The difference between the temperature dependence of fracture strain in Pt DPH and Pt-10%Rh DPH is also indicated in SEM investigations.

Pt DPH shows less necking than was observed for Pt-10%Rh DPH, combined with the appearance of larger pores and agglomerations of oxide particles on the fracture face.

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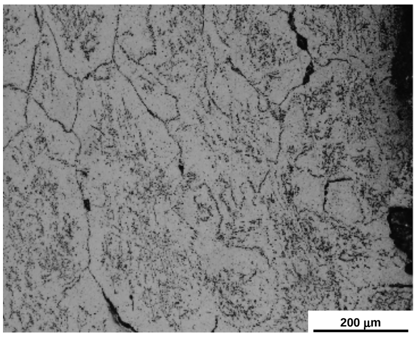
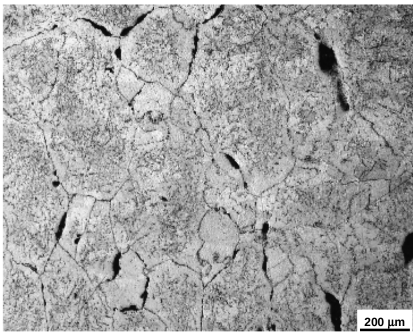
Optical micrographs showing structure of Pt DPH after tensile test (longitudinal sections, points of rupture)



1200°C



1600°C

Optical metallography of longitudinal sections close to the rupture point of Pt DPH specimens shows that there is very little difference between the failure mechanisms operating at 1200°C and 1600°C.

At both temperatures, elongated pores are observed along the grain boundaries. These show a great similarity to the microstructure observed in Pt-10%Rh DPH specimens tested at the lower temperature, but significant differences to the observations at 1600°C.

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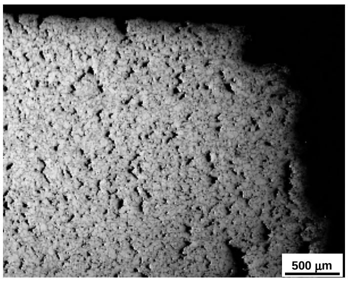
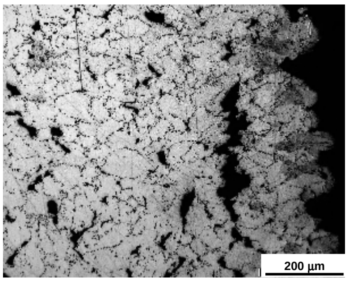
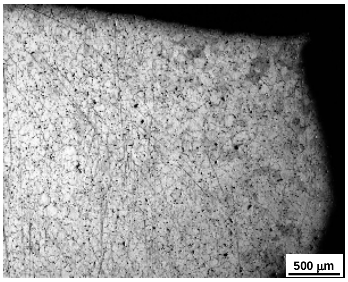
Optical micrographs showing structure of Pt-5%Au DPH after tensile test (longitudinal sections, points of rupture)



1200°C



1200°C



1600°C

The microstructure of Pt-5%Au DPH close to the rupture points of specimens tested at 1200°C and 1600°C shows similar formations of pores to those observed in Pt-10%Rh DPH at the same temperatures.

Once again the appearance of the 1600°C specimen differs considerably from that found in Pt DPH at that temperature.

It is not yet clear why Pt DPH and the alloyed DPH materials exhibit different failure characteristics at the highest test temperatures.

Advantages of the novel platinum DPH materials:

- Workability and weldability are very good.
- High strength after conventional welding.
- Very good ductility at high temperatures.

Using these materials it is possible to produce welded constructions with outstanding properties for applications at extremely high temperatures.

The present results complement the previous observations on the performance of the DPH materials.

The excellent workability and weldability are now well established in large-scale industrial applications.

One of the most remarkable features is that, in contrast to other ODS materials, the strength is largely maintained after conventional welding (e.g. TIG).

Additionally, we have now been able to confirm the excellent ductility at high temperatures.

The DPH materials are ideal for complex welded structural components for use in applications up to very high temperatures.

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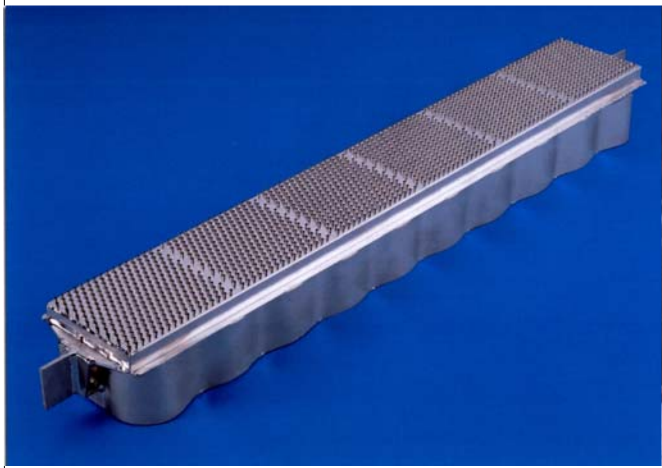
Practical trials in the glass industry



Glass fibre bushing of Pt-Rh alloy

Glass fibre bushing in use

One of the most demanding applications for platinum materials is in bushings for the manufacture of glass fibres.

Because of its unique combination of properties, Pt-10%Rh DPH has proved itself to be an excellent structural material for this application. It is now widely used in bushings with both pressed and welded tips (i.e. the individual glass fibre nozzles).

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Practical trials in the glass industry Bushings made from Pt-10%Rh DPH

Previous bushings made from Pt-10%Rh

- partial operating period on average approx. 1 200 hours
- total service live on average approx. 10 000 hours

Results on bushings made from Pt-10%Rh DPH

- increase in the partial operating period from 1 200 to approx. 1 800 hours
- service live on average approx. 20 000 hours
- the same good fibre drawing properties, fibre quality and usable product yield with the same glass output
- small decrease in energy consumption per kg glass
- reduction in sheet thickness (for 9 200 g total weight approximately 800 g platinum saving)
- fewer repair welds due to better mechanical stability and higher corrosion resistance against aggressive glass melts
- approx. 30% platinum saving for repairs per 10 000 hours service time

→ economic advantages

The status of detailed industrial trials on bushings of Pt-10%Rh DPH was reported two years ago at the 25th IPMI Annual Conference in Tucson.

We would therefore like to bring you up to date.

Some of these bushings are still in operation, having reached more than 20,000 hours service compared with an average of about 10,000 hours for comparable bushings of conventional Pt-10%Rh.

The advantages in reduced wall thickness and the reduction in repair costs have been maintained.

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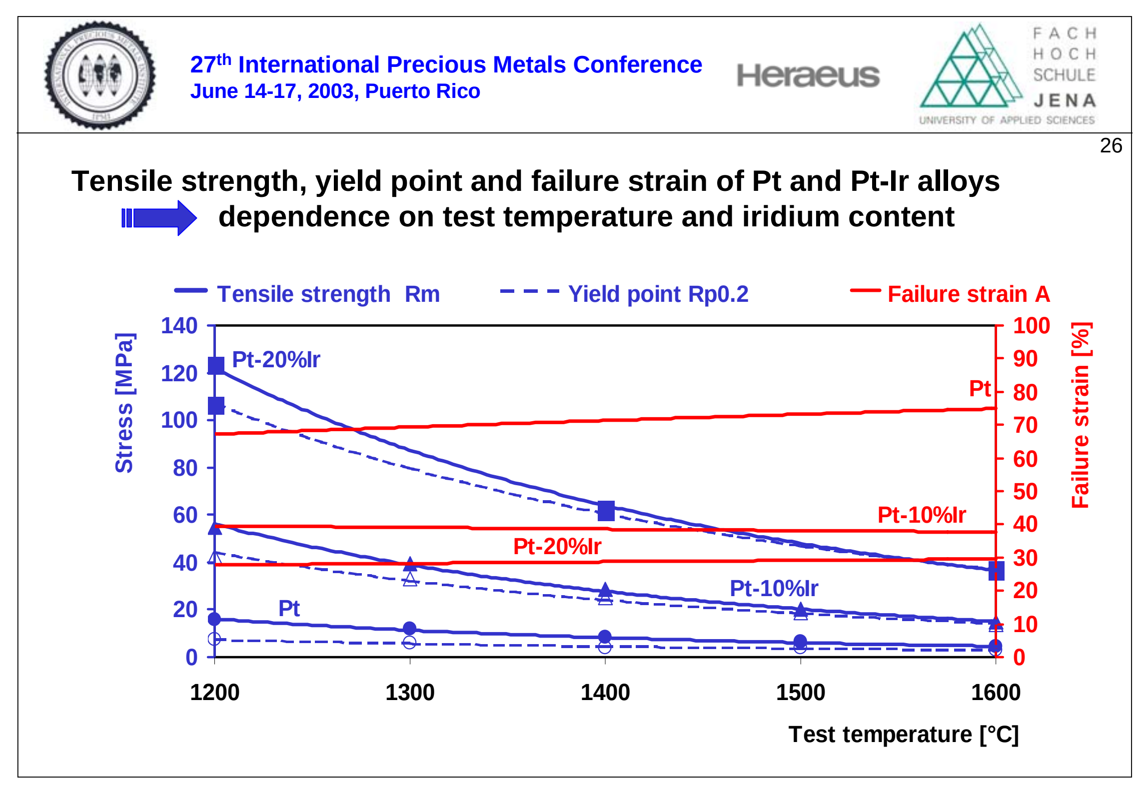
Practical trials in the glass industry Bushings made from Pt-10%Rh DPH

Increased service life gives economic advantages;
Cost savings by reductions in:

- **replacement of unserviceable bushings**
- **disassembly, assembly and devitrification after each cycle (partial operating period)**
- **repair welding**
- **periods of interruption during assembly and disassembly**
- **insulating materials (the bushings are installed in ceramic insulating material)**

These improvements in operating practice continue to yield significant advantages in the economics of glass fibre production, as shown in the above summary.

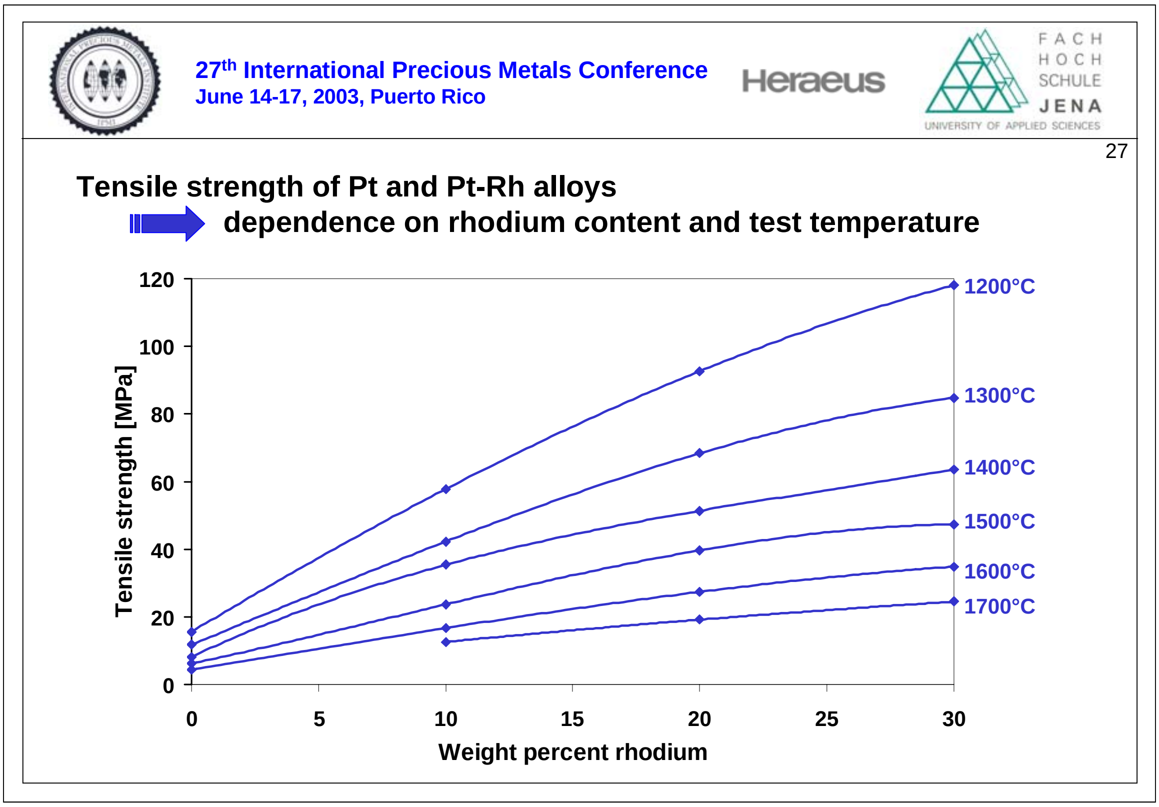


To complete our paper we would like to review the results of the high temperature tensile tests on conventional Pt-Ir and Pt-Rh alloys.

In this figure we see the influence of temperature on the tensile properties ($R_{p0.2}$, R_m, A) of Pt-10%Ir and Pt-20%Ir in comparison to unalloyed Pt.

As would be expected, the mechanical strength properties of all materials decrease steadily with increasing temperature. The strength also increases substantially with increasing Ir concentration.

However, in contrast to the results on the alloyed DPH materials, the ductility of the Pt-Ir alloys increases only slightly with temperature and even at 1600°C reaches a maximum failure strain of nearly 40% for Pt-10%Ir and 30% for Pt-20%Ir.



This figure demonstrates the influence of Rh content and test temperature on the tensile strength of Pt and Pt-Rh alloys with up to 30% Rh.

In common with results we have previously reported on stress-rupture and creep properties and the elastic properties of these materials, it is seen that the strength increases relatively rapidly with Rh content up to about 20%. The further increase in strength from 20% to 30% Rh is less significant.

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Summary

- High temperature tensile test system up to 3000°C
- Tensile strength, yield point and failure strain of Pt, Pt-Rh, Pt-Ir, Pt-Au alloys and the novel Pt DPH materials up to 1600°C (1700°C)
- The novel platinum DPH materials represent a new material class with substantially improved properties.
- Their application is well established in the glass industry.

We would now like to summarize the contents of our paper.

A new test system has been demonstrated for conducting high temperature tensile tests on metallic materials at temperatures up to 3000°C with continuous determination of stress and strain.

The yield point ($R_{p0.2}$), tensile strength (R_m) and failure strain (A) of unalloyed Pt, Pt-Rh, Pt-Ir, and Pt-Au alloys have been determined for the first time at temperatures up to 1600°C and 1700°C for Pt-10%Rh.

Similar determinations have been carried out on Pt DPH and Pt-5%Au DPH up to 1600°C and Pt-10%Rh DPH up to 1700°C.

The results underline the previous observations that the novel platinum DPH materials represent a fundamentally new material class with substantially improved properties. In particular they demonstrate high ductility combined with excellent strength properties up to the highest temperatures.

These materials have continued to establish themselves in demanding applications in the glass industry.